

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 511

STANDARDS FOR PAINTS - WHITE LEAD

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PIGMENTS FOR PAINTS — WHITE LEAD ^{PIGMENT/1} FOR PAINTS

1. SCOPE AND FIELD OF APPLICATION

This International Standard specifies the requirements and the methods of test ^{corresponding} for white lead pigment.

2. REFERENCES Paints and varnishes —

- ISO 787, General methods of test for pigments
- ISO 842, Sampling raw materials for paints and varnishes

3. DEFINITION

White lead: A white pigment which consists of basic lead carbonate with a composition varying between the limits mentioned in section 4.

4. REQUIRED CHARACTERISTICS AND THEIR TOLERANCES

White lead should have the following characteristics: — in the table below contents in % (m/m)

Characteristic	Requirement	Test method
Lead carbonate content	64 to 80 % (m/m)	6.1
Lead hydroxide content	19 to 35 % (m/m)	6.4
Sum of lead carbonate and lead hydroxide/min.	99 % (m/m)	6.5
Matter volatile at 105 °C, max.	0,5 % (m/m)	ISO 787 Part II
Matter soluble in water, max.	0,5 % (m/m)	ISO 787 Part III
Alkalinity of aqueous extract, max.	0,2 ml of 0,1 N acid solution per gramme	ISO 787 Part IV
Residue on sieve, max.	0,5 % (m/m)	ISO 787 Part VI or VII
Colour	Equal to the agreed reference sample	ISO 787 Part I
Lightening power	As agreed between purchaser and vendor in the interested parties	ISO 787 Part VII
Hiding power		To be agreed between parties
Oil absorption value		ISO 787 Part VIII

to the interested

- For painting building interiors the White Lead (Painting) Convention, 1921 (No. 13) of the International Labour Organization prohibits the use of white lead and of all products containing it, except where the use of white lead is considered necessary for railway stations or industrial establishments by the competent authority, after consultation with the employers' and workers' organizations concerned.
- All percentages are calculated from the original sample.
- Two alternative methods for determination of residue on sieve are recommended in ISO 787, identified respectively as the "oil method" and the "water method". Both methods are acceptable for the purpose of this ISO recommendation but it is recommended that the method to be used in a particular case should be specified in the contract or otherwise agreed between the interested parties and should in any case be mentioned in the test report.

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5. SAMPLING

- 5.1 A representative sample of the pigment should be taken in accordance with ISO *8420*
Sampling raw materials for paints and varnishes.
- 5.2 The sample agreed between *the interested parties,* purchaser and vendor, to which reference is made in the *Table* shall comply with all the requirements specified for the pigment under test. *shall comply with all the requirements specified for the pigment under test.*

6. METHODS OF TEST

NOTE - All reagents used *shall* be of recognized analytical reagent quality. Distilled water or water of at least *equivalent* purity should be used. *Sn. 6*

6.1 Determination of lead carbonate (PbCO₃) content

The determination of *the* lead carbonate content is derived from the determination of carbon dioxide (CO₂) content.

6.1.1 Reagents

6.1.1.1 Nitric acid, 4 M. *addition*

6.1.1.2 Sulphuric acid, 96% (m/m), $\rho_{20} = 1,84 \text{ g/ml}$ *addition*

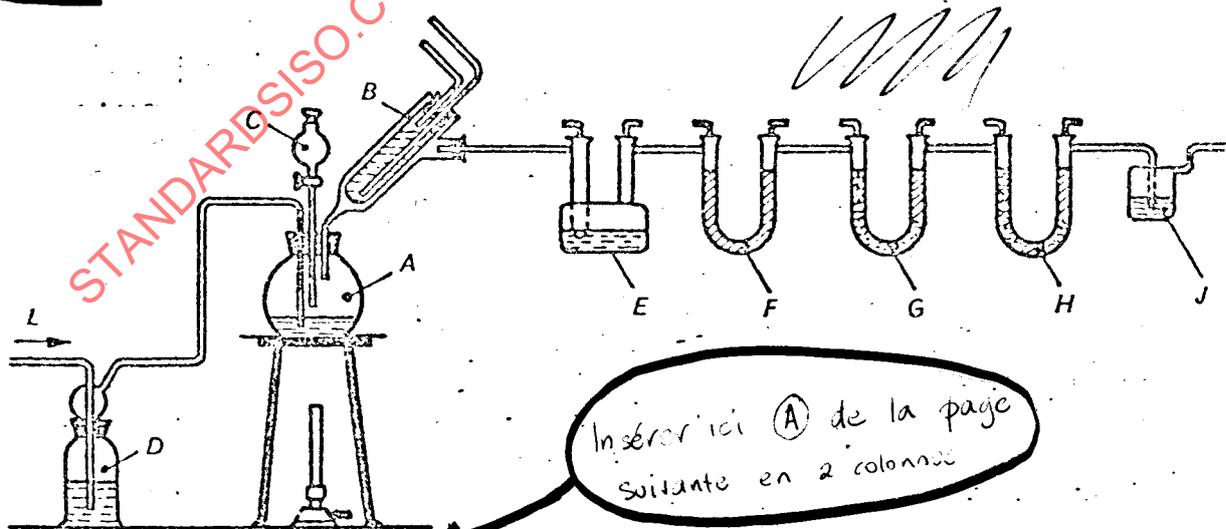
6.1.1.3 Potassium hydroxide, 33% (m/m) solution. } Dissolve 50 g of potassium hydroxide in 100 ml of water.

6.1.1.4 Magnesium perchlorate, anhydrous. *L. nov. 1966*

6.1.1.5 Indicating soda lime or soda asbestos.

6.1.2 Apparatus

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Use the apparatus *shown in the figure 2 below* for the determination of carbon dioxide (CO₂) *shown in the figure 2 below*.
Before making a determination, *shall be prepared* prepare the apparatus by slowly aspirating air and *should be performed* a blank test without any sample, following the procedure given in *figure 6.1.3.*
The apparatus should be regarded as satisfactory only if less than 1 mg of carbon dioxide is obtained, when the blank test is carried out.



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FIGURE 2 — Apparatus for the determination of the percentage of carbon dioxide (CO₂)

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- A Boiling flask with round bottom and short neck, nominal capacity 250 ml.
- B Short reflux condenser.
- C Stopped dropping funnel.
- D Wash-bottle containing potassium hydroxide solution (6.1.1.3).
- E Washing vessel containing sulphuric acid, (6.1.1.2).
- F U-tube containing a glass-wool plug to trap spray and filled with anhydrous magnesium perchlorate (6.1.1.4).
- G and H Two ground glass stoppered U-tubes filled with indicating soda lime or soda asbestos (6.1.1.5) (size 1,4 to 2 mm) in first two-thirds of space, and anhydrous magnesium perchlorate (6.1.1.4) in the remaining space (at the side where the gas stream passes out).
- J Indicating bubble counter containing concentrated sulphuric acid (6.1.1.2). (Alternatively, a third U-tube filled with anhydrous magnesium perchlorate in first third of space and soda asbestos in the remaining two-thirds may be used.)
- L Stream of air.

NOTE— The absorption tubes E to J, as well as the other components of the apparatus, need not necessarily be of the form illustrated in the figure.

6.1.3 Procedure

Weigh, to the nearest 1 mg, 0,5 to 1,0 g of white lead ~~(PbO)~~ into the flask A. Place 10 ml of water in the flask and assemble the apparatus, as shown in the figure. Turn the water on in the condenser B and draw air through the apparatus for 15 min. Close the taps in the absorption tubes G and H and remove the tubes. Wipe the tubes with a clean cloth and allow them to remain in the balance case for 30 min. Use a similar U-tube as a counterpoise and before weighing open all the taps momentarily to equalize the internal pressure. Reconnect the tubes to the apparatus after weighing. Pour 50 ml of the nitric acid into the dropping funnel C and allow it to run gradually into the flask A until the funnel is almost empty.

When the reaction has subsided, heat the flask, regulating the heat to give a steady flow through the washing vessel E. Allow the contents of the flask to boil slowly, aspirating a slow stream of air for a few minutes. Remove the source of heat and draw air through the apparatus at the rate of two to three bubbles per second for a further 20 min. Then, close the taps of the absorption tubes, disconnect the tubes and weigh them, using the same procedure as for the first weighing.

6.1.4 Expression of results

Calculate the lead carbonate (PbCO₃) content, a, of white lead ~~(PbO)~~ as a percentage by mass, by the following formula:

$$a = \frac{607,4 m_1^H}{m_0^H}$$

where

m_1^H is the increase in mass of the absorption tubes, in grams, and
 m_0^H is the mass, in grams, of the test portion.

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SUR 2 colonnes

6.2 Determination of total lead content by the chromate method 1) 7

6.2.1 Reagents

- 6.2.1.1 Nitric acid 4 M. *solution*
- 6.2.1.2 Acetic acid solution, 20 g/l.
- 6.2.1.3 Congo paper.
- 6.2.1.4 Ammonium acetate solution, 2 M, freshly prepared.
- 6.2.1.5 Potassium dichromate solution, 50 g/l.

6.2.2 Procedure

1/6.2.1.1)

Weigh, to the nearest 1 mg, 0,5 to 1,0 g of white lead, transfer to a conical flask of nominal capacity 500 ml and dissolve in 10 ml of the nitric acid if necessary by heating. Add to this solution as much of the ammonium acetate solution as is necessary until the liquid gives no acid reaction with the Congo paper (6.2.1.4)

Filter insoluble matter, if present, and wash thoroughly with the ammonium acetate solution.

1/6.2.1.5)

Dilute the filtrate combined with the washing with water to about 200 ml and then heat to boiling. Precipitate from the boiling liquid lead as lead chromate (PbCrO4) by adding an excess of the potassium dichromate solution. Keep the liquid boiling until the precipitate has turned dark orange-red and then keep heated (on the water-bath) for 1 1/2 to 2 hours. *continue heating*

1/6.2.1.2)

After cooling, filter the precipitate on a weighed sintered glass filter crucible of maximum pore diameter 5,25 μm, wash with the acetic acid solution, then with hot water and dry in a drying oven (100 ± 2 °C) or a vacuum desiccator to constant mass. *of porosity grade P4 or P10 (pore size index 4-16 μm)*

6.2.3 Expression of results

Calculate

The total lead (Pb) content of white lead (3), as a percentage by mass, by the following formula:

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d = (63,75 m3) / m2

where

- m3 mass, in grams, of the dried residue of lead chromate;
- m2 mass, in grams, of the test portion.

6.3 Determination of total lead content by the sulphate method 2) 7

6.3.1 Reagents

- 6.3.1.1 Hydrochloric acid 3 M. *solution*
- 6.3.1.2 Nitric acid 4 M. *solution*
- 6.3.1.3 Nitric acid 4 M, saturated with bromine. *solution*
- 6.3.1.4 Sulphuric acid 500 g/l. *solution*

1) This method should be used when the pigment is known to be free from adulteration.
 2) This method should be used when the purity of the pigment is unknown.

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- 6.3.1.5 Potassium hydroxide solution, 100 g/l.
- 6.3.1.6 Ammonium acetate solution, 335 g/l.
- 6.3.1.7 Sodium sulphide solution, 100 g/l.
- 6.3.1.8 Ethanol or denatured spirit, approximately 95% (V/V).
- 6.3.1.9 Hydrogen sulphide.

6.3.2 Procedure

6.3.2.1 Weigh to the nearest 1 mg, 0,5 g of white lead and transfer to a beaker of 400 ml nominal capacity. Add 10 ml of the nitric acid, cover the beaker and heat gently until all the white lead has dissolved. (6.3.1.2)

NOTE - Should the white lead be found to contain impurities, treat the contents of the beaker as follows:

↑ (6.3.1.1)
↓ (6.3.1.9)

Filter off any material remaining insoluble in nitric acid and wash the filter with hot water until free from soluble lead. Evaporate the filtrate to dryness. Add 2 ml of the hydrochloric acid, stir to mix and again evaporate to dryness on a water bath. Repeat this operation once more. Add a further 2 ml of the hydrochloric acid, followed by 200 ml of water. Boil the contents of the beaker to dissolve the lead chloride and pass hydrogen sulphide until cold. Filter the precipitate of lead sulphide on paper and wash with a saturated solution of hydrogen sulphide.

If antimony is present, wash the bulk of the precipitate back into the beaker and digest with 10 ml of the potassium hydroxide solution and 10 ml of the sodium sulphide solution for 10 min without boiling. (6.3.1.5) (6.3.1.7)

Again filter the lead sulphide onto the same paper and wash with sodium sulphide solution diluted with 10 times its volume of water. Pierce the paper with a pointed glass rod and wash as much as possible of the lead sulphide down into the original beaker. Then dissolve the remaining lead sulphide from the paper with the nitric acid saturated with bromine and warm the beaker to dissolve all the lead sulphide into solution. (6.3.1.4) (6.3.1.3)

6.3.2.2 Add 20 ml of the sulphuric acid to the solution and then evaporate gently without boiling, until copious fumes are evolved. To the contents of the beaker add 100 ml of water followed by 100 ml of ethanol or denatured spirit and allow to stand for 2 hours. (6.3.1.8)

Transfer the precipitate to a weighed Gooch crucible packed with asbestos (alternatively, a sintered silica crucible of porosity grade P4 or P10, i.e. pore size index 4-16 μm, may be used) and wash with ethanol or denatured spirit. Heat the crucible, gently at first and then to 500 °C for 10 min, cool and weigh. (6.3.1.6)

Pour hot ammonium acetate solution onto the filter to extract the lead sulphate completely. Wash the residue with hot distilled water, dry, heat to 500 °C for 10 min, cool and reweigh. The difference between these two weighings is the mass of lead sulphate.

6.3.3 Expression of results

Calculate the total lead (Pb) content of white lead as a percentage by mass, by the following formula:

(b)

$$z = \frac{68,32 (m_5 - m_6)}{m_4}$$

where

- m_5 = mass, in grams, of the first precipitate;
- m_6 = mass, in grams, of the residue after extraction with ammonium acetate; and
- m_4 = mass, in grams, of the test portion.

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