SCHEDULE OF EXAMINATION NO. 6

Issued by Cleveland
Tech. Service Dept.
July 26th, 1922.

DETERMINATION OF TRUE RED LEAD CONTENT

in

LITHARGE AND RED LEAD

The following method is nearly identical with the procedure of the American Society for Testing Materials and is one found in general usage.

Careful manipulation and strict adherance to the details of the procedure are necessary to obtain maximum accuracy.

REAGENTS AND MATERIALS:

1. C.P. Crystallized Sodium Accetate

2. C.P. Potassium Iodide

3. N/10 Sodium-thiosulphate Solution. This solution is prepared as follows: Dissolve 24.83 grams of C.P. sodium thiosulphate, freshly pulverized and dried between filter paper, and dilute with water toone liter at the temperature at which the subsequent titrations are to be made. The solution is best made with well boiled water free from carbon dioxide, or allowed to stand 8 to 14 days before standardizing or using. Standardize with pure, resublimed iodine as described in Treadwell-Hall, "Analytical Chemistry", Vol. II, p.602 (1910), and also against pure potassium iodate. The two methods of standardizing should give results that agree within 0.1 per cent iodine value.

4. N/10 Iodine Solution. 30-25 grams of C.P. potassium iodide are dissolved in as little water as possible in a liter flask. 12.7 grams of commercial iodine are weighed out roughly and added to the potassium iodide solution. The contents of the flask are shaken until the iodine is all dissolved. When this is accomplished the solution is diluted tothe one liter mark with water and standardized on the N/10 sodium-thisulphate solution.

5. Starch Solution. Stir up 2 to 3 grams of potato starch with 100 cc. of 1% salicylic acid solution, and boil the mixture until the starch is practically dissolved, then dilute to one liter.

PROCEDURE:

Weigh 3 grams of the litharge or gram of the red lead into a 200 cc. Erlenmeyer flask. Add a few drops of distilled water and rub the mixture with a flat rubber tipped policeman to a smooth paste. Mix in a small beaker 30 grams of C.P. Tested Purity crystalized sodium acetate, 2.4 grams of C.P. potassium iodide, 10 cc. distilled water and 10 cc. of 50% acetaic acid. Warm gently and stir until all is dissolved adding if necessary 2 to 3 cc. water. Cool to room temperature and pour into the flask containing the lithange or red lead sample. Rub with the policeman until

the litharge or red lead is completely dissolved, add 30 cc. water containing 5 to 6 grams of sodium acetate,and titrate at once with the N/10 sodium-thiosulphate, adding the latter rather slowly and keeping the liquid in constant motion by whirling the flask. When the solution has become light yellow, rub any undissolved particles up with the red until free iodine no longer forms. Wash off the rod, add the sodium-thiosulphate solution until pale yellow,then add 2 to 3 cc. starch solution and titrate until colorless. With deci-normal iodine solution titrate back until the blue color is just restored and substract the cc N/10 iodine solution used from the volume of sodium-thiosulphate that had been added,thus obtaining the number of cc. actually used.

CALCULATION:

The [illegible] of the sodium-thiosulphate solution multiplied by 0.94193 = PbO_2; the iodine value multiplied by 2.69973 = Pb_3O_4; the PbO_2 value multiplied by 2.86616 = Pb_3O_4.

Bulletin No. 23

Feb. 16, 1923.
(Dict. 2-9-23)

Subject SCHEDULE OF EXAMINATION No. 15
" " " No. 16

For Mr. C. S. Neal - Cleveland

We hand you herewith Schedule of Examination No. 15 for the estimation of Zinc in Zinc Oxides, Leaded Zincs, Lithopones, etc., and Schedule of Examination No. 16 for the estimation of Free Zinc Oxide in Lithopone.

Please bring these schedules to the attention of all persons properly concerned in your organization, and acknowledge receipt.

H. A. DERR

Supt. Tech. Service Dept.

HAD

FEB 16 1923

D. Lamme CC

Materials Inspection Division
DC:FMG

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Tech. Service File File

C. S. NEAL

SCHEDULE OF EXAMINATION No. 15

ESTIMATION OF ZINC IN ZINC OXIDES, LEADED ZINCS, LITHOPONES, etc.

SOLUTIONS:

1. Standard Potassium Ferrocyanide. 46 to 48 grams of the C. P. crystallized salt are dissolved in 1000 cubic centimetres of distilled water.

2. Uranium indicator. A 5% solution of uranium acetate or uranium nitrate is made in distilled water slightly acidified with acetic acid.

STANDARDIZATION OF THE FERROCYANIDE:

Weigh into 300 c. c. beakers several portions of c. p. zinc, using about 0.3 gram. Cover with distilled water and dissolve in about 10 c. c. of hydrochloric acid. Now add ammonia water till slightly alkaline, barely acidify with hydrochloric acid, and add 3 c. c. in excess. Add sufficient water to bring the volume to about 200 c. c. and heat to almost boiling. Transfer about one-third of the solution to another beaker, and run the standard ferrocyanide solution into the main portion with constant stirring until a drop tested on a white porcelain plate with a drop of the indicator, turns brown. Add nearly all of the reserved portion, saving about 10 c. c., and again titrate till a drop turns uranium indicator brown, being careful not to add too large an excess. Now add the remainder of the reserved solution, rinsing the beaker out with a little water, and finish the titration, adding potassium ferrocyanide drop by drop till a drop tested with a drop of the indicator turns brown after standing one minute. A blank should be run with the same amounts of reagents and water as in the standardization. The amount of ferrocyanide solution required for the blank should be subtracted from the amounts used in standardization and in the subsequent titration of the sample. The standardization must be made under the same conditions of temperature, volume and acidity as obtain when the sample is titrated.

CALCULATION: The number of cubic centimetres required (minus the number for the blank), divided into the weight of zinc taken, equals the zinc equivalent for 1 c. c. of standard solution. This factor multiplied by 1.2448 gives the zinc oxide equivalent, and by 1.4906 the zinc sulphide equivalent.

PROCEDURE FOR THE SAMPLE:

For zinc oxides and leaded zincs weigh accurately 0.5 gram. Dissolve in hydrochloric acid, make ammoniacal, reacidify, dilute to 200 c. c., etc., and titrate just as in standardization, taking care that the same conditions of volume, acidity and temperature obtain. In the

case of lithopone weigh 1 gram, dissolve in hydrochloric acid and boil till all hydrogen sulphide is expelled. Dilute with water, and filter off the barium sulphate, then proceed as before.

No. of c. c. of standard ferrocyanide multiplied by the correct factor, divided by weight of sample, multiplied by 100, gives the result.

RAW MATERIALS SCHEDULE OF EXAMINATION No. 23

Revised by Cleveland
Tech. Service Dept.
January 22, 1924.

TESTS FOR COMPOUNDS IN LEAD CHROMATE YELLOWS WHICH CAUSE THICKENING WITH PAINT VEHICLES.

As it appears that Chrome Yellows are apt to contain impurities which cause thickening or "livering" when ground into paint, the following tests have been worked out to detect such impurities:

1. THICKENING WITH (488):

4 ounces of the sample are weighed into a 1/2 pint can; 3 liquid ounces of (488), Wood Oil Gum Rubbing Varnish, are measured out and added with constant stirring. The mixture is thoroughly stirred, the can then covered tightly, and allowed to stand 24 hours. The can is then opened, the mixture first stirred, and the consistency then noted by allowing it to run off the spatula. It should not be appreciably thicker than when it was first mixed.

Ordinarily, the foregoing will be the only test applied. In case, however, it is considered desirable to determine the nature and amount of injurious impurities, the following methods may be used:

2. DETERMINATION OF LIME (Total)

A 2-gram sample is weighed into a beaker, and dissolved in dilute nitric acid, which dissolves the calcium compounds without reducing the chromium. The solution is then made alkaline with ammonia, and acidified with acetic acid. Potassium bichromate solution is then added in excess, to precipitate the excess lead, and the solution heated to boiling. It is then filtered and the precipitate washed. Ammonium oxalate is added to the filtrate to precipitate the lime, then ammonium hydroxide in excess. The precipitate is kept warm for about an hour, then filtered, and thoroughly washed with hot water. The calcium may now be determined either gravimetrically or volumetrically. If the gravimetric method is followed, the precipitate is ignited gently till completely charred, then with the full heat of a blast burner, cooled and weighed as Calcium Oxide. If the volumetric method is preferred, a hole is punched in the paper and the paper and beaker in which precipitation was made, treated with hot dilute sulfuric acid. The solution in the beaker (which must not contain any pieces of filter paper) is heated to boiling, allowed to stand two or three minutes, then titrated with tenth-normal potassium permanganate, added cautiously from a burette until a permanent pink color is obtained. One cc N/10 $K MnO_4$ = 0.0028 gm. CaO.

3. DETERMINATION OF WATER-SOLUBLE IMPURITIES:

Among the impurities apt to be present on account of insufficient washing are alkali chromates and sulphates. A preliminary test for their presence is made by pouring hot water over some of the pigment in a beaker, stirring and filtering. A portion of the filtrate is acidified with acetic acid and tested by adding a few drops of lead acetate, which will precipitate any chromate present. Another portion is tested for sulphate by acidifying with H Cl and adding Barium Chloride. If only chromates are present, proceed as in paragraph (a). If only sulphates are present, proceed as in paragraph (b). If both are present, proceed as in paragraph (c).

Procedure when only Chromates are present:

(a) 5 grams of the pigment are weighed into a 250 c c beaker. About 100 c c of boiling water are poured over it, and the mixture thoroughly stirred from time to time for a period of about 15 minutes. It is then filtered by decantation. The pigment remaining in the beaker is treated with 4 more portions of boiling water and filtered as before. The combined filtrates are then acidified with acetic acid. 10 c c of 10% lead acetate is added and the solution boiled 2 or 3 minutes. It is then filtered through a Gooch crucible, the precipitate washed with hot water, dried at 100°C and weighed as Pb Cr O_4. This is calculated to Cr O_3 by multiplying by the factor 0.3097.

Procedure when only Sulphates are present:

(b) 5 grams of pigment are extracted with 5 portions of boiling water as in paragraph (a), the combined filtrate acidified with 5 cc H Cl. The solution is brought to boiling, 10 c c 10% Ba Cl_2 are added, the precipitate allowed to settle overnight, filtered, washed, ignited and weighed as Ba SO_4. Ba SO_4 x 0.343 = SO_3.

Procedure when both Sulphates and Chromates are present:

(c) 10 grams of the pigment are weighed into a 250 cc beaker. 90 c c of boiling water are poured over it, and the mixture stirred thoroughly from time to time for a period of about 15 minutes. It is then filtered by decantation. The pigment remaining in the beaker is treated with 4 more successive 90 cc portions of boiling water decanting through a filter each time as before. The combined filtrates are then transferred to a 500 c c volumetric flask, the solution made up to the mark and thoroughly mixed. This is then divided into two portions of 250 cc each. For the determination of sulphates, one portion is acidified with 5 cc conc. H Cl, 25 cc of alcohol added, and the mixture boiled for 15 minutes. 10 c c of 10% Ba Cl_2 solution are added, the solution allowed to settle overnight, filtered, washed with 2% hydrochloric acid solution, ignited and weighed as Ba SO_4. Ba SO_4 x 0.343 = S O_3. The other

portion for the determination of Cr O_3, is acidified with 10 c c conc. H Cl, 25 c c alcohol are added to reduce the chromium, and the solution boiled for 15 or 20 minutes, when the color should be a grass green. Ammonium hydroxide is now added carefully until the solution is alkaline to litmus and the odor of ammonia is barely perceptible. The precipitate is boiled for about 10 minutes, then a drop or two of ammonia is added; the solution will then settle out clear. The chromic hydroxide is filtered off and washed carefully with hot water. The well-drained precipitate is dried, ignited with the full heat of a Meker burner, cooled and weighed as $Cr_2 O_3$. $Cr_2 O_3$ x 1.3158 = Cr O_3 .

RAW MATERIAL SCHEDULE OF EXAMINATION #35

Issued by Cleveland
Tech. Service Dept.
January 29, 1924.

DETERMINATION OF SULPHUR DIOXIDE IN LEADED ZINC PIGMENTS.

ZINC OXIDES, ETC.

Weigh 5 grams of the pigment into a 250 cc beaker. Add 100 cc of freshly boiled distilled water, and 5 cc of concentrated sulphuric acid. Stir thoroughly.

Allow to stand for 15 mimutes, and then titrate with N/20 iodine solution, using starch solution as an indicator.

Calculation:- No. of cc of N/20 iodine solution $\times$ 0.0016 (SO_2 equivalent of 1 cc of N/20 iodine solution) $\div$ by weight of sample (5 grams) $\times$ 100 = % of SO_2.

Reactions:

$$ZnSO_3 + H_2SO_4 = ZnSO_4 + H_2O + SO_2$$

$$SO_2 + 2I + 2H_2O = H_2SO_4 + 2HI$$

RAW MATERIAL SCHEDULE OF EXAMINATION #34

Issued by Cleveland
Tech. Service Dept.
January 29, 1924.

DETERMINATION OF ZINC SULPHATE IN ZINC OXIDE AND LEADED ZINC PIGMENTS, ETC.

The following method is essentially the same as the A. S. T. M. standard method.

Weigh 5 grams of the pigment into a 250 cc beaker. Add 150 cc of distilled water and 50 cc of alcohol, and heat the mixture nearly to boiling. Hold at a nearly-to-boiling temperature for 30 minutes. Allow the pigment to settle, then filter, and wash thoroughly with 1 : 3 alcohol and water mixture.

Heat the filtrate to boiling, expel most of the alcohol, and add 5 cc of 10% barium chloride solution, drop by drop. Boil for 5 minutes, allow to settle, filter through "ashless filter paper", wash, ignite precipitate, and weigh, all in the usual manner for this precipitation as barium sulphate.

Calculation:- Weight of precipitate $\times$ 0.6916 (zinc sulphate equivalent), $\div$ by weight of sample $\times$ 100 = % of zinc sulphate.

44

SCHEDULE OF EXAMINATION NO. 44

Issued by Cleveland
Technical Service Dept.,
January 2, 1925.

ANALYSIS OF YELLOW, ORANGE, AND VERMILION PAINTS AND PIGMENTS

(This Schedule is derived from Chapter XXIV of Dr. Holley's book, "Analysis of Paint & Varnish Products", edition of 1912)

1. COMPOSITION

The lemon yellow chromes usually contain sulphate of lead, sometimes carbonate of lead. The red chromes, known by the various names of scarlet chrome, chrome red, Chinese red, American vermilion, and vermilion substitute, may be considered as basic chromates of lead. Often these basic chromes are brightened up by having precipitated on them an organic color; this may be tested for by treating a portion of the pigment with alcohol, which will dissolve the organic color, giving a strongly colored solution. See analysis of vermilions.

2. HYGROSCOPIC MOISTURE

Heat 2 grams at 105° C. for 3 hours. Loss in weight represents hygroscopic moisture.

3. BARYTES, SILICA, AND CLAY

One gram of the pigment is boiled for 5 minutes with 30 c.c. of concentrated hydrochloric acid in a covered beaker. While boiling add half a dozen drops of alcohol one at a time. Fifty c.c. of water is added and the boiling continued for 10 or 15 minutes. Filter, wash thoroughly with boiling water, ignite, and weigh. The insoluble residue is fused with sodium carbonate, and the barium, silica, and alumina separated as described under analysis of white paints.

4. LEAD

The filtrate from the insoluble residue is neutralized with dilute ammonia until the further addition of another drop would cause the formation of a permanent precipitate, diluted to about 250 c.c. to 300 c.c., and hydrogen sulphide passed in for 10 minutes.

Solutions containing large amounts of chromium, if neutralized with ammonia until a permanent precipitate appears, seem to require an excess of hydrochloric acid for their resolution, sufficient to prevent the satisfactory precipitation of the lead with the hydrogen sulphide.

Allow the precipitate to settle thoroughly, as it renders the filtering much easier, filter, wash with hydrogen sulphide water. Boil, filter, and precipitate with dilute nitric acid, until all of the lead has dissolved, filter with aid of suction, washing thoroughly with hot water. Add 5 c.c. of concentrated sulphuric acid, diluted with an equal volume of water, to the filtrate. Evaporate on hot plate until the white fumes of sulphur trioxide appear. Cool, dilute with water, add an equal volume of alcohol, filter, washing with dilute alcohol, ignite gently, and weigh as lead sulphate. Save filtrate.

5. CHROMIUM

The alcoholic filtrate from the lead sulphate is evaporated nearly to dryness to expel alcohol, and the filtrate from the lead sulphide heated until the hydrogen sulphide is expelled. The two filtrates are mixed, diluted if necessary, and made just perceptibly alkaline with ammonia; boil, settle, filter, wash thoroughly, ignite, and weigh as chromic oxide.

Wt. chromic oxide x 1.3137 = wt. chromic anhydride.

Occasionally these pigments contain a small quantity of iron, which should be tested for qualitatively in a separate portion of the pigment. If found to be present, the precipitate of ferric and chromium hydroxides is dissolved on the filter with hydrochloric acid, the filter washed thoroughly with hot water, and the iron and chromium in the filtrate reprecipitated with ammonia and treated with sodium peroxide to dissolve the chromium as described under the analysis of chrome greens.

6. CALCIUM

The filtrate from the chromium is treated with ammonium oxalate, allowed to stand in a warm place for an hour or so, filtered, washed thoroughly, strongly ignited, and weighed as calcium oxide.

7. MAGNESIUM

The magnesium is estimated in the filtrate from the calcium in the usual manner.

8. COMBINED SULPHURIC ACID

The combined sulphuric acid may be estimated by either of the two methods given in the chapter devoted to the analysis of white paints. In fact, the latter method may be used for the rapid analysis of a chrome lead, the insoluble lead carbonate being filtered off, the chromium precipitated as the hydroxide in the usual manner, and the combined sulphuric acid estimated in the filtrate from the chromium.

Wt. barium sulphate x 0.3433 = combined sulphuric acid.

9. CALCULATIONS

If calcium is absent, or present as carbonate, the combined sulphuric acid is calculated to lead sulphate, the chromic anhydride to lead chromate, and excess of lead to lead oxide. If calcium sulphate and carbonate of lead are present, the carbon dioxide must be determined and the amount of calcium present as sulphate estimated by Thompson's method as described under analysis of white paints.

Wt. comb. sulphuric acid x 3.788 = wt. lead sulphate.

Wt. chromic anhydride x 3.230 = wt. lead chromate.

Wt. lead chromate x 0.6406 = wt. lead.

Wt. lead x 1.0773 = wt. lead oxide.

ANALYSIS OF MIXED PAINTS CONTAINING CHROME YELLOWS AND OCHRES.

10. BARYTES, SILICA, AND CLAY are estimated as described under analysis of chrome leads.

11. LEAD, both as sulphate and carbonate, is estimated as described under chrome leads, the filtrate from the lead sulphate being saved as before.

12. IRON

The filtrate from the lead sulphide is heated until all of the hydrogen sulphide has been expelled and added to the filtrate from the lead sulphate from which the alcohol has been expelled by boiling. A few drops of nitric acid are added and the solution boiled for a minute or two, then made just distinctly alkaline with ammonia, boiled, settled, and filtered.

Dissolve on the filter with hot dilute hydrochloric acid, wash with hot water. Cool. Reprecipitate with ammonia, avoiding excess, without waiting for the precipitate to settle, carefully add a sufficient quantity of sodium peroxide (1 gram is usually sufficient), keeping the beaker covered meanwhile. Digest until all of the chromium and aluminum have passed into solution, adding more peroxide if necessary. The iron remains undissolved, while the chromium and aluminum go into solution; filter, wash thoroughly, ignite strongly, and weigh as ferric oxide, or dissolve in dilute hydrochloric acid and titrate. The treatment with peroxide is preferably performed in a porcelain evaporating dish.

13. CHROMIUM

The filtrate from the iron is made up to 250 c.c. in a graduated flask. An aliquot portion is rendered acid with acetic acid and a slight excess of

lead nitrate solution added, allowed to remain on the hot plate until thoroughly settled, filtered onto a weighed Gooch crucible, washed, dried, and weighed as lead chromate.

14. ALUMINUM

An aliquot portion of the 250 c.c. solution is made just acid with hydrochloric acid, and then just distinctly alkaline with ammonia, allowed to settle, filtered onto a Gooch crucible, ignited, and weighed as alumina.

15. ZINC

The filtrate from the chromium, iron and aluminum hydroxides, under iron is mixed with the filtrate from the lead sulphate from which the alcohol has been expelled, and the mixed solution saturated thoroughly with hydrogen sulphide, boiled with the addition of solid ammonium chloride to render the precipitate less slimy, and filtered. The zinc sulphide dissolved with hydrochloric acid, boiled to expel hydrogen sulphide, and titrated with standard ferrocyanide of potassium as described under analysis of white paints.

16. CALCIUM, MAGNESIUM, AND COMBINED SULPHURIC ACID are estimated as described under analysis of chrome leads and the calculations made as there described.

ANALYSIS OF VERMILION

17. PROPERTIES

Vermilion is a bluish scarlet powder, having a specific gravity of 8.2. It is insoluble in any single acid such as hydrochloric or nitric acid and in the alkalies. Heated in contact with the air, it burns with a pale blue lambent flame. Pure vermilion will burn away entirely or at least leave but a small fraction of 1 per cent of ash. This is a reliable test for it, as other adulterants would be left behind on heating.

The most common adulterants of vermilion are red lead, oxide of iron, lead chromes, vermilionette lakes, para reds, and alizarine reds.

18. DETECTION OF VERMILIONETTES, PARA AND ALIZARINE REDS

(a). Boil a little of the dry color with water, settle, and filter. Vermilionettes give a deep red solution, para reds a pale brownish or orange, and the alizarine reds a colorless solution.

(b). Boil a little of the dry color with a mixture of methyl and ethyl alcohol, filter, heat, and settle. Vermilionettes give a bright red solution, usually having a yellow "bloom," para reds an orange-red solution, alizarine reds a practically colorless solution.

(c). Boil another portion of the dry pigment with some freshly distilled aniline, settle, and filter. Vermilionettes give a purple-red, alizarine lakes a pale brown, and the para reds an intense orange-red solution.

(d). Boil some of the dry color with a solution of caustic soda. Vermilionettes give a red solution with a green "bloom," para reds a bluish red solution, while alizarine reds yield a characteristic deep violet solution.

19. BARYTES, SILICA, AND CLAY

Dissolve 1 gram in 30 c.c. of concentrated hydrochloric acid, 50 c.c. of water with the aid of 1 to 2 grams of potassium chlorate added in small portions and warming. Evaporate to dryness on water bath. Take up in 50 c.c. of water acidulated with hydrochloric acid, heat to boiling to dissolve any lead chlorides, filter, wash with boiling water, ignite, and weigh any insoluble residue. Fuse with sodium carbonate and estimate the barium sulphate, silica, and alumina as described under analysis of white paints.

20. LEAD

If lead is present, calcium compounds being absent, the filtrate is treated with sulphuric acid, evaporated carefully to expel excess of hydrochloric acid, diluted with water and alcohol, the lead sulphate filtered off on a Gooch crucible in the usual manner.

21. MERCURIC SULPHIDE (VERMILION)

The filtrate from the insoluble residue, if lead is absent, or the filtrate from the lead sulphate, is heated with a little sulphurous acid to reduce any iron present to the ferrous condition, made neutral with ammonia, and then just acid to litmus with hydrochloric acid.

The solution is diluted to about 350 c.c. and hydrogen sulphide passed in for 10 minutes. The mercuric sulphide is filtered off on a weighed Gooch crucible, washed with hydrogen sulphide water, the crucible removed to another holder and washed with alcohol and carbon bisulphide to remove sulphur, dried in steam oven, and weighed.

22. ESTIMATION OF LEAD AND MERCURY, CALCIUM COMPOUNDS PRESENT

The filtrate from the insoluble residue from 19 is precipitated with hydrogen sulphide as described under 21, collected on a weighed filter, and dried at 100° C., weighed, and mixed uniformly.

An aliquot part is introduced into the bulb of Fig. 10, (See Dr. Holley's book) a slow stream of washed chlorine gas passed through it, and a gentle heat applied to the bulb, increasing this gradually to faint redness. The escaping chlorine is conducted into a flue. First, sulphur chloride distills over, which decomposes with the water in E and F. The mercuric chloride formed volatilizes, condensing partly in E, partly in C.

Cut off that part of the tube, rinse the mercuric chloride into E, and mix the contents of the latter with the water in F. Mix the solution with excess of ammonia and warm gently until no more nitrogen is evolved, acidify with hydrochloric acid, filter, and determine the mercury in the filtrate as under 21.

23. FERRIC OXIDE

The filtrate from the sulphides is heated until all of the hydrogen sulphide has been expelled and the iron chromium and alumina precipitated with ammonia, filtered and separated as described under analysis of chrome greens.

24. ZINC OXIDE

The filtrate from the iron and alumina precipitate is made distinctly alkaline with ammonia and the zinc precipitated with hydrogen sulphide. The liquid containing the zinc sulphide precipitate is heated to boiling, and about 5 grams of solid ammonium chloride added, which renders the precipitate easier to filter. Settle, filter, wash thoroughly. Pierce filter, wash through into a clean beaker with water, dissolving the residue on filter with dilute hydrochloric acid, and washing with hot water. Dilute, heat to expel hydrogen sulphide, and titrate with ferrocyanide as previously described. If iron is absent in the paint, the zinc may be estimated directly as described under analysis of white pigments.

25. CALCIUM AND MAGNESIUM

Estimated as usual in the filtrate from the zinc sulphide.

26. CALCULATIONS

If chromium is present, it is calculated to basic lead chromate, and any excess of lead above that required to form the chromate is calculated to red lead.

27. ANTIMONY VERMILION AND ORANGE

These two pigments have the same composition, corresponding to the formula of antimony trisulphide. They are insoluble in dilute acids, but soluble in strong hydrochloric acid. It is seldom necessary to make a complete analysis of these pigments. Adulteration will be indicated by the pigment not being completely soluble in strong hydrochloric, though a trace of sulphur may remain undissolved, floating on top of the acid.

45

SCHEDULE OF EXAMINATION NO. 45

Issued by Cleveland
Technical Service Dept.,
January 13, 1925.

ANALYSIS OF BLACK PIGMENTS AND PAINTS

(This Schedule is derived from Chapter XXI of Dr. Holley's book, "Analysis of Paint & Varnish Products", edition of 1912)

1. COMPOSITION

The ordinary black pigments — lampblack, vegetable black, bone black, ivory drop black, gas black, graphite, etc. -- contain carbon as their essential constituent, and while all of these products are said to be of a black color, they vary greatly in shade and still more so in tinting strength.

Lampblack and vegetable black are essentially soot blacks, being the soot deposited from the combustion of oily bodies such as dead oil. Lampblack has a distinct gray tint, as may be shown by comparison with ivory black. These blacks are apt to contain varying quantities of oil, owing to the nature of their manufacture; less than 1 per cent of oil often being sufficient to retard seriously the drying of lampblack paints. Vegetable blacks are more voluminous than lampblacks and are usually of a jet black color.

Carbon black is usually produced from the incomplete combustion of natural gas. While its tinting power is very great, its use has been largely abandoned owing to its tendency to produce a streaky color when used in tinting paints.

Bone black is, as its name indicates, obtained by the charring of bones in retorts. The carbon content varies usually between 12 and 22 per cent, the balance consisting of moisture, phosphate of calcium, and carbonate of calcium. The best grades of bone black are made from selected sheep bones. An exceedingly intense black is made by digesting selected bones in hydrochloric acid until all of the mineral matter is dissolved, leaving the carbon in a flocculent state. This black is often sold under the name of black toner, and is one of the highest priced blacks.

Animal black is a name sometimes given to bone black but is also used to designate a wide variety of blacks prepared in the same way as bone black from waste animal products of all kinds, as leather scrap parings, horn trimmings, etc.

Frankfort black, drop black, and German black are terms used to designate blacks made from a variety of organic materials, such as vine twigs, refuse of wine making, peach stones, bone shavings, etc. These blacks vary in hue from a bluish black to a reddish black.

Graphite, while not used to any extent in house paints, is largely used in bridge, elevator, and warehouse paints. It is rarely used by itself for these purposes, silica, calcium carbonate and iron oxide pigments, zinc and lead being the other usual constituents. It may be tested qualitatively in the extracted pigment by rubbing a portion of the sample between the palms, which soon assume the characteristic appearance produced by stove polish. Of all the black pigments graphite alone gives this test.

Charcoal black and vine black are produced by the charring of wood products, and contain besides carbon the ash ingredients common to wood. Charcoal blacks are usually made from maple, willow, and basswood, and vine blacks from the charring of the grapevine. Paints containing considerable quantities of these blacks are liable to saponify badly owing to the moisture and potash salts present.

Mineral black, which is still occasionally used, is black slate finely ground.

2. MOISTURE

Dry 2 grams at 105° C. for 3 hours. Loss in weight represents approximately the amount of moisture present.

3. OILS

Extract 2 grams with ether in a fat extraction apparatus. After removing the ether and drying, any increase in weight represents the amount of oily matter present.

4. ASH

Two grams are weighed into a crucible and heated over a Bunsen burner until all the carbon is burned off. If the ash constitutes only a small per cent, it may be cooled and weighed directly. Otherwise the residue is moistened with a solution of ammonium carbonate, heated gently, and weighed. The object of this operation is to restore the carbon dioxide which may have been expelled from the bases by the strong heat to which they have been subjected.

5. CARBON

The carbon is usually estimated by difference, by adding together the moisture, oil, and ash, and subtracting from 100.

6. CALCIUM

Digest the residue from 4 in a mixture of 25 c.c. of concentrated hydrochloric acid and 5 c.c. of concentrated nitric acid on the hot plate for one-half hour, dilute, filter, and make up to 250 c.c. in a graduated flask. Any appreciable residue on the filter may indicate addition of barytes, silica,

clay, or alumina. Determine the calcium and magnesium in an aliquot portion of the solution by adding ammonia in small quantities until a precipitate is formed, then acetic acid in excess until redissolved, except for traces of iron which may be removed by filtration. Ammonium oxalate is added, and the calcium precipitate treated in the usual manner.

7. PHOSPHORIC ACID

Take an aliquot portion of the solution prepared above, neutralize with ammonia, and clear with a few drops of nitric acid, add about 5 grams of dry ammonium nitrate or a solution containing that amount. To the hot solution add 50 c.c. of molybdic solution for every decigram of phosphoric acid that is present. Digest at about 65° for an hour, filter, and wash with cold water, or preferably ammonium nitrate solution. Test the filtrate for phosphoric acid by renewed digestion and addition of more molybdic solution. Dissolve the precipitate on the filter with ammonia and hot water, and wash into a beaker to a bulk of not more than 100 c.c. Nearly neutralize with hydrochloric acid, and add magnesia mixture from a burette; add slowly (about 1 drop per second), stirring vigorously. After 15 minutes add 30 c.c. of ammonia solution of density 0.96. Let stand for some time; 2 hours is usually enough. Filter, wash with 2.5 per cent ammonia, ignite to whiteness or to a grayish white, and weigh as magnesium pyrophosphate.

Molybdic solution

Dissolve 100 grams of molybdic acid in 400 grams or 417 c.c. of ammonia, specific gravity 0.96, and pour the solution thus obtained into 1500 grams or 1250 c.c. of nitric acid, specific gravity 1.20. Keep the mixture in a warm place for several days, or until a portion heated to 40° deposits no yellow precipitate. Decant the solution from any sediment and preserve it in glass-stoppered vessels.

Magnesia Mixture

Dissolve 110 grams of crystallised magnesium chloride, 280 grams of ammonium chloride, in 700 c.c. of ammonia of specific gravity 0.96, and sufficient water to make 2000 c.c.

8. MAGNESIUM

The filtrate, from which the calcium has been precipitated, is evaporated to a small bulk and made alkaline with ammonia. After standing several hours the magnesium precipitate is filtered, ignited, and weighed, and calculated for magnesium by multiplying by 21.88.

9. CALCULATIONS

The magnesium is calculated to magnesium phosphate, and the remainder of the phosphoric acid to calcium phosphate. Any calcium remaining is calculated to calcium carbonate.

Germine ivory black, made by carbonizing waste fragments and turnings of ivory, is often adulterated with bone black, which is somewhat similar in composition, but contains only a small amount of magnesium phosphate as compared with the ivory black.

ANALYSIS OF MIXED PAINTS TINTED WITH BLACK AND OXIDE OF IRON PIGMENTS

10. CARBON

One gram of the pigment is dissolved in hydrochloric acid as described under white pigments, and the residue filtered through an ashless filter, that has been dried in the hot-water oven and weighed. After washing the residue with boiling water the filter and contents are dried and weighed, then ignited until all the carbon is burned off, and weighed again. The percentage of carbon is obtained by difference. Where the percentage of color is small, it is often estimated by difference, adding together the determined constituents and subtracting from 100.

11. FERRIC OXIDE

If the filtrate from the insoluble residue is of an appreciable yellow color, it indicates that the tint has been "warmed up" by the addition of an ochre or oxide, in which case the lead is precipitated and estimated as described under analysis of white pigments, the filtrate from the lead sulphide heated until all of the hydrogen sulphide has been expelled and the iron and alumina precipitated with ammonia after having been oxidized by boiling with a few drops of nitric acid, filtered and ignited and weighed in a porcelain crucible, the residue fused with bisulphate of potassium, dissolved in water with the aid of a little hydrochloric acid, heated to boiling, reduced with stannous chloride; mercuric chloride and manganous sulphate solution added and titrated with permanganate in the manner described under analysis of Venetian reds.

12. ALUMINA

The alumina is calculated by difference from the data obtained under 11.

13. ZINC OXIDE

The filtrate from the iron and alumina precipitate and the filtrate from the lead sulphate precipitate, the alcohol having been removed by evaporation, are mixed, made distinctly alkaline with ammonia, and the zinc precipitated with hydrogen sulphide. The liquid containing the zinc sulphide precipitate is heated to boiling, and about 5 grams of solid ammonium chloride added, which renders the precipitate easier to filter. Settle, filter, and wash thoroughly. Pierce filter, wash through into a clean beaker with water, dissolving the residue on filter with dilute hydrochloric acid, and wash with hot water. Dilute, heat to expel hydrogen sulphide, and titrate with ferrocyanide as previously described. If iron is absent in the paint the zinc may be estimated directly as described under analysis of white paints.

14. CALCIUM AND MAGNESIUM

Estimate as usual in the filtrate from the zinc sulphide.

15. RESIDUE INSOLUBLE IN HYDROCHLORIC ACID

Fuse with sodium carbonate as previously described. Dissolve in water and filter. Iron not previously dissolved will remain on the filter as ferric oxide along with any barium that may be present. This residue after thorough washing is dissolved with the aid of a small quantity of hydrochloric acid, the barium precipitated as usual, and the iron estimated in the filtrate from the barium sulphate. The silica and alumina are estimated as usual.

16. LEAD SULPHATE

Determine the combined sulphuric acid as described under analysis of white paints and calculate to lead sulphate in the absence of calcium sulphate. If calcium carbonate and calcium sulphate are both present the nitric acid-alcohol separation should be used.

17. GRAPHITE PAINTS

The term graphite as applied to paints does not necessarily mean that such paints are composed of graphite wholly or even in part. In fact there is no accepted standard of purity for graphite itself. One paint manufacturer may purchase a natural graphite for his line of graphite paints which contains 20 per cent of graphitic carbon, the other 80 per cent being silica and various silicates; others may purchase graphites containing 80 per cent or more of graphitic carbon; and some purchase products which are very nearly pure graphitic carbon, prepared electrically. While it is a far from settled question, perhaps the majority of paint manufacturers and those who buy graphite paints under strict specifications are of the opinion that a graphite paint affords better service value when it contains in part something besides graphitic carbon.

18. COLORED GRAPHITE PAINTS

Having established his graphite paint on the market, the manufacturer soon has requests for colors other than black, and in order to meet the requirements of his trade he finds it necessary to add considerable amounts of tinting pigments, with the natural result that in time many of his formulas contain little or no graphite.

The following analyses will give some idea of the pigment combinations to be found on the market:

	I. Natural Graphite.	II. Olive Graphite.	III. Indian Red Graphite.	IV. Seal Brown Graphite.
	Per cent.	Per cent.	Per cent.	Per cent.
Graphitic carbon	32.14	21.89	...	5.31
Calcium carbonate	42.86	19.16	44.59	26.12
Magnesium silicate and silicate	25.00	...	12.17	33.72
Ochre	...	58.95	...	...
Ferric oxide	...	...	43.24	34.85
	100.00	100.00	100.00	100.00

The pigment portion of one sample analyzed by the author was nothing but Keystone filler; another, carbon black, which while as expensive as the ordinary grades of graphite is nevertheless radically different. Graphite paints which are sold according to specifications for structural iron and steel work are usually high-grade products.

SCHEDULE OF EXAMINATION No. 75

DETERMINATION OF THE TRUE LEAD CONTENT, Pb_3O_4, IN [illegible] PAINTS CONTAINING CARBON AND OTHER MATERIALS THAT INTERFERE WITH THE THIOSULPHATE TITRATION WHEN THE REGULAR METHOD IS

- 1 -

PREFERRED METHOD - Weigh out two grams of the dry pigment (where a small amount of Red Lead is present) and transfer to a 250 c.c. beaker. Add from 50 to 60 c.c. of the solution of Acetic Acid containing Potassium Iodide and saturated with Sodium Acetate. (See solution No. 4). Immediately add from a pipette exactly 25 c.c. of N/10 Sodium Thiosulphate, stir and place on the hot plate or steam bath for 15 to 20 minutes at about 60° to 70° C. (Don't boil). Filter while hot through a Gooch Crucible. Wash thoroughly with hot water and titrate the excess Thiosulphate with N/10 Iodine solution.

One c.c. of N/10 $Na_2S_2O_3$ is equal to [illegible]

Therefore:

$$\frac{(\text{c.c. N/10 } Na_2S_2O_3 \times F_1) - (\text{c.c. N/10 Iodine} \times F_2)(0.[illegible])}{\text{Weight of sample}}$$

equals the per cent of Pb_3O_4, or True Red Lead, where F_1 and F_2 represent the normality factors of the Thiosulphate and Iodine solutions respectively.

SOLUTIONS

1. Iodine Solution - Same as in Schedule of Examination No. 6 on Determination of True Red Lead content in Litharge and Red Lead.

2. Sodium Thiosulphate - Same as in Schedule of Examination No. 6.

3. Starch Solution - Same as in Schedule of Examination [illegible]

4. Acetic Acid, Potassium Iodide, Sodium Acetate Solution
   300 grams tested Purity Crystallized Sodium Acetate
   24 grams C.P. Potassium Iodide
   150 c.c. Water
   50 c.c. Galacial Acetic Acid
   Warm gently and add 20 to 30 c.c. of water if necessary. If some of the Sodium Acetate crystallizes upon cooling it does not affect the result.

INTERFERRING SUBSTANCES - Other materials that liberate Iodine from Potassium Iodide, such as Chromates interfere with this determination but would not ordinarily be

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a railroad paint of this type. Small amounts of unextracted oil does not interfere with this determination.

This method gives the True Red Lead content, Pb_3O_4, only. For instance, if the red lead pigment used contains 93% True Red Lead, then this method determines only the 93% which is Pb_3O_4 and does not include the other 8% which is Litharge, PbO. To calculate the total number of pounds of such Red Lead pigment used, (such as 302 dry), it is thus necessary to divide the weight of True Red Lead determined as above by .93 or whatever is the true Pb_3O_4 content of the Red Lead pigment used.

II

ALTERNATE METHOD - Weigh out exactly two grams Ferrous Ammonium Sulfate, transfer to beaker, add 25cc dilute HCl and 75 cc water, and stir till dissolved. Then add exactly 1 gram of pigment (or a larger amount if only a small percentage of Pb_3O_4 is present), boil for a few minutes, cool and titrate with N/5 $K_2Cr_2O_7$, using Potassium Ferricyanide as indicator. The number of cc required for a blank of 2 grams ferrous salt is determined.

Calculation:

$$\frac{\text{No. cc req'd. for blank} - \text{No. for sample} \times Pb_3O_4 \text{ factor of } K_2Cr_2O_7 \times 100}{\text{weight of sample}}$$

x per cent of Pb_3O_4

1 cc N/5 $K_2Cr_2O_7$ = 0.0685 g Pb_3O_4

This method gives the True Red Lead content, Pb_3O_4, only. For instance, if the Red Lead pigment used contains 93% True Red Lead, then this method determines only the 93% which is Pb_3O_4, and does not include the other 8% which is Litharge, PbO. To calculate the total number of pounds of such Red Lead pigment used, (such as 303 dry), it is thus necessary to divide the weight of True Red Lead determined as above by .93 or whatever is the true Pb_3O_4 content of the Red Lead pigment used.

SCHEDULE OF EXAMINATION No. 86

Issued by Paint
Research Division
Detroit, Michigan
July 15, 1932

STABILITY TEST LEADED ZINC AND OTHER BASIC LEAD SULPHATE PIGMENTS

GENERAL

This test should be regarded as tentative.

This test is based on the reaction which takes place when Basic Lead Sulphate pigments, such as Leaded Zincs and Sublimed White Lead, are rubbed up in Glycerine, producing a pinkish discoloration, more or less pronounced, depending on the particular pigment, grade of Glycerine, temperature and light conditions.

Some Basic Lead Sulphate pigments which develop this coloration rapidly show a similar tendency in certain vehicles when exposed in paste form to ultraviolet light, and much more slowly on exposure to sunlight in a paint film.

This "Glycerine test" is easily performed and serves to distinguish Basic Lead Sulphate pigments which are relatively light stable from those which are relatively unstable.

In order to avoid misleading results the test must be conducted under carefully controlled conditions and against a standard of proven stability in various vehicles under ultraviolet light.

GLYCERINE FOR TEST

Because of the sensitivity of this test and variations in different Glycerines, a stock of tested Glycerine will be kept at the laboratory of the Ozark Smelting & Mining Co., Coffeyville, Kansas, and supplied to the different laboratories on request.

TEST PROCEDURE

Four grams of Leaded Zinc or other Basic Lead Sulphate pigment is placed on a glass slab and 2-1/2 c.c. of Glycerine added. Thorough incorporation of the pigment with the Glycerine is effected with a spatula. A portion of this paste is spread on a piece of plate glass approximately 2" x 12" to form a smear the width of the slide and 2" in length (2" x 2" smear).

A space of 2" is then left on the slide and then a smear of the standard Leaded Zinc is put on. Up to this point it is essential that the operations be conducted in a dull light only, not bright daylight or direct sunlight. The Glycerine is to be used at normal room temperature. The plate carrying the smear is then placed in an oven and maintained at 80° C. for one hour. At the expiration of this time, remove from the oven and cool at room temperature for fifteen minutes.

#6

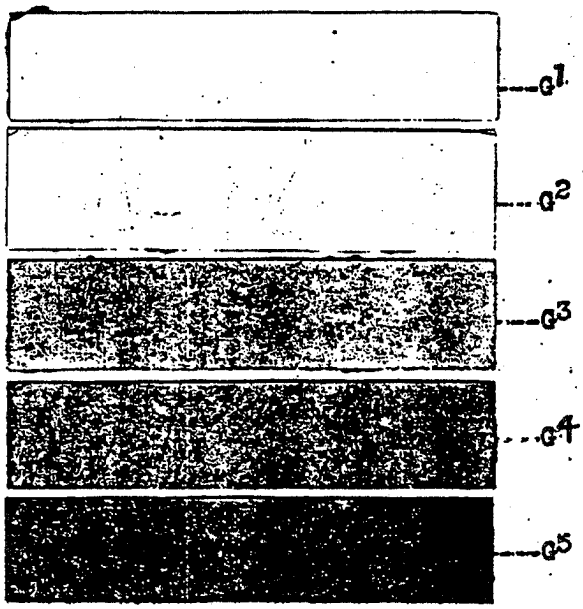
A fresh paste of pigment and glycerine is now to be made and laid down against the heated and cooled smear. This paste must be freshly made and not a portion of the first paste held over. Examination is then made for change in coloration of the particular pigment in the heating with Glycerine. Due consideration must be given the flatting effect of the heat on the paste, the change in the color only is considered.

Each pigment is examined against itself. The use of the standard is a check on the test and not on the sample under test. If properly conducted there will be no change (or at most a faint apparent darkening due to flatting of vehicle rather than of the pigment) with the standard pigment. The discoloration, if any, may be reported on a numerical system using the standard light sensitivity chart in estimating.

G^0 - No change
G^1 - Very faint darkening or pink
G^2 - Easily detectable darkening or pink
G^3 - Decided darkening or pink
G^4 - Considerable darkening or pink
G^5 - Salmon red or Yellow red

A test result of between G^0 and G^2 on 412 dry or 1526 dry appears to represent a satisfactory pigment for general usage. Stocks on hand manufactured prior to April 1932 may require a tolerance of G^3.

Unless the conditions above specified as to the degree of light permitted during the preparation of the sample are strictly adhered to, the results may be misleading.



SCHEDULE OF EXAMINATION No. 87

Issued by Paint Research
Division, Detroit, Mich.
January 4, 1933

TESTING OF DRY COLOR SHIPMENTS
(Federation of Paint & Varnish Production Clubs - revised 11-1 32)

APPARATUS & MATERIALS

1. Oil - White Refined Linseed Oil (Acid No. approximately 4).
2. Balance - Regulation Laboratory Balance.
3. Burette - Accurate one cubic centimeter burette, graduated in tenths of a cubic centimeter, or the apparatus known as the Brown Burette.
4. Muller - To have rubbing face of from 2-3/4" to 3". Face to be free from blowholes and flaws.
5. Rubbing Surface - To be lithographer's stone or Polished Plate Glass Slab, that has been ground to a satin finish with a muller and fine emery flour wet with kerosene.
6. Tinting and Strength Tests - (a) A standard Zinc Oxide paste in oil in tubes for colored pigments
   (b) A standard Ultramarine Blue paste in oil in tubes for white pigments.
7. Balanced watch glasses or counter balanced weighing pans.
8. Flexible spatula for mixing oil and pigments (3" blade).
9. French scraper or body glaze scraper - with true edge (3" to 4" wide).
10. Bright tin panels or clear glass slides (for exhibition of color daubs).

EXPLANATION.

1. Oil - White refined linseed oil with an acid number of approximately 4 is generally acceptable for white and colored pigments. Some pigments particularly earth colors have a slightly greater tendency to flood in refined oil than when mulled in raw linseed oil. The use of two rather similar oils would call for additional equipment and be the possible source of error, so for simplicity we suggest the general use of refined linseed oil. It is necessary therefore that all colors should be examined immediately after having been placed on slides. Oil should be changed frequently.

   The use of bodied oils or varnish is essential for some trades. This point is one that should be arranged for between the individual buyer and seller.

2. Balance - Should be the regulation laboratory balance, sensitive enough to be accurate to three decimal places. To be used in weighing dry pigment, oil and reduction or tinting pastes.

3. Burettes - Should be either standard one cubic centimeter burettes, graduated in tenths of a cubic centimeter, (pet cock controlled) or the apparatus known as the Brown Burette, which consists of a small reservoir to supply the burette. This burette is graduated in tenths of a cubic centimeter and is stop-cock controlled. The tips of all burettes should be ground optically flat. No droppers or weighing bottles should be used.

4. Mullers - Should be of the standard type on the market with a grinding face of from 2-3/4" to 3" diameter. The face should be free from blow-holes and other imperfections. The use of "weighted" mullers is not recommended.

5 Rubbing Surfaces - Should be lithographer's stones or Ground Plate Glass Slabs, the surface of which are kept sharp by sand-blasting when necessary. Different slabs or stones should be available for RED, YELLOW, BLUE GREEN, BLACK and WHITE if at all possible to guard against contamination.

6. (a) Tinting and Strength Tests should be made using (a) Zinc Oil Paste for colors. Recommended formulae for White Zinc Paste. Either

1. 30# Green Seal Zinc Oxide
64# Poppy Oil
2# Calcium Stearate
1# Turpentine

Ground Fine and placed in small collapsible tubes.

This formula yields a short paste, handy to use and which has little tendency to settle.

or 2 90# Green Seal Zinc Oxide
10# Refined Linseed Oil
1# 4 hour cooked Linseed Oil (560F)

Mixture placed in tubes.

This formula yields a paste, that is slightly stringier than #1 and settles very little.

Some manufacturers prefer the use of Zinc Oxide ground in "C" or "000" lithographic oil.

6 (b) Tinting & Strength Tests for white pigments should be made using (b) a blue paste in oil. Recommended formulae for the blue paste:- Either

1. 140# Ultramarine Blue — Ground fine and placed in small collapsible tubes.
   50# Poppy Oil
   3# Calcium Stearate
   2# Castile Soap
   1/2# Turpentine

This formula yields a short paste, handy to use and which has little tendency to settle.

or 2. 70# Ultramarine Blue
   27# Refined Linseed Oil
   3# 4 hour cooked Linseed Oil (560F)

This formula yields a paste, that is slightly stringier than #1 Blue formula.

When running the tinting tests or strength determinations dry blue or dry white pigment should not be added to a mulled sample under examination as this calls for additional mulling to incorporate the tinting pigment. The objections to this method of procedure are as follows:-

1st - A waste of time and energy
2nd - Additional mulling may develop color that is unobtainable in the plant.
3rd - This is contrary to general plant practise of tinting.

It is also considered a waste of time to weigh up and rub out portions of the contrasting dry pigments- for instance, a dry sample of a white shipment and dry blue. The white has already been rubbed up for shade and is available on the slab in paste form for tinting purposes. Then again the hardness of each pigment must be considered when dispersing color.

7. Balanced watch glasses or counter balanced weighing pans - should be used in weighing out the samples of dry colors, oils and pastes. For regular control work we recommend the use of the burette in measuring the oil used instead of the scale because of the simplicity, speed and tolerant accuracy of the burette. In case of doubt and dispute over any color, all portions should be weighed.

8. Flexible spatula for mixing oil and pigment should have a three inch blade.

9. French Scraper or Body Glazing Scraper, should be used in smoothing down the color daubs that are placed on the glass or tin, so that both shipment and standard are seen on the same plane. Scraper should have a blade that is about 3" or 4" wide with a good straight edge.

10. Bright Tin or Clear Glass Panels - should be used for observing color matches. If glass is used rub outs should be observed from the top and not through the glass for we are interested mainly in how colors look on top. Looking at the under side of rub-outs on a glass panel is useful if properly interpreted. It should not permit to modify ones judgement of a color as made from the top side.

METHOD TO BE FOLLOWED IN RUBBING OUT PIGMENTS.

Carefully weigh out sample of pigment to be tested and counter-balance with the standard sample. If oil is to be weighed, add oil to the dry samples accurately. Transfer the mass to the rubbing slab. If oil is to be measured, transfer weighed samples of the pigments to the slab and add oil by dropping (slowly) from the burette, working up paste with the small spatula all the while. Be sure to allow enough time for burette to drain to its true level.

When all dry pigment is worked up by means of spatula, the mass should then be mulled. The stoke should be from 3 to 4 inches wide, and from 12 to 15 inches in length. In counting rubs given a color, one stroke up and one stroke back is considered one rub - not two rubs. Muller should be allowed to traveler up one side and back the other side, twisting muller slightly at the top and bottom of each half stroke to help work the pigment in.

A pigment demanding			300	rubs	shall be	"picked-up"	after	each	50	rubs		
"	"	"	200	"	"	"	"	"	"	40	"	
"	"	"	100	"	"	"	"	"	"	25	"	
"	"	"	50	"	"	"	"	"	"	15	"	

By "picking-up" we mean that face of muller shall be scraped with the spatula and the paste on slab gathered up in a daub once again. Then mulling is started once more.

When mulling is completed, shipment and standard samples are placed in juxtaposition on a clean piece of bright tin or clear glass. Daub shall be about two inches long and at least an inch wide. The French Scraper shall be lightly drawn down over the pastes to even off the ridge and so present both daubs on an even plane. Mass tone should be judged immediately. Observation for floating, bronzing, etc., should be taken note of after 2 or 3 hours, if this fact is important to the consumer.

Tinting Value or Reduction should be run as follows:- Weigh up the required amount of the mulled paste from slab and counter-balance with the standard sample. Then add the correct amount of the "Reduction Paste" to shipment and counter-balance. Next thoroughly mix the pastes on the watch glass or weighing pan and transfer samples to the slab mixing them thoroughly with spatula only, till no more streaking is noticeable.

Transfer samples to the tin or glass, placing them in juxtaposition, daub being about 2 inches long and at least an inch wide. Smooth down with the French Scraper and judge color immediately.

Strength tests shall be made in a similar manner to the reduction tests except amounts of reduction pastes or mulled pastes of shipment are regulated as is necessary

PIGMENT RUB OUT SCHEME

Color or Class	Grams of Color	C.C. of Oil	Rubs or Rubs	Reduction Grams of Reduction Paste to Grams of Mulled Paste
C.P. Iron Blues	1.0	0.80	300	5 .. 1
C P. Light & Medium Para Toners	0.5	0 80	200	5 .. 1
C P. Dark Para Toners	0.6	0.80	300	5 .. 1
10% Para Toners on Whiting	1.0	0.50	50	2 .. 1
C.P. Toluidine	0.5	0 50	150	5 .. 1
C.P. Light Chrome Green	1 0	0.40	100	2 .. 1
C P Med. Chrome Green	1.0	0 50	150	2 .. 1
C P. Deep Chrome Green	1.0	0 60	200	2 .. 1
35% Grinder's Green Light	1.0	0 30	50	2 .. 1
25% Grinder's Green Medium	1.0	0.40	50	2 .. 1
25% Grinder's Green Deep	1.0	0.40	75	2 .. 1
C.P. Chromium Oxide	2.0	0 40	50	2 .. 1
C P. Primrose Chrome Yellow	1.0	0 20	50	2 .. 1
C P. Light Chrome Yellow	1.0	0.50	50	2 .. 1
C.P. Med. Chrome Yellow	1.0	0.30	50	2 .. 1
C.P. Park Chrome Yellow	2.0	0.40	50	2 .. 1
C P. Zinc Yellow	1.0	0.40	50	2 .. 1
English Vermil.	2.0	0.50	100	2 .. 1
Ultramarine Blue	1.0	0.50	150	2 .. 1
Cobalt Blue	1.0	0.50	150	2 .. 1
High Strength Carbon Black	0.3	2.80	150	2 .. 1
Bone Black	1.0	0.80	100	2 .. 1

SCHEDULE OF EXAMINATION No. 87..... SHEET 6

Color or Class	Grams of Color	C.C. of Oil	Mulls or Rubs		Reduction-Grams of Reduction Paste to Grams of Mulled Paste
Lamp Black	0.3	1.00	100		2 - .1
Carbon Black	0.3	1.30	150		2 - .1
Ferrite Yellow	0.5	0.30	150		2 - .1
French Yellow Ochre	1.0	0.50	150		2 - .1
Indian, Spanish, Venetian, Persian and Light Red Oxides	2.0	0.60	150		2 - .1
Raw & Bt. Sienna	1.0	0.60	200		2 - .1
Raw & Bt. Umber	1.0	0.80	200		2 - .1
White Leads	2.0	0.40	100	For whites use	.1 - 4
Zinc Oxides	2.0	0.60	100	Ultramarine Blue	.1 - 4
35% Leaded Zinc	2.0	0.40	100	Reduction Paste	.1 - 4
Titanox	2.0	0.50	100		.1 - 4
Titanium Dioxide	2.0	0.90	100		.1 - 2
Cryptone	2.0	0.50	100		.1 - 4
Lithopones	2.0	0.50	100		.1 - 4
Zinc Sulphide	2.0	0.60	100		.1 - 2

SCHEDULE OF EXAMINATION No. 91

Issued by Paint
Research Division
Detroit, Michigan
June 15, 1934

DRYING QUALITIES OF SEMI-DRYING OILS

1. STATEMENT

Because of lack of constant temperature, constant humidity and constant illumination facilities, drying tests - especially if pigmented are made on a comparative basis using a standard oil. Drying tests made by pouring the semi-drying oil on a vertical strip of tin results in such thin films that important differences which would occur in a pigmented product, are difficult to evaluate.

In order to spread the drying interval so that differences in drying qualities become more noticeable, a pigment was selected which retards normal drying, viz: Ferrite Yellow Light, having no calcium or soluble iron sulphate salts.

2. MATERIALS

(a) - Soybean Oil standard. Preserved from contamination and oxidation.

(b) - Ferrite Yellow (Light) Standard. Free from calcium compounds. Contains 86.5% Fe_2O_3 equivalent and not more than 0.002% soluble iron salts or 1.0% (SO_3).

(c) - Ground glass plate or stone slab.

(d) - Glass muller having a face of approximately 3 inches diameter and weighing about 1-3/4 lbs.

(e) - Burette measuring to tenths of cc's.

(f) - Tin strips 5-1/2" x 3".

(g) - Steel spatula.

(h) - Prepared standard drier solution, 5 gallons of which has the equivalent active metal Lead and Manganese content of 2 gallons (31).

3. PREPARATION OF DRIER

Any desired portion of the following 5 gallon drier formula can be accurately made up and preserved in small, tightly closed glass containers, kept in the dark:

1.55 lb. of metal Lead equivalent, obtained from Soligen or Nuodex Lead Solutions.
0.051 lbs. of metal Manganese equivalent, obtained from Soligen or Nuodex Manganese solution.
Turpentine - balance needed to complete formula on 5 gallon basis.

4. PROPORTIONS OF DRIER AND OIL

Five volumes - measured from a burette - of above drier are thoroughly mixed with 95 volumes of the oil to be evaluated, immediately before incorporation with the pigment.

5. GRINDING PROCEDURE

13 cc of the Oil-Drier mixture are added to 5 grams of Ferrite Yellow Light and mulled 25 times with only a gentle pressure.

6. PAINT-OUT AND DRYING PROCEDURE

The paint prepared as above is spread on a cleaned sheet of tin 5-1/2" x 3" and spun on a rapidly rotating disc in such a manner as to produce a film thickness of 0.002 inch. If facilities for spinning are not available then brush out to approximately the specified thickness.

Place the standard panel prepared as above which serves as the standard for comparison of drying along with the prepared panels of the oils to be evaluated in a well lighted, well ventilated place, free from excessive dust and note the time required to "dry to touch" and subsequent "tack" on further drying.