BATES LABELS 0007-SWP-043600 THROUGH 0007-SWP-043601

NOT USED IN THIS PRODUCTION

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 in 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 with a spatula on a 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 Glycerine and the pigment is made. Up to this point it is essential that the operations be conducted in daylight 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.

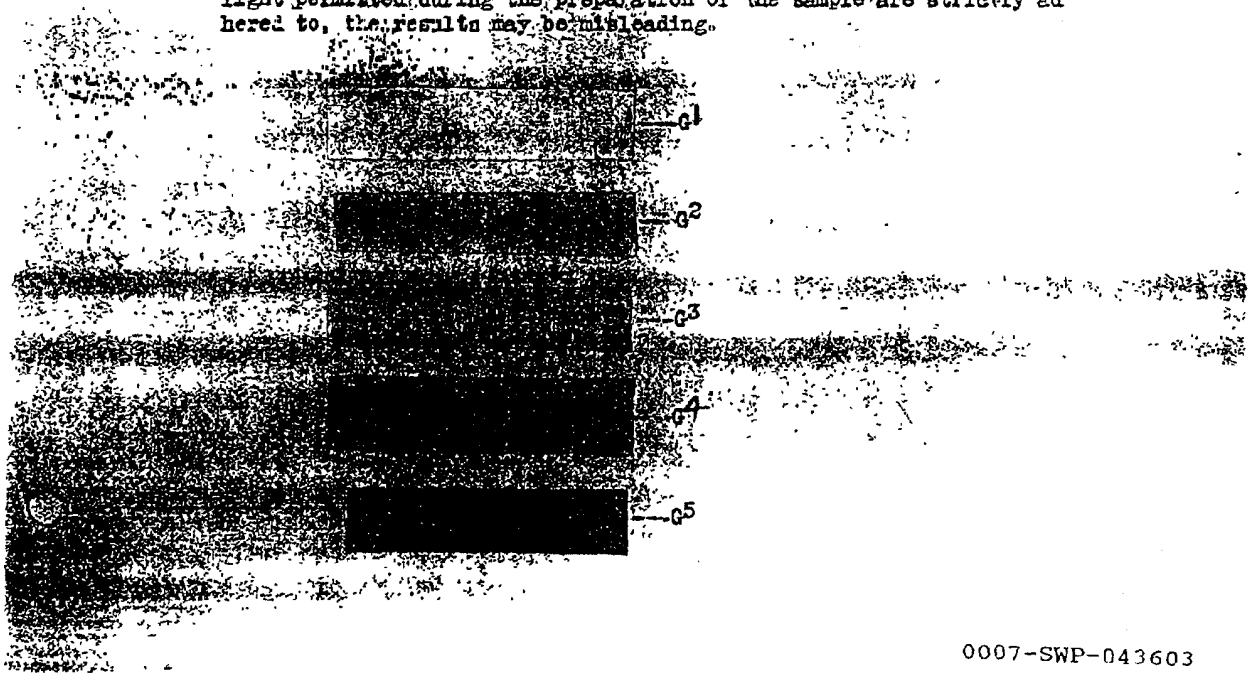
A fresh paste of pigment and glycerine is now to be made and laid down against the heated and cooled smear This paste must be freshly made and not a portion of the first paste held over Examination is then made for change in coloration of the particular pigment in the heating with Glycerine Due consideration 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 4.2 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.




0007-SWP-043603

SCHEDULE OF EXAMINATION No. 42

Issued by Cleveland
Technical Service Dept.,
November 21, 1924.

## ANALYSIS OF WHITE PAINTS (PIGMENT PORTION)

(This Schedule is derived from Chapters XVI and XVII of Dr. Holley's book, "Analysis of Paint and Varnish Products", edition of 1912)

### 1. QUALITATIVE ANALYSIS

In the majority of cases a complete qualitative analysis of the pigments present is hardly worth the time it requires, as there is but little time lost in following the regular quantitative scheme. If, however, a qualitative analysis is desired, the following outline will be found sufficient in most instances, the removal of the vehicle previous to these tests being understood. (See Schedule of Examination No. 41)

**Carbonates**

Effervescence with concentrated hydrochloric acid indicates carbonates, or hydrogen sulphide if zinc sulphide be present, the latter being distinguished by its odor and by the fumes blackening a piece of filter paper moistened with lead acetate.

**Barytes, silica, clay, or other silicates**

Boil above mixture five minutes, dilute with boiling water, filter. An insoluble residue may be barytes, silica, clay, or other silicates. Test for barytes with flame test, using a platinum wire. A characteristic green color indicates barium.

**Sulphates**

Test a small portion of the acid filtrate for combined sulphuric acid with a few drops of barium chloride.

**Lead**

Test another small portion of the acid filtrate with sulphuric acid. A white precipitate at once or on standing indicates lead.

**Zinc**

Take another small portion of the acid filtrate and add a few drops of potassium ferrocyanide. A white precipitate with a bluish tinge indicates zinc.

Calcium

The remaining portion of the acid filtrate is made alkaline with ammonia and hydrogen sulphide passed in for five minutes. Filter and test filtrate for calcium with ammonium oxalate, setting aside in a warm place.

Magnesium

After completely precipitating the calcium add a few drops of hydrogen sodium phosphate. A precipitate on standing indicates the presence of magnesium compounds.

The identification of the forms in which the lead may occur can only be determined by the quantitative scheme if both sulphates and carbonates are present.

## QUANTITATIVE ANALYSIS OF WHITE PAINTS

### 2. TOTAL LEAD

Weigh 1 gram of the dry pigment into a 250 c.c. beaker. Add 30 c.c. of strong hydrochloric acid, boil 5 minutes, add 50 c.c. of hot water, heat 15 minutes longer, settle, filter while hot, and wash thoroughly with boiling water. The washing should be begun the instant the solution has filtered through, in order to avoid any crystallization of lead chlorids in the pores of the filter paper. Once formed the crystals can only be dissolved with difficulty and with the use of an excess of wash water, which, as stated, must be at boiling temperature. This operation is best conducted by the aid of suction. Casein and other products of a similar nature are occasionally used in the manufacture of mixed paints in considerable quantities, and the analyst should always be on the lookout for the possible presence of these substances.

The solution is made just alkaline with ammonia, then just acid to litmus with hydrochloric acid. It is very necessary that the solution be only barely acid, as a comparatively small quantity of free acid will keep considerable lead from precipitating. Having been made barely alkaline, which is indicated by the precipitation of the lead, the solution is brought to a faintly acid condition by using dilute hydrochloric acid (1 to 19). Dilute to about 350 c.c. Cool, pass in hydrogen sulphide gas, noting the color of the precipitate: if gray, some zinc is being thrown down; if reddish black, the solution is too acid. Add a few drops of dilute acid or ammonia as the case requires. Settle, filter, and wash with cold water.

Place filter and precipitate in 25 c.c. of nitric acid and 25 c.c. of water, heat gently until the lead has all dissolved, as shown by the residual sulphur having a yellow appearance. Do not boil hard enough thoroughly to disintegrate the filter paper. Difficulty is experienced in dissolving the lead contained in the sulphur precipitate. It is better to collect them into a ball with the aid of a stirring rod and remove to a small beaker and treat with a few cubic centimeters concentrated nitric acid, and heat until dissolved, then pour back into the larger beaker.

Pour solution and filter paper on to a suction funnel provided with a platinum cone. If any fine particles pass through, pour the filtrate back again. This procedure permits the washing of the filter mass with a very small amount of water, thus saving considerable time in the subsequent evaporation. If the above apparatus is not available, filter through paper in the ordinary way, washing carefully. Add 5 c.c. of dilute sulphuric acid to filtrate, and evaporate until sulphur trioxide fumes appear. Cool, add 25 c.c. of water, 25 c.c. of alcohol; allow to stand one-half hour with occasional stirring; filter, using Gooch crucible, wash with dilute alcohol, dry, heat gently over ordinary lamp, and weigh as lead sulphate.

3. CALCIUM

The filtrate containing the zinc, calcium, and possibly magnesium is made slightly alkaline with ammonia, a few drops of a mercuric chloride solution (1 to 10) added, and a stream of hydrogen sulphide gas passed into the solution for about ten minutes.

The addition of the mercuric chloride renders the precipitate granular and very easy to filter, and entirely obviates the difficulty of filtering a slimy zinc sulphide precipitate. In the analysis of tints where the zinc cannot be titrated until it has been freed from iron, the addition of the mercuric chloride will not cause any trouble, as treatment with hydrochloric acid results in the solution of the zinc only, the mercuric sulphide being insoluble in hydrochloric acid. Settle, decant, filter, and wash.

Evaporate the filtrate from above precipitate to about 150 c.c., make alkaline with ammonia, add ammonium oxalate (50 c.c. for 1 gram of lime), usually 20 c.c. is sufficient, and set in a warm place for two or three hours. Filter, wash, ignite, and weigh as calcium oxide, or titrate precipitate with permanganate by placing filter and the thoroughly washed precipitate in a 400 c.c. beaker, adding 200 c.c. of boiling water and 25 c.c. of dilute sulphuric acid, and titrate with standard tenth-normal potassium permanganate. Filter paper must be removed from the solution before titrating.

1 c.c. tenth-normal permanganate = 0.0028 gram CaO
1 c.c. tenth-normal permanganate = 0.0050 gram $CaCO_3$.

Barium carbonate is still to be found in certain mixed paints, and it is advisable to test for the presence of soluble barium with a few drops of sulphuric acid before precipitating the calcium.

4. MAGNESIUM

The filtrate from the calcium oxalate should be tested for magnesium by treating with hydrogen sodium phosphate. Allow to stand one-half hour, add 25 c.c. of ammonia, allow to stand one hour, then filter on to a Gooch crucible, wash with dilute ammonia, ignite, and weigh.

Weight precipitate x 0.7575 = weight magnesium carbonate.

## 5. ZINC [redacted]

### Standard Zinc solution

Dissolve 10 grams of chemically pure zinc in hydrochloric acid in a graduated liter flask, add 50 grams of ammonium chloride, and make up to 1 liter.

1 c.c. = 0.01 gram zinc or 0.01245 gram zinc oxide.

### Standard potassium ferrocyanide solution

Dissolve 46 to 48 grams of crystallized potassium ferrocyanide in water, make to 1000 c.c.

### Uranium nitrate solution

Dissolve 15 grams of uranium nitrate in 100 c.c. of water.

### Standardizing the ferrocyanide solution

To determine the value of the potassium ferrocyanide solution, pipette 25 c.c. of the zinc solution into a 400 c.c. beaker. Dilute somewhat and make faintly alkaline with ammonia, bring to a faintly acid condition with hydrochloric acid, and then add 5 c.c. excess of the concentrated acid, dilute to a total volume of about 200 c.c., heat to 90° C., and titrate as follows: Pour off about 10 c.c. of the zinc solution into a small beaker and set aside, run the ferrocyanide into the remainder from a burette, a few cubic centimeters at a time, [redacted] until a drop of the solution [redacted] in contact with a drop of the uranium nitrate solution on a porcelain plate [redacted] a distinct brownish color. Often the end point has been passed by quite a little.

[redacted] c.c. of zinc solution that has been reserved is now added and the [redacted] adding a drop of the solution [redacted] on the [redacted] of ferrocyanide solution. [redacted] little time [redacted] change color, so that the end point may have been passed slightly. [redacted] corrected for by making a comparison of the porcelain [redacted], having the test drops arranged in regular order and taking as the [redacted] showing a distinct brownish tinge. Having noted the number of cubic centimeters of ferrocyanide required for the titration of the [redacted] zinc solution, the value of 1 c.c. may be readily calculated.

### [redacted] of sample

[redacted] gram, if the zinc content [redacted] determined in section 3, is [redacted] hydrochloric acid and 10 c.c. of water, [redacted] described above for the standard zinc [redacted] titrate to exactly the same depth of color on the [redacted]. If the method is carefully carried out, the procedure being [redacted] in each determination, the results will be found satisfactorily

6. LEAD SULPHATE

Dissolve 0.5 gram in a slight excess of dilute hydrochloric acid. Dilute to 200 c.c. and add a piece of aluminum foil which about covers the bottom of the beaker. It is important that this be held at the bottom by a glass rod. Boil gently until the lead is precipitated. Completion of this is shown by the lead ceasing to coat or cling to the aluminum. Decant through a filter, pressing the lead sponge into a cake to free it from solution. Add to filtrate a slight excess of barium chloride solution, allow precipitate to settle for an hour on hot plate, filter, wash, ignite, and weigh as barium sulphate. Calculate to lead sulphate by multiplying by 1.3 as a factor, unless calcium sulphate is present, in which case it is advisable to make use of Thompson's separation.

In the absence of barium sulphate the combined sulphuric acid may be estimated by H. Mannhardt's method: Grind 1 gram of pigment with 1 gram of sodium carbonate very intimately in an agate mortar. Boil gently for ten minutes, the combined sulphuric acid, and in the case of colors containing chromates the chromic acid, will pass into solution and may be estimated in the filtrate in the usual manner. If necessary collect the insoluble portion on a filter, dry, detach, and triturate a second time.

7. BASIC CARBONATE OF LEAD (WHITE LEAD)

After deducting the amount of lead present in the pigment as sulphate of lead, calculate the rest of the lead as white lead by multiplying the remaining sulphate by 0.852, unless sublimed lead is suspected to be present, in which case the combined lead oxide must be taken into consideration.

8. INSOLUBLE RESIDUE

The insoluble residue from the original hydrochloric acid treatment may contain barytes, magnesium silicate, silica, and clay. Ignite filter paper and residue until white, weigh as total insoluble matter; grind in agate mortar with about 10 times its weight of sodium carbonate, fuse for 1 hour in a platinum crucible, and dissolve out in hot water.

Barium sulphate

The solution from the fusion is filtered. The residue consists of barium carbonate, magnesium carbonate, etc., and is washed with hot water. The filtrate and washings are saved. Pierce filter paper and wash precipitate into clean beaker with hot dilute hydrochloric acid; finish washing with hot water, heat to boiling, add 10 c.c. of dilute sulphuric acid to precipitate barium, filter, ignite, and weigh as barium sulphate.

Silica

The filtrate from the barium sulphate is added with care to the filtrate reserved in the preceding paragraph, making distinctly acid; evaporate to complete dryness, cool, add 15 c.c. of hydrochloric acid, heat to boiling, cool, settle, filter, ignite, and weigh as silica.

Alumina

The filtrate from the silica will contain all of the alumina except that which was dissolved in the original treatment with hydrochloric acid. This is quite constant, varying from .004 to .005 gram per gram of clay. The acid filtrates are made slightly alkaline with ammonia, and boiled until odor disappears. Settle, filter, wash, ignite, and weigh as alumina.

Weight alumina x 2.5372 = weight clay.
Weight clay x .4667 = weight of silica in clay.

Any difference greater than 5 per cent may be considered as free or added silica, according to Scott.

Calcium and Magnesium oxides

If qualitative test shows presence of magnesium in insoluble residue from the first hydrochloric acid treatment it was present probably as magnesium silicate. Treat filtrate from the aluminum hydroxide for calcium and magnesium oxides. Magnesium silicate contains 3 to 5 per cent combined water.

9. HYDROFLUORIC ACID TREATMENT

Instead of resorting to fusion with sodium carbonate, the insoluble residue, which should be weighed up in a clean platinum crucible, may be treated with several drops of pure concentrated hydrofluoric acid and sulphuric acid and heated gently on a sand bath under the hood, using only sufficient heat slowly to volatilize the silica and sulphuric acid. Dissolve out in water acidulated with hydrochloric acid. The residue, which is barium sulphate, is filtered off and estimated as such. The filtrate will contain any aluminum, calcium, and magnesium present which may be estimated and calculated as oxides as above described. The combined weight of the barium sulphate, alumina, calcium, and magnesium oxides subtracted from the weight of the insoluble residue used gives the weight of silica. This operation is much shorter than resorting to a fusion and equally as accurate.

10. WHITE CARBONATES AND SULPHATES

Occasionally paints are met with which contain calcium sulphate, calcium carbonate, sulphate of lead, and white lead (basic carbonate of lead), in which case it is necessary to make a separation of the calcium compounds, which may be effected by Thompson's method as follows:

To 1 gram of the sample are added 20 c.c. of a mixture of nine parts alcohol (95 per cent) and one part of concentrated nitric acid. Stir, and allow to stand 20 minutes. Decant on a filter and repeat the treatment with the acid-alcohol mixture four times, allowing it to stand [illegible] before decanting. The calcium carbonate will go into solution, while the calcium sulphate or gypsum remains undissolved. Add filter and contents to the residue remaining in the beaker; dissolve in hydrochloric acid with sufficient water to insure the solution of the calcium. Make alkaline with ammonia, pass in hydrogen sulphide for 10 minutes, boil, settle, filter. The filtrate and washings are concentrated to about 150 c.c. and the calcium precipitated with ammonium oxalate in the usual manner. The ignited precipitate is calculated to hydrated calcium sulphate.

11. ESTIMATION OF CARBON DIOXIDE IN THE PRESENCE OF ZINC SULPHIDE

This method, devised by Mannhardt, is often useful in determining complex mixtures of pigments. One gram of the pigment is ground for several minutes in a smooth glass mortar with an excess of bichromate of potash, using considerable pressure; the mixture is then placed in the carbon dioxide generator and the carbon dioxide liberated and estimated in the usual manner.

12. CALCULATIONS

The ignited precipitate of calcium oxide obtained from the portion insoluble in the acid-alcohol mixture is subtracted from the total calcium weighed as oxide; the remaining calcium oxide is calculated to calcium carbonate. The total carbon dioxide is determined in a portion of the sample, the portion due to the calcium carbonate is deducted from the total amount, and the remainder calculated to basic carbonate of lead. The combined sulphuric acid due to the sulphate of lime is deducted from the total combined sulphuric acid, and the remainder calculated to sulphate of lead.

Wt. calcium oxide x 3.0715 = hydrated calcium sulphate.
Wt. calcium oxide x 1.784 = calcium carbonate.
Wt. calcium carbonate x 0.440 = carbon dioxide.
Wt. carbon dioxide x 8.6068 = basic carbonate of lead.
Wt. of hydrated sulphate of lime x 0.4661 = combined sulphuric acid.
Wt. of combined sulphuric acid x 3.788 = sulphate of lead.

## ANALYSIS OF WHITE PAINTS ACCORDING TO THOMPSON
(J. Soc. Chem. Ind., June 30, 1896)

Schemes for the separation of the constituents from each other and into their proximate combinations depend on the constituents present, and we can treat this subject in no better way than by taking typical cases, which we now do.

13. SAMPLE 1 is a mixture of barytes, white lead, and zinc oxide.

Two 1-gram portions are weighed out. One is dissolved in acetic acid and filtered, the insoluble matter ignited and weighed as barytes, the lead in the soluble portion precipitated with bichromate of potash, weighed in Gooch crucible as chromate, and calculated to white lead.

The other portion is dissolved in dilute nitric acid, sulphuric acid added in excess, evaporation carried to fumes, water added, the zinc sulphate solution filtered from barytes and lead sulphate and precipitated directly as carbonate, filtered, ignited, and weighed as oxide.

14. SAMPLE 2 is a mixture of barytes and so-called sublimed white lead.

Weigh out three 1-gram portions. In one determine zinc oxide, as in case 1. Treat a second portion with boiling acetic acid, filter, determine lead in filtrate, and calculate to lead oxide. Treat third portion by boiling with acid ammonium acetate, filter, ignite, and weigh residue as barytes; determine total lead in filtrate, deduct from it the lead as oxide, and calculate the remainder to sulphate. Sublimed lead contains no hydrate of lead, and its relative whiteness is probably due to the oxide of lead being combined with the sulphate as basic sulphate. Its analysis should be reported in terms of sulphate of lead, oxide of lead, and oxide of zinc.

15. SAMPLE 3 is a mixture of barytes, sublimed lead, and white lead.

Determine barytes, zinc oxide, lead soluble in acetic acid and in ammonium acetate, as in case 2; also, determine carbonic acid, which calculate to white lead, deduct lead in white lead from the lead soluble in acetic acid, and calculate the remainder to lead oxide.

16. SAMPLE 4 is a mixture of barytes, white lead, and carbonate of lime.

Determine barytes and lead soluble in acetic acid (white lead), as in case 1. In filtrate from lead chromate precipitate lime as oxalate, weigh as sulphate, and calculate to carbonate. Chromic acid does not interfere with the precipitation of lime as oxalate from acetic acid solution.

17. SAMPLE 5 is a mixture of barytes, white lead, zinc oxide, and carbonate of lime.

Determine barytes and white lead as in case 1. Dissolve another portion in acetic acid, filter, and pass sulphureted hydrogen through the boiling solution, filter, and precipitate lime in filtrate as oxalate; dissolve mixed sulphides of lead and zinc in dilute nitric acid, evaporate to fumes with sulphuric acid, separate, and determine zinc oxide, as in case 1.

18. SAMPLE 6 is a mixture of barytes, white lead, sublimed lead, and carbonate of lime.

Determine barytes, lead soluble in acetic acid and in ammonium acetate, as in case 2, lime and zinc oxide, as in case 5, and carbonic acid. Calculate lime to carbonate of lime, deduct carbonic acid in it from total carbonic acid, calculate the remainder of it to white lead, deduct lead in white lead from lead soluble in acetic acid, and calculate the remainder to oxide of lead.

19. SAMPLE 7 contains as insoluble matter, barytes, china clay, and silica.

After igniting and weighing the insoluble matter, carbonate of soda is added to it and the mixture fused. The fused mass is treated with water and the insoluble portion filtered off and washed. This insoluble portion is dissolved in dilute hydrochloric acid, and the barium present precipitated with sulphuric acid in excess. The barium sulphate is filtered out, ignited, weighed, and if this weight does not differ materially - say by 2 per cent - from the weight of the total insoluble matter, the total insoluble matter is reported as barytes. If the difference is greater than this, add the filtrate from the

SUPPLEMENT TO SCHEDULE OF EXAMINATION NO. 42

Issued by Paint Research
Division - Detroit
November 25, 1927

## ANALYSIS OF WHITE PAINTS (PIGMENT PORTION)

TITANIUM OXIDE - (Qualitative analysis) - [illegible] small amount of the dried pigment with [illegible] cool, dilute with water, and filter. To 5 or 10 c.c. of the filtrate in a test tube add 2 c.c. hydrogen peroxide. A distinct yellow coloration indicates the presence of Titanox. Nothing found in a White Paint interferes with this test. The yellow color increases with the amount of Titanium present and is not altered by either an excess of hydrogen peroxide or sulphuric acid. However, the amount of sulphuric acid present must be at least 5% by volume. It is well to test out the hydrogen peroxide used in making this test on a sample known to contain Titanium (say 10% dry), as inaccurate results are obtained in the presence of hydrofluoric acid, consequently it is not permissible to use hydrogen peroxide for this test which has been prepared from barium peroxide by means of hydrofluosilicic acid. Furthermore, chromic, vanadic and molybdic acids must not be present since they also give colorations with hydrogen peroxide. The presence of small amounts of iron does not affect the reaction - any more than a trace of iron would not be present in a white pigment. If, however, phosphoric acid is added to the ferric solution it becomes decolorized and from such solutions the determination of the presence of Titanium offers no difficulties. This reaction is delicate to 0.00005 grams of Titanium Oxide present as sulphate in 50 cc. of solution.

QUANTITATIVE DETERMINATION OF TITANIUM OXIDE - Transfer [illegible] dried sample to a 250 c.c. Pyrex beaker, add 30 [illegible] concentrated sulphuric acid and 7 to 8 grams of ammonium sulphate. Mix well and heat on hot plate until fumes of sulphuric acid are evolved, and then continue the heating over a strong flame until solution is complete (usually requires not over 5 minutes) or it is apparent that the residue is [illegible] siliceous matter and barium sulphate. Cool the [illegible] with 100 c.c. water, (caution), stir, heat [illegible] fully to boiling while stirring, let settle, filter through [illegible] transfer the precipitate completely to the paper [illegible] with cold 5% (by volume) sulphuric acid [illegible].

Dilute the filtrate to 200 c.c. and add about [illegible] hydroxide (sp. gr. 0.90) to [illegible] acidity by 5% (by volume) sulphuric acid.

Wash out a Jones [illegible] sulphuric acid and water, [illegible]

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the reductor to fill the upper level of the zinc. (These washings should require not more than one or two drops of 0.1N permanganate to obtain a pink color). Empty the receiver and put in it 25 c.c. (measured in a graduate) of ferric sulphate solution. (See reagents). Reduce the prepared Titanium solution as follows:

(1)- Run 50 c.c. of 5% sulphuric acid through the reductor at a speed of about 100 c.c. per minute.

(2)- Follow this with the Titanium solution

(3)- Wash out with 100 c.c. 5% sulphuric acid.

(4)- Finally run through about 100 c.c. of water. Care should be taken that the reductor is always filled with solution or water to the upper level of the zinc. Gradually release the suction, wash thoroughly the glass tube that was immersed in the ferric sulphate solution, remove the receiver, and titrate immediately with 0.1N potassium permanganate solution. One c.c. 0.1N potassium permanganate equals 0.00481 grams Titanium or 0.00801 grams Titanium Oxide. Run a blank determination, using the same reagents, washing the reductor as in the above determination. Subtract this permanganate reading from the original reading and calculate the final reading to Titanium dioxide. ($TiO_2$). This will include iron, chromium, arsenic, and any other substances which is reduced by zinc and acid but none of these would be present except as a trace in a white pigment. The most common Titanium pigment is a composite pigment containing 25% Titanium Oxide and 75% barium sulphate.

PREPARATION OF FERRIC SULPHATE SOLUTION - A solution containing two (2) per cent iron as ferric sulphate is desired and it may be prepared as follows:

Dissolve 20 grams pure iron or plain carbon steel wire in [illegible] excess of hydrochloric acid, oxidized with [illegible] acid. Add about 80 c.c. of sulphuric acid and heat until sulphur trioxide fumes are evolved. [illegible] dilute with water to 1000 c.c., digest on a steam bath until sulphates are dissolved, and filter if necessary. [illegible] potassium permanganate until a faint pink color [illegible] minutes. (to oxidize any ferrous iron [illegible] may be present).

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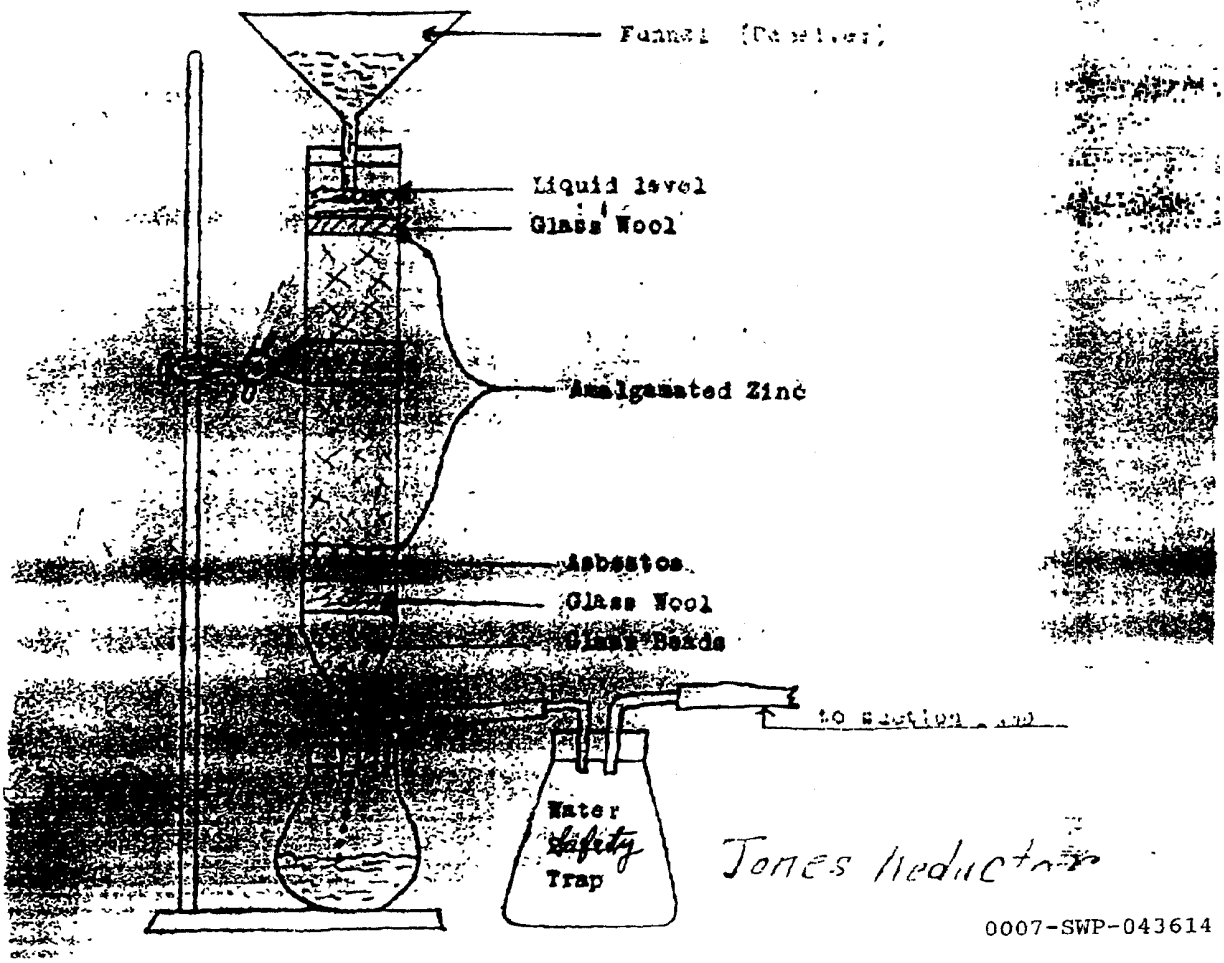
JONES REDUCTOR - The Jones Reductor (see fig.) is made by placing a platinum spiral (or glass beads) in the bottom of a glass stoppered tube which is 30 cm. long and has an inside diameter of about 18 mm. Upon the spiral, or glass beads, is placed a plug of glass wool and then a thin layer of asbestos such as is used for Gooch crucibles. The tube is then filled with amalgamated zinc to within 5 cm. of the top. The zinc can be prepared by shaking 20 to 30 mesh zinc, cleaning it with a little hydrochloric acid, and then covering with mercuric chloride until hydrogen is no longer evolved. In this condition the zinc is scarcely acted upon at all by hydrochloric acid, but is capable of reducing an iron or Titanium solution just as effectively as if it were not amalgamated. On top of the column of zinc is placed a little glass wool to serve as a filter.




Jones Reductor

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SEPARATION OF ZINC OXIDE FROM ZINC SULPHIDE. - With pigments containing Zinc Oxide and Zinc Sulphide the zinc oxide may be determined as follows:

Weigh accurately 2.5 grams of pigment, transfer to a 250 c.c. Erlenmeyer flask, moisten with a few drops of alcohol, add about 200 c.c. of 2% to 3% acetic acid, (20 c.c. glacial acetic in 1000 c.c. water), shake vigorously and let stand for 30 min. at room temperature, shaking once every five minutes. Then let stand at room temperature at least five hours, preferably overnight. Filter through a dry paper into a 250 c.c. graduated volumetric flask. Wash residue with 2% to 3% acetic acid and fill volumetric flask to mark with 2% to 3% acetic acid. Transfer 100 c.c. of the filtrate (corresponding to 1 gram) to a 400 c.c. beaker. To the clear solution add 30 c.c. of Hcl (1:3), 100 c.c. of water, and a small piece of litmus paper; add strong ammonium hydroxide until alkaline, render just acid with hydrochloric acid, then add 3 c.c. concentrated hydrochloric acid, heat nearly to boiling and titrate with potassium ferro-cyanide solution as in running total zinc. Calculate the percentage of zinc oxide (any zinc carbonate or zinc sulphate is included in the zinc oxide). Subtract this result from the percentage of total zinc as zinc oxide and calculate the difference to zinc sulphide.