INTERNATIONAL STANDARD ISO 3711

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Lead chromate pigments and lead chromate-molybdate pigments for paints

Pigments à base de chromate de plomb, et pigments à base de chromate et molybdate de plomb pour peintures

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FOREWORD

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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 3711 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
Brazil
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Germany
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Romania
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Turkey
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The Member Body of the following country expressed disapproval of the document on technical grounds :

Bulgaria

Printed in Switzerland

Lead chromate pigments and lead chromate-molybdate pigments for paints

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies the requirements and the corresponding test methods for the pigments for use in paints classified in table 1, which gives the Colour Index Number[1] and the approximate composition of the pigments.

2 REFERENCES

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

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

3 DEFINITIONS AND TYPES

Lead chromate pigments and lead chromate-molybdate pigments are divided into two types :

3.1 standard type (type 1) : Yellow to red pigments consisting of normal or basic lead chromate with or without lead sulphate and/or molybdate or other co-precipitated water-insoluble compounds of lead. Such pigments shall be free from organic colouring matter and shall not contain extenders. Pigments corresponding to Pigment Yellow Colour Index No. 34 and Pigment Red Colour Index No. 104 may contain co-precipitated compounds of, for example, aluminium and/or silicon when these are required solely for the control of the crystal structure of the pigment.

3.2 stabilized type (type 2) : Yellow to red pigments consisting of normal or basic lead chromate with or without lead sulphate and/or molybdate or other co-precipitated water-insoluble compounds of lead. These pigments contain other materials introduced during manufacture specifically to improve certain pigmentary properties. They shall be free from organic colouring matter and shall not contain extenders. If type 2 is specified, the purchaser may require the vendor to state the nature of the improved properties which are claimed to result from the additions and to declare the minimum content of total lead.

4 REQUIRED CHARACTERISTICS AND THEIR TOLERANCES

Lead chromate pigments and lead chromate-molybdate pigments shall have the characteristics shown in table 2.

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 table 2, shall be one and the same and shall comply with all the requirements specified for the pigment under test.

TABLE 1 — **Classification of lead chromate pigments and lead chromate-molybdate pigments**

Type of pigment	Shade of pigment	Colour Index No.	Approximate composition
Lead chromate	Primrose and lemon	Pigment yellow No. 34 Part 2 Ref. 77 603	Lead sulphochromate
	Middle	Pigment yellow No. 34 Part 2 Ref. 77 600	Lead chromate
	Orange	Pigment orange No. 21 Part 2 Ref. 77 601	Basic lead chromate
Lead chromate-molybdate	Orange to red	Pigment red No. 104 Part 2 Ref. 77 605	Lead sulphochromate-molybdate

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[1] The Colour Index is published by the Society of Dyers and Colourists, Bradford, England, and the American Association of Textile Chemists and Colorists, Lowell, Mass. (U.S.A.), 1956.

TABLE 2 – **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 105 °C	%(*m*/*m*)	max. 1	ISO 787 Part II
Matter soluble in water, cold extraction method	% (*m*/*m*)	max. 1	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
pH value of aqueous suspension		4 to 8	ISO 787 Part IX
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. 0,3	ISO 787 Part VII
	oil method % (*m*/*m*)	max. 0,5	ISO 787 Part VI
Ease of dispersion		Shall not be inferior to that of the agreed sample	ISO 787 Part XX1)
Light fastness		Shall not be inferior to that of the agreed sample	ISO 787 Part XV
Total lead content	% (*m*/*m*) Pb	Shall not differ by more than 3 % (absolute) from the value agreed between the interested parties	Clause 6
Acid-soluble lead content	% (*m*/*m*) Pb	If required, to be agreed between the interested parties	Clause 7

1) 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 reagent grade, and only distilled water or water of equivalent purity.

6 DETERMINATION OF TOTAL LEAD CONTENT

6.1 Reagents

6.1.1 Ammonium acetate, crystals.

6.1.2 Hydrogen sulphide.

6.1.3 Tartaric acid.

6.1.4 Nitric acid/bromine reagent.

Saturate 4 N nitric acid solution with bromine.

6.1.5 Hydrochloric acid, ρ 1,18 g/ml.

6.1.6 Sulphuric acid, ρ 1,84 g/ml.

6.1.7 Sulphuric acid solution.

Dilute 5 ml of the sulphuric acid (6.1.6) by adding it to water, with cooling, and make up to 100 ml with water.

6.1.8 Ethanol, 95 % (*V*/*V*).

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

6.1.10 Ammonium acetate, saturated solution.

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

6.2 Apparatus

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

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

6.2.3 Beakers, of capacity 600 ml.

6.2.4 Drying oven, capable of being maintained at 105 ± 2 °C.

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

6.3 Procedure

6.3.1 *Test portion*

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

6.3.2 *Determination*

Add to the test portion (6.3.1) in a 600 ml beaker (6.2.3), 100 ml of water, 15 ml of the hydrochloric acid (6.1.5) and 2 ml of the ethanol (6.1.8), 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 (6.1.9).

Combine the filtrate and washings.

Slowly add the ammonium hydroxide solution (6.1.11), while stirring, until a faint permanent precipitate forms. Then add 0,5 g of the tartaric acid (6.1.3), and the hydrochloric acid (6.1.5) until the pH value of the solution is between 1 and 2. Pass the hydrogen sulphide (6.1.2) at room temperature (23 ± 2 °C) to saturation, dilute the solution to 400 ml and again saturate with hydrogen sulphide. Allow the precipitated lead sulphide to settle, preferably over-night, and pour off the clear supernatant liquor through a sintered crucible (6.2.1) 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 (6.1.4) 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 (6.2.1). 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 (6.1.6), 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 (6.1.8) and allow the whole to stand overnight.

Pour off the clear liquor through the tared sintered silica crucible (6.2.2), wash the precipitate once by decantation with a mixture of equal parts of the ethanol and the diluted sulphuric acid (6.1.7), 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 (6.2.4) and heat the crucible at dull red heat (approximately 500 °C) to constant mass in the furnace (6.2.5). Cool in a desiccator and weigh.

Fill the crucible with ammonium acetate crystals (6.1.1) and slowly pour 50 ml of the boiling ammonium acetate solution (6.1.10) 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.

6.4 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.

7 DETERMINATION OF ACID-SOLUBLE LEAD CONTENT

7.1 Reagents

7.1.1 Hydrogen sulphide.

7.1.2 Nitric acid/bromine reagent.

Saturate 4 N nitric acid solution with bromine.

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

7.1.4 Sulphuric acid, ρ 1,84 g/ml.

7.1.5 Sulphuric acid, dilute solution.

Dilute 5 ml of the sulphuric acid (7.1.4) by adding it to water, with cooling, and make up to 100 ml with water.

7.1.6 Ethanol, 95 % (*V/V*).

7.2 Apparatus

7.2.1 Beaker, of capacity 1 000 ml.

7.2.2 Beakers, of capacity 600 ml.

7.2.3 Mechanical stirrer.

7.2.4 Water bath capable of being maintained at 23 ± 2 °C.

7.2.5 Drying oven capable of being maintained at 105 ± 2 °C.

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

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

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

7.3 Procedure

Place 500 ml of the dilute hydrochloric acid solution (7.1.3) in the 1 000 ml beaker (7.2.1) which is fitted with the mechanical stirrer (7.2.3) and immersed in the water bath (7.2.4) maintained at a temperature of 23 ± 2 °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 a 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 (7.2.7), 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 (7.2.2) with a rapid stream of hydrogen sulphide (7.1.1) to saturation. Allow the precipitated lead sulphide to settle, preferably overnight, and pour off the clear supernatant liquor through another sintered glass crucible (7.2.7) 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 (7.1.2) 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 (7.1.4) 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 (7.1.6) and allow the whole to stand overnight.

Filter the contents of the beaker through the tared sintered silica crucible (7.2.8), transferring the precipitate to the filter with a jet of a mixture of equal parts of the ethanol and the sulphuric acid solution (7.1.5), 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 (7.2.5) and heat it at dull red heat (approximately 500 °C) to constant mass in the furnace (7.2.6). Cool in a desiccator and weigh.

7.4 Expression of results

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

$$\frac{m_1 \times V_0}{m_0 \times V_1} \times 68{,}32$$

where

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

m_1 is the mass, in grams, of the residue ($PbSO_4$);

V_0 is the volume, in millilitres, of the hydrochloric acid solution (7.1.3) used for the extraction;

V_1 is the volume, in millilitres, of the extract used for the determination.

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

$$\frac{m_1 \times V_0}{m_0 \times V_1} \times 73{,}6$$

where m_0, m_1, V_0 and V_1 have the same meaning as in the preceding formula.

8 TEST REPORT

The test report shall contain at least the following information :

a) a reference to this International Standard or a corresponding national standard;

b) the type and identification of the product tested;

c) the results of the tests, and whether or not the product complies with the relevant specification limits;

d) any deviation, by agreement or otherwise, from the procedures specified;

e) the date of the tests.