

INTERNATIONAL STANDARD



3710

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Lead chrome green pigments for paints

Pigments verts de chromate de plomb pour peintures

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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.

International Standard ISO 3710 was drawn up by Technical Committee ISO/TC 35, *Paints and varnishes*, and was circulated to the Member Bodies in December 1974.

It has been approved by the Member Bodies of the following countries :

Austria	Ireland	Spain
Brazil	Italy	Sweden
Bulgaria	Netherlands	Switzerland
France	New Zealand	Turkey
Germany	Portugal	United Kingdom
India	Romania	Yugoslavia
Iran	South Africa, Rep. of	

No Member Body expressed disapproval of the document.

Lead chrome green pigments for paints

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies the requirements and the corresponding methods of test for lead chrome green pigments for paints.

2 REFERENCES

ISO 787, *General methods of test for pigments.*

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

ISO 2495, *Iron blue pigments for paints.*

ISO 3711, *Lead chromate and lead chromate-molybdate pigments for paints.*

3 DEFINITION

lead chrome green pigment : A pigment produced solely from lead chromate pigment complying with ISO 3711 and iron blue pigment complying with ISO 2495, without

addition of extenders and organic colouring matter. Surface active agents may be present.

4 REQUIRED CHARACTERISTICS AND THEIR TOLERANCES

Lead chrome green pigments for paints shall have the characteristics shown in the table.

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 at several points in the table, shall be one and the same and shall comply with all the requirements specified for the pigment under test.

TABLE — Required characteristics and their tolerances

Characteristic	Requirement	Test method
Colour	Shall closely match that of the agreed sample	ISO 787 Part I
Colour on reduction		ISO 787 Part XVI
Relative tinting strength		ISO 787 Part XVI
Matter volatile at 60 °C % (m/m)	max. 3	Clause 6
Matter soluble in water, cold extraction method % (m/m)	max. 2	ISO 787 Part VIII, taking a test portion of 20 g
Acidity or alkalinity of aqueous extract	max. 20 ml of 0,1 N solution per 100 g of pigment	ISO 787 Part IV, taking a test portion of 20 g
Oil absorption value	Shall not differ by more than 15 % from the value agreed between the interested parties	ISO 787 Part V
Residue on sieve (63 µm)	water method % (m/m)	max. 1,5 ¹⁾
	oil method % (m/m)	max. 1,5
Ease of dispersion	Shall not be inferior to that of the agreed sample	ISO 787 Part XX ²⁾
Total lead content % (m/m) Pb	Shall not differ by more than 3 % (absolute) from the value agreed between the interested parties	Clause 7
Acid-soluble lead content % (m/m) Pb	If required, to be agreed between the interested parties	Clause 8

1) Types with high iron blue content may give higher residues on sieve. In such cases the maximum percentage residue shall be agreed between the interested parties.

2) The fineness shall be tested after 2 1/2 min, 5 min and then after every 5 min.

METHODS OF TEST

During the analysis, use only reagents of recognized analytical grade, and only distilled water or water of equivalent purity.

6 DETERMINATION OF MATTER VOLATILE AT 60 °C

6.1 Procedure

Into a tared weighing bottle of diameter about 65 mm, weigh, to the nearest 1 mg, a quantity of the pigment such that the depth of the uniform layer of the pigment does not exceed 5 mm.

Heat the weighing bottle and contents for 16 h (overnight, for example) at 60 ± 2 °C. Cool in a desiccator containing dry silica gel and reweigh.

6.2 Expression of results

The matter volatile at 60 °C is given, as a percentage by mass, by the formula

$$\frac{m_1 - m_2}{m_0} \times 100$$

where

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

m_1 is the mass, in grams, of the weighing bottle and test portion before heating;

m_2 is the mass, in grams, of the weighing bottle and test portion after heating.

7 DETERMINATION OF TOTAL LEAD CONTENT

7.1 Reagents

7.1.1 Ammonium acetate, crystals.

7.1.2 Hydrogen sulphide.

7.1.3 Tartaric acid.

7.1.4 Nitric acid/bromine reagent.

Saturate 4 N nitric acid solution with bromine.

7.1.5 Hydrochloric acid, ρ 1,18 g/ml.

7.1.6 Sulphuric acid, ρ 1,84 g/ml.

7.1.7 Sulphuric acid solution.

Dilute 5 ml of the sulphuric acid (7.1.6) by adding it to water, with cooling, and make up to 100 ml with water.

7.1.8 Ethanol, 95 % (V/V).

7.1.9 Sodium sulphide, 50 g/l solution, freshly prepared.

7.1.10 Ammonium acetate, saturated solution.

7.1.11 Ammonium hydroxide solution, ρ 0,88 g/ml.

7.2 Apparatus

7.2.1 Sintered glass crucibles, of porosity grade P 16 (pore size index 10 to 16 μm).

7.2.2 Sintered silica crucible, of porosity grade P 16 (pore size index 10 to 16 μm).

7.2.3 Beakers, of capacity 600 ml.

7.2.4 Drying oven, capable of being maintained at $105 \pm 2^\circ\text{C}$.

7.2.5 Furnace, capable of being maintained at approximately 500°C .

7.3 Procedure

7.3.1 Test portion

Weigh, to the nearest 0,1 mg, about 0,5 g of the sample.

7.3.2 Determination

Add to the test portion (7.3.1), in a 600 ml beaker (7.2.3), 100 ml of water, 15 ml of the hydrochloric acid (7.1.5) and

2 ml of the ethanol (7.1.8), cover the beaker and heat to boiling. Boil gently until all odour of aldehyde has been removed. Dilute to 200 ml with hot water. Filter the solution whilst hot through a fine filter paper and wash the filter and residue well with hot water until a few drops of the filtrate give no coloration with the sodium sulphide solution (7.1.9).

Combine the filtrate and washings.

Slowly add the ammonium hydroxide solution (7.1.11), while stirring, until a faint permanent precipitate forms. Then add 0,5 g of the tartaric acid (7.1.3) and the hydrochloric acid until the pH value of the solution is between 1 and 2. Pass the hydrogen sulphide (7.1.2) at room temperature ($23 \pm 2^\circ\text{C}$) to saturation, dilute the solution to 400 ml and again saturate with hydrogen sulphide. Allow the precipitated lead sulphide to settle, preferably overnight, and pour off the clear supernatant liquor through the sintered glass crucible (7.2.1) using gentle suction. Wash the precipitate once by decantation with saturated hydrogen sulphide water and transfer it to the crucible with a jet of hydrogen sulphide water using a rubber-tipped glass rod to aid transfer. Wash the precipitate on the crucible five times with hydrogen sulphide water and reject the filtrate and washings.

Dissolve the lead sulphide precipitate on the sintered glass crucible into the original beaker by treatment with the nitric acid/bromine reagent (7.1.4) and wash the crucible with hot water, collecting the washings in the same beaker. Cover the beaker, heat the contents under a fume hood and filter through another sintered glass crucible (7.2.1). Wash the beaker, the cover and the crucible five times with hot water, transferring the filtrate and washings to a second 600 ml beaker. Cool the solution, add 15 ml of the sulphuric acid (7.1.6), and evaporate the solution carefully until dense white fumes are evolved. Cool the beaker and contents, wash down the sides with water and re-evaporate the contents to fuming. Cool the beaker again, add 250 ml of water and 100 ml of the ethanol (7.1.8) and allow the whole to stand overnight.

Pour off the clear liquor through the tared sintered silica crucible (7.2.2), wash the precipitate once by decantation with a mixture of equal parts of the ethanol and the diluted sulphuric acid (7.1.7), and transfer to the crucible by a jet of the same wash fluid, using a rubber-tipped glass rod to aid transfer. Wash the precipitate with the ethanol until the washings are neutral, dry the crucible and precipitate in the drying oven (7.2.4) and heat the crucible at dull red heat (approximately 500°C) to constant mass in the furnace (7.2.5). Cool in a desiccator and weigh.

Fill the crucible with the ammonium acetate crystals (7.1.1) and slowly pour 50 ml of the boiling ammonium acetate solution (7.1.10) through it. Wash very thoroughly with hot water until a few drops of the filtrate give no coloration with the sodium sulphide solution, dry, ignite, cool and re-weigh as before.

7.4 Expression of results

The total lead content, expressed as a percentage by mass of Pb, is given by the formula

$$\frac{68,32 \times m_1}{m_0}$$

where

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

m_1 is the mass, in grams, of the residue (PbSO_4), which is the difference between the two weighings before and after extraction with ammonium acetate.

NOTE — If it is required to express the total lead content as a percentage by mass of PbO, use the formula

$$\frac{73,6 \times m_1}{m_0}$$

where m_0 and m_1 have the same meaning as in the preceding formula.

8 DETERMINATION OF ACID-SOLUBLE LEAD CONTENT

8.1 Reagents

8.1.1 Hydrogen sulphide.

8.1.2 Nitric acid/bromine reagent.

Saturate 4 N nitric acid solution with bromine.

8.1.3 Hydrochloric acid, dilute solution 0,07 N (containing approximately 0,25 % (m/m) of hydrogen chloride).

8.1.4 Sulphuric acid, ρ 1,84 g/ml.

8.1.5 Sulphuric acid solution.

Dilute 5 ml of the sulphuric acid (8.1.4) by adding it to water, with cooling, and make up to 100 ml with water.

8.1.6 Ethanol, 95 % (V/V).

8.2 Apparatus

8.2.1 Beaker, of capacity 1 000 ml.

8.2.2 Beakers, of capacity 600 ml.

8.2.3 Mechanical stirrer.

8.2.4 Water bath, capable of being maintained at $23 \pm 2^\circ\text{C}$.

8.2.5 Drying oven, capable of being maintained at $105 \pm 2^\circ\text{C}$.

8.2.6 Furnace, capable of being maintained at approximately 500°C .

8.2.7 Sintered glass crucibles, of porosity grade P 16 (pore size index 10 to $16\mu\text{m}$).

8.2.8 Sintered silica crucible, of porosity grade P 16 (pore size index 10 to $16\mu\text{m}$).

8.3 Procedure

Place 500 ml of the dilute hydrochloric acid solution (8.1.3) in the 1 000 ml beaker (8.2.1) which is fitted with the mechanical stirrer (8.2.3) and immersed in the water bath (8.2.4) maintained at a temperature of $23 \pm 2^\circ\text{C}$.

Allow the diluted acid to attain the temperature of the bath. Add about 0,5 g of the sample, weighed to the nearest 1 mg. Stir the mixture continuously for exactly 1 h at rate sufficient to keep the whole of the pigment in continuous suspension. Stop the stirring and allow the beaker to remain in the water bath for 1 h. Filter the mixture as rapidly as possible through a sintered glass crucible (8.2.7), preferably by suction, rejecting the first 25 ml of filtrate. Do not wash the crucible.

Treat 400 ml of the perfectly clear filtrate in a 600 ml beaker (8.2.2) with a rapid stream of the hydrogen sulphide (8.1.1) to saturation. Allow the precipitated lead sulphide to settle, preferably overnight, and pour off the clear supernatant liquor through another sintered glass crucible (8.2.7) using gentle suction. Wash the precipitate once by decantation with saturated hydrogen sulphide water and transfer it to the crucible with a jet of hydrogen sulphide water, using a rubber-tipped glass rod to aid transfer. Wash the precipitate on the crucible five times with hydrogen sulphide water and reject the filtrate and washings.

Dissolve the lead sulphide precipitate from the crucible by treatment with the nitric acid/bromine reagent (8.1.2) and wash the crucible well five times with hot water. Transfer the filtrate to a 600 ml beaker and rinse the filter flask three times with small quantities of hot water. Add to the filtrate and washings 8 ml of the sulphuric acid (8.1.4) and evaporate the mixture carefully until dense white fumes are evolved. Cool the beaker and contents, wash down the sides with water and re-evaporate the contents to fuming. Cool the beaker again, add 125 ml of water and 50 ml of the ethanol (8.1.6) and allow the whole to stand overnight.

Filter the contents of the beaker through the tared sintered silica crucible (8.2.8), transferring the precipitate to the crucible with a jet of a mixture of equal parts of the ethanol and the sulphuric acid solution (8.1.5), using a rubber-tipped glass rod to aid transfer. Wash the precipitate with the ethanol until neutral, dry the crucible and the precipitate in the drying oven (8.2.5) and heat at dull red heat (approximately 500°C) to constant mass in the furnace (8.2.6). Cool in a desiccator and weigh.