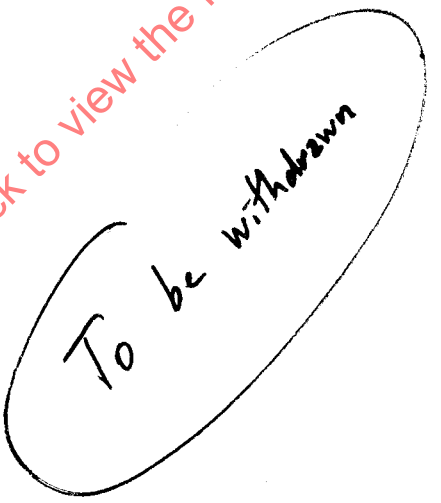
INTERNATIONAL STANDARD 511

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

White lead pigments for paints

Céruse pour peintures

First edition — 1974-07-01



UDC 667.622 **Ref. No. ISO 511-1974 (E)**

Descriptors : paints, pigments, white lead, materials specifications, chemical composition, tests, determination of content, lead carbonates.

Price based on 4 pages

FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up has the right to be represented on that Committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

Prior to 1972, the results of the work of the Technical Committees were published as ISO Recommendations; these documents are now in the process of being transformed into International Standards. As part of this process, International Standard ISO 511 replaces ISO Recommendation R 511-1966 drawn up by Technical Committee ISO/TC 35, *Paints and varnishes.*

The Member Bodies of the following countries approved the Recommendation :

Argentina	Denmark	Portugal
Australia	Egypt, Arab Rep. of	Spain
Austria	France	Sweden
Belgium	Germany	Turkey
Bulgaria	India	United Kingdom
Canada	Italy	U.S.S.R.
Chile	Japan	Yugoslavia
Colombia	Morocco	
Czechoslovakia	Netherlands	

No Member Body expressed disapproval of the Recommendation.

Printed in Switzerland

White lead pigments for paints

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies the requirements and corresponding test methods for white lead pigments suitable for use in paints.

2 REFERENCES

ISO 787, *General methods of test for pigments*

ISO 842, *Raw materials for paints and varnishes — Sampling.*

3 DESCRIPTION

White lead is a white pigment which consists of basic lead carbonate with a composition varying between the limits specified in clause 4.[1)]

4 REQUIRED CHARACTERISTICS AND THEIR TOLERANCES[2)]

White lead shall have the characteristics shown in the table below.

5 SAMPLING

5.1 A representative sample of the pigment shall be taken in accordance with ISO 842.

5.2 The sample agreed between the interested parties, to which reference is made in the table, shall comply with all the requirements specified for the pigment under test.

TABLE — **Required characteristics and their tolerances**

Characteristic		Requirement	Test method
Lead carbonate content	% (*m*/*m*)	64 to 80	clause 6
Lead hydroxide content	% (*m*/*m*)	19 to 35	clause 9
Sum of lead carbonate content and lead hydroxide content	% (*m*/*m*)	min. 99	clause 10
Matter volatile at 105 °C	% (*m*/*m*)	max. 0,5	ISO 787 Part II
Matter soluble in water	% (*m*/*m*)	max. 0,5	ISO 787 Part III
Alkalinity of aqueous extract	ml of 0,1 N acid solution per gram	max. 0,2	ISO 787 Part IV
Residue on sieve	% (*m*/*m*)	max. 0,5	ISO 787 Part VI or VII[3)]
Colour		Equal to the agreed reference sample	ISO 787 Part I
Lightening power		As agreed between the interested parties	ISO 787 Part XVII
Hiding power			To be agreed between the interested parties
Oil absorption value			ISO 787 Part V

1) For painting building interiors the White Lead (Painting) Convention, 1921 (No. 13) of the International Labour Organisation 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" (Part VI) and the "water method" (Part VII). Both methods are acceptable for the purpose of this International Standard 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.

METHODS OF TEST

All reagents used shall be of recognized analytical reagent quality. Distilled water or water of at least equivalent purity shall be used.

6 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 Reagents

6.1.1 Nitric acid, 4 M.

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

6.1.3 Potassium hydroxide solution, 33 % (*m/m*).

Dissolve 50 g of potassium hydroxide in 100 ml of water.

6.1.4 Magnesium perchlorate, anhydrous.

6.1.5 Indicating soda lime or soda asbestos.

6.2 Apparatus

Use the apparatus for the determination of carbon dioxide (CO_2) shown in the figure below.

Before making a determination prepare the apparatus by slowly aspirating air and perform a blank test without any sample, following the procedure given in 6.3. 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.

KEY TO FIGURE

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 (6.1.3).
E	Washing vessel containing sulphuric acid (6.1.2).
F	U-tube containing a glass-wool plug to trap spray and filled with anhydrous magnesium perchlorate (6.1.4).
G and H	Two ground glass stoppered U-tubes filled with indicating soda lime or soda asbestos (6.1.5) (size 1,4 to 2 mm) in the first two-thirds of the space, and anhydrous magnesium perchlorate (6.1.4) in the remaining space (at the side where the gas stream passes out).
J	Indicating bubble counter containing concentrated sulphuric acid (6.1.2). (Alternatively, a third U-tube filled with anhydrous magnesium perchlorate in the first third of the 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.

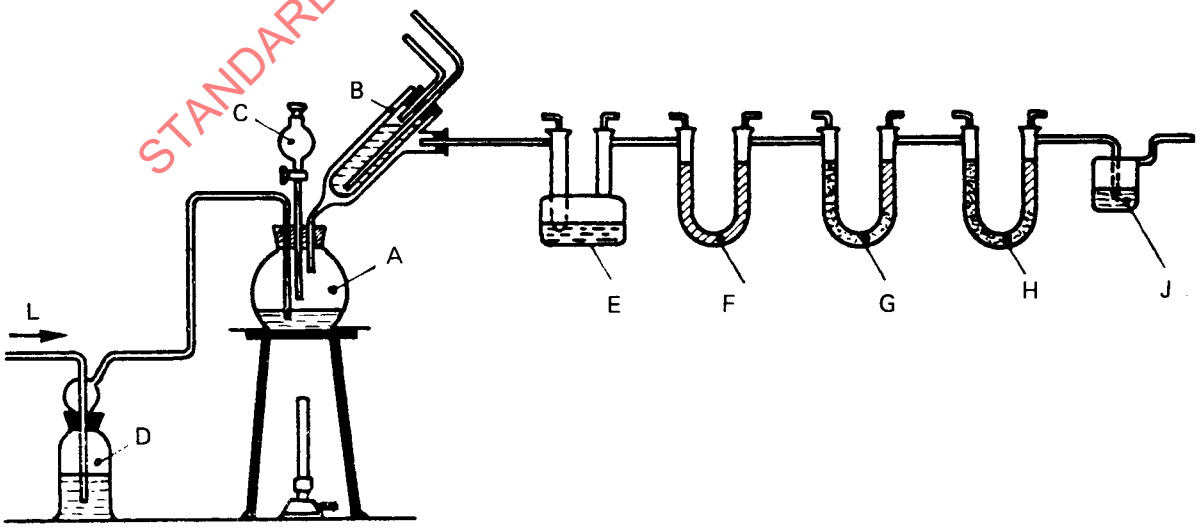


FIGURE – **Apparatus for the determination of the percentage of carbon dioxide (CO_2)**

6.3 Procedure

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) 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.

6.4 Expression of results

Calculate the lead carbonate ($PbCO_3$) content, a, of white lead, as a percentage by mass, by the following formula :

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

where

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

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

7 DETERMINATION OF TOTAL LEAD CONTENT BY THE CHROMATE METHOD[1]

7.1 Reagents

7.1.1 Nitric acid, 4 M.

7.1.2 Acetic acid solution, 20 g/l.

7.1.3 Congo paper.

7.1.4 Ammonium acetate solution, 2 M, freshly prepared.

7.1.5 Potassium dichromate solution, 50 g/l.

7.2 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 (7.1.1), if necessary by heating. Add to this solution as much of the ammonium acetate solution (7.1.4) as is necessary until the liquid gives no acid reaction with the Congo paper (7.1.3).

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

Combine the filtrate with the washing water and dilute with water to about 200 ml, then heat to boiling. Precipitate, from the boiling liquid, lead as lead chromate ($PbCrO_4$) by adding an excess of the potassium dichromate solution (7.1.5). Keep the liquid boiling until the precipitate has turned dark orange-red and continue heating (on the water-bath) for 1 1/2 to 2 h.

After cooling, filter the precipitate on a weighed sintered glass filter crucible of porosity grade P16 (pore size index 10 to 16 μm), wash with the acetic acid solution (7.1.2), then with hot water and dry in a drying oven at 100 ± 2 °C or in a vacuum desiccator to constant mass.

7.3 Expression of results

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

$$b = \frac{0{,}637\,5\, m_3}{m_2} \times 100 = \frac{63{,}75\, m_3}{m_2}$$

where

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

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

0,637 5 is an empirical conversion factor of lead chromate to lead.

8 DETERMINATION OF TOTAL LEAD CONTENT BY THE SULPHATE METHOD[2]

8.1 Reagents

8.1.1 Hydrochloric acid, 3 M.

8.1.2 Nitric acid, 4 M.

8.1.3 Nitric acid, 4 M, saturated with bromine.

8.1.4 Sulphuric acid, 500 g/l.

8.1.5 Potassium hydroxide solution, 100 g/l.

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.

8.1.6 Ammonium acetate solution, 335 g/l.

8.1.7 Sodium sulphide solution, 100 g/l.

8.1.8 Ethanol or **denatured spirit,** approximately 95 % (*V*/*V*).

8.1.9 Hydrogen sulphide.

8.2 Procedure

8.2.1 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 (8.1.2), cover the beaker and heat gently until all the white lead has dissolved.

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

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 (8.1.1), 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 in hydrogen sulphide (8.1.9), 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 (8.1.5) and 10 ml of the sodium sulphide solution (8.1.7) for 10 min without boiling.

Again filter the lead sulphide onto the same paper and wash with the 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 (8.1.3) and warm the beaker to dissolve all the lead sulphide.

8.2.2 Add 20 ml of the sulphuric acid (8.1.4) 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 (8.1.8) and allow to stand for 2 h.

Transfer the precipitate to a weighed Gooch crucible packed with asbestos (alternatively, a sintered silica crucible of porosity grade P16, i.e. pore size index 10 to 16 μm, may be used) and wash with ethanol. Heat the crucible, gently at first and then to 500 °C for 10 min, cool and weigh.

Pour hot ammonium acetate solution (8.1.6) 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.

8.3 Expression of results

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

$$b = \frac{0{,}683\,2\,(m_5 - m_6)}{m_4} \times 100 = \frac{68{,}32\,(m_5 - m_6)}{m_4}$$

where

m_4 is the mass, in grams, of the test portion;

m_5 is the mass, in grams, of the first precipitate;

m_6 is the mass, in grams, of the residue after extraction with ammonium acetate;

0,683 2 is the conversion factor of lead sulphate to lead.

9 CALCULATION OF LEAD HYDROXIDE CONTENT

Calculate the lead hydroxide [$Pb(OH)_2$] content, c, as a percentage by mass, by the following formula :

$$c = 1{,}164\,(b - 0{,}775\,5\,a)$$

where

a is the lead carbonate content, as a percentage by mass, determined according to clause 6;

b is the total lead content, as a percentage by mass, determined according to clause 7 or 8.

10 CALCULATION OF SUM OF LEAD CARBONATE CONTENT AND LEAD HYDROXIDE CONTENT

Calculate the sum, d, of the lead carbonate content and the lead hydroxide content, as a percentage by mass, by the formula :

$$d = a + c$$

where

a is the lead carbonate content, as a percentage by mass, determined according to clause 6;

c is the lead hydroxide content, as a percentage by mass, calculated according to clause 9.