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R 511

PIGMENTS FOR PAINTS —WHITE LEAD

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[illegible]

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INTERNATIONAL STANDARD ISO ~~R~~ 511-1973(E)

PIGMENTS FOR PAINTS —WHITE LEAD ~~FOR PAINTS~~ ~~PIGMENT~~

1. SCOPE AND FIELD OF APPLICATION

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

2. REFERENCES

ISO 787, Paints and varnishes – 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~~ specified in section 4.[1]

4. REQUIRED CHARACTERISTICS AND THEIR TOLERANCES[2]

White lead shall ~~should~~ have the ~~following~~ characteristics shown 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 gram ~~es~~	ISO 787 Part IV
Residue on sieve, max.	0,5 % (m/m)	ISO 787 ~~Part 6 or 7~~ Part VI or VII[3]
Colour	Equal to the agreed reference sample	ISO 787 Part I
Lightening power	As agreed between ~~purchaser and vendor~~ the interested parties	ISO 787 Part VII
Hiding power		To be agreed between parties
Oil absorption value		ISO 787 Part V

the interested

2 x content

Sor +2 pica

VI (Part 6)

VII (Part 7)

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[1] 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.

[2] All percentages are calculated from the original sample.

[3] Two alternative methods for the 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.

International Standard

ISO 511-1973 (E)

Texte sur 2 colonnes

5 SAMPLING

5.1 A representative sample of the pigment ~~should be~~ shall be taken in accordance with ISO 842. ~~*Sampling raw materials for paints and varnishes.*~~

5.2 The sample agreed between ~~purchaser and vendor,~~ the interested parties, to which reference is made in the Table ~~should~~ shall comply with all the requirements specified for the pigment under test.

6 METHODS OF TEST

C8 NOTE – All reagents used ~~should~~ shall be of recognized analytical reagent quality. Distilled water or water of at least ~~equal~~ equivalent purity ~~should~~ shall be used.

6.1 Determination of lead carbonate ($PbCO_3$) content

The determination of the lead carbonate content is derived from the determination of carbon dioxide (CO_2) content.

6.1.1 *Reagents*

6.1.1.1 *Nitric acid*, solution, 4 M.

6.1.1.2 *Sulphuric acid*, solution, 96 % (m/m), ρ_{20} = 1,84 g/ml.

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

6.1.1.4 *Magnesium perchlorate, anhydrous.*

6.1.1.5 *Indicating soda lime or soda asbestos.*

6.1.2 *Apparatus*

bon

~~Use~~ The apparatus ~~shown in the figure below shall be used~~ for the determination of carbon dioxide (CO_2) ~~shown in the figure below.~~

Before making a determination, ~~prepare~~ the apparatus ~~shall be prepared~~ by slowly aspirating air and ~~perform~~ a blank test ~~shall be performed~~ without any sample, following the procedure given in ~~clause~~ 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.

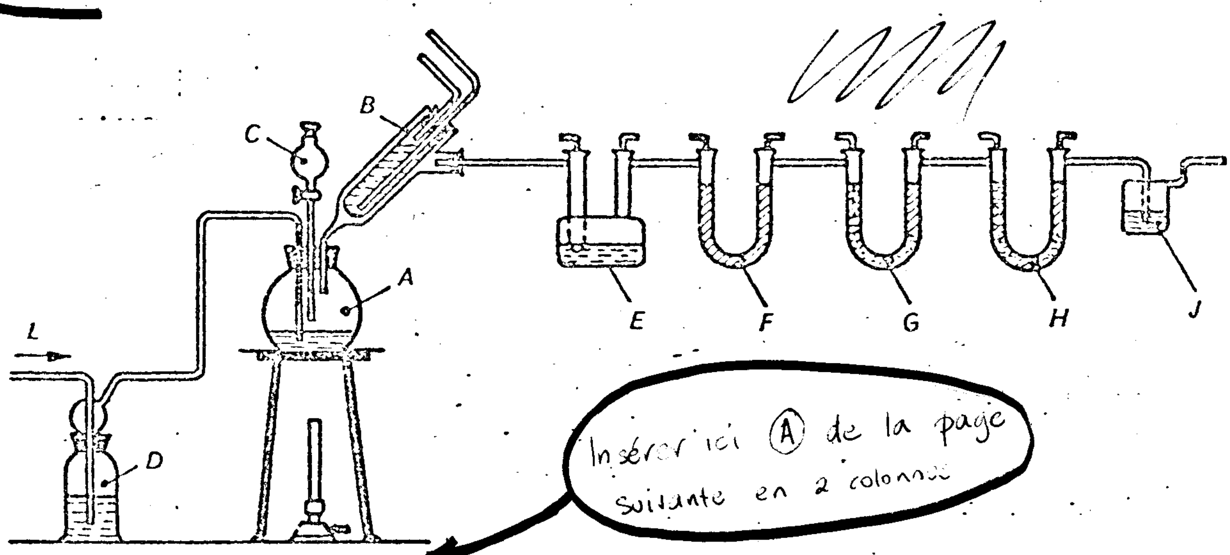

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FIGURE – Apparatus for the determination of the percentage of carbon dioxide (CO_2)

mettre figure en pied

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C8

A

à la page précédente

A	Boiling flask with round bottom and short neck, nominal capacity 250 ml.
B	Short reflux condenser.
C	Stoppered 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.

8 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.

sur 2 colonnes

6.1.3 *Procedure*

b.c.

Weigh, to the nearest 1 mg, 0,5 to 1,0 g of white lead 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 (6.1.1.1) 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.

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~~Calculation and~~

6.1.4 *Expression of results*

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~~Calculate~~ The lead carbonate (PbCO$_3$) content, a, of white lead as a percentage by mass, ~~as given~~ by the following formula:

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

where

m_1 is the increase in mass of the absorption tubes, in grams;

m_0 is the mass, in grams, of the test portion.

sur 2 colonnes

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

6.2.1 *Reagents*

6.2.1.1 *Nitric acid* solution, 4 M.

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*

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 (6.2.1.1), if necessary by heating. Add to this solution as much of the ammonium acetate solution (6.2.1.4) as is necessary until the liquid gives no acid reaction with the Congo paper (6.2.1.3).

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

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 ($PbCrO_4$), by adding an excess of the potassium dichromate solution (6.2.1.5). Keep the liquid boiling until the precipitate has turned dark orange-red and ~~then keep heated~~ continue heating (on the water-bath) for 1½ to 2 h.

After cooling, filter the precipitate on a weighed sintered glass filter crucible ~~of maximum pore diameter 5 to 15 µm~~ of porosity grade P4 or P16 (pore size index 4-16 µm), wash with the acetic acid solution (6.2.1.2), then with hot water and dry in a drying oven at 100 ± 2 °C or in a vacuum desiccator to constant mass.

6.2.3 *Expression of results*

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

$$b = \frac{63{,}75\ m_3}{m_2}$$

where

b is the

m_3 is the mass, in grams, of the dried residue of lead chromate;

m_2 is the mass, in grams, of the test portion.

bon

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

6.3.1 *Reagents*

6.3.1.1 *Hydrochloric acid* solution, 3 M.

6.3.1.2 *Nitric acid* solution, 4 M.

6.3.1.3 *Nitric acid* solution, 4 M, saturated with bromine.

6.3.1.4 *Sulphuric acid* solution, 500 g/l.

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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.

bon