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Lead chromate pigments and lead chromate-molybdate pigments — Specifications and methods of test

Pigments à base de chromate et de chromomolybdate de plomb — Spécifications et méthodes d'essai

Reference number
ISO 3711:1990(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established 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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 3711 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*.

This second edition cancels and replaces the first edition (ISO 3711:1976), of which it constitutes a technical revision.

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Lead chromate pigments and lead chromate-molybdate pigments — Specifications and methods of test

1 Scope

This International Standard specifies the requirements and the corresponding methods of test for the following pigments identified by Colour Index numbers[1]: orange 21, yellow 34 and red 104. These pigments are suitable for general use.

NOTE 1 The chemical identity of these pigments is given in table 1.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 385-1:1984, *Laboratory glassware — Burettes — Part 1: General requirements.*

ISO 648:1977, *Laboratory glassware — One-mark pipettes.*

ISO 787-1:1982, *General methods of test for pigments and extenders — Part 1: Comparison of colour of pigments.*

ISO 787-2:1981, *General methods of test for pigments and extenders — Part 2: Determination of matter volatile at 105 °C.*

ISO 787-4:1981, *General methods of test for pigments and extenders — Part 4: Determination of acidity or alkalinity of the aqueous extract.*

ISO 787-5:1980, *General methods of test for pigments and extenders — Part 5: Determination of oil absorption value.*

ISO 787-7:1981, *General methods of test for pigments and extenders — Part 7: Determination of residue on sieve — Water method — Manual procedure.*

ISO 787-8:1979, *General methods of test for pigments and extenders — Part 8: Determination of matter soluble in water — Cold extraction method.*

ISO 787-9:1981, *General methods of test for pigments and extenders — Part 9: Determination of pH value of an aqueous suspension.*

ISO 787-15:1986, *General methods of test for pigments and extenders — Part 15: Comparison of resistance to light of coloured pigments of similar types.*

ISO 787-16:1986, *General methods of test for pigments and extenders — Part 16: Determination of relative tinting strength (or equivalent colouring value) and colour on reduction of coloured pigments — Visual comparison method.*

ISO 787-20:1975, *General methods of test for pigments — Part 20: Comparison of ease of dispersion (Oscillatory shaking method).*

ISO 842:1984, *Raw materials for paints and varnishes — Sampling.*

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[1] The Colour Index is published by The Society of Dyers and Colourists, PO Box 244, Perkin House, 82 Grattan Road, Bradford, West Yorkshire BD1 2JB, United Kingdom

and the

American Association of Textile Chemists and Colorists, National Headquarters, Box 12215, Research Triangle Park, NC 27709, USA.

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks.*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 3856-1:1984, *Paints and varnishes — Determination of "soluble" metal content — Part 1: Determination of lead content — Flame atomic absorption spectrometric method and dithizone spectrophotometric method.*

ISO 4793:1980, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation.*

3 Classification

In this International Standard, lead chromate pigments and lead chromate-molybdate pigments are classified as one of the following two types:

Standard type (type 1): Yellow to red pigments consisting of normal or basic lead chromate with or without lead sulfate 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.

Stabilized type (type 2): Yellow to red pigments consisting of normal or basic lead chromate with or without lead sulfate 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 associated tolerances

4.1 For lead chromate pigments and lead chromate-molybdate pigments complying with this International Standard, the essential requirements are specified in table 2 and the conditional requirements are listed in table 3. The reference pigment and the conditional requirements listed in table 3 shall be specified by agreement between the interested parties.

4.2 The agreed reference pigment shall comply with the requirements of table 2.

Table 1 — Classification of lead chromate pigments and lead chromate-molybdate pigments

Type of pigment	Shade of pigment	Colour Index No.	Chemical identity
Lead chromate	Primrose and lemon	Pigment yellow No. 34, Part 2, Ref. 77 603	Lead sulfochromate
	Yellow	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 sulfochromate-molybdate

Table 2 — Essential requirements

Characteristic	Unit	Requirement	Method of test
Matter volatile at 105 °C	% (*m*/*m*)	max. 1	ISO 787-2
Matter soluble in water (cold extraction method)	% (*m*/*m*)	max. 2	ISO 787-8, taking a test portion of 20 g
Acidity or alkalinity of the aqueous extract	ml of 0,1 mol/l solution per 100 g of pigment	max. 20	ISO 787-4, taking a test portion of 20 g
pH value of aqueous suspension		4 to 8	ISO 787-9
Residue on sieve (45 µm)	% (*m*/*m*)	max. 0,3	ISO 787-7

Table 3 — Conditional requirements

Characteristic	Unit	Requirement	Method of test
Colour		Equal to that of the agreed reference pigment (see 4.2) to within a tolerance agreed between the interested parties	ISO 787-1
Colour on reduction			ISO 787-16
Relative tinting strength			
Ease of dispersion		Shall not be inferior to that of the agreed reference pigment (see 4.2)	ISO 787-20, measuring fineness of grind after 2,5 min, 5 min and thereafter every 5 min.
Resistance to light		Shall not be inferior to that of the agreed reference pigment (see 4.2)	ISO 787-15
Oil absorption value		Shall not differ by more than 15 % from the agreed value	ISO 787-5
Total lead content as Pb	% (*m*/*m*)	Shall not differ by more than 3 % (*m*/*m*) from the agreed value	See clause 6
Soluble lead content as Pb in 0,07 mol/l HCl	% (*m*/*m*)	If required, to be agreed between the interested parties	See clause 7

5 Sampling

Take a representative sample of the product to be tested, as described in ISO 842.

6 Determination of total lead content

For the determination of the total lead content, two methods (one gravimetric, the other titrimetric) are specified. The method to be used shall be agreed on between the interested parties. The gravimetric method (6.1) shall be used as the referee method in cases of dispute.

6.1 Gravimetric method

6.1.1 Principle

Dissolution of the test portion in hydrochloric acid. Separation of the lead as lead sulfide and gravimetric determination of the lead after precipitation as lead sulfate and subsequent extraction with ammonium acetate.

6.1.2 Reagents

During the analysis, use only reagents of recognized analytical grade and only water of at least grade 3 purity as defined in ISO 3696.

WARNING — Use the reagents in accordance with the appropriate health and safety regulations.

6.1.2.1 Ammonium acetate, crystals.

6.1.2.2 Hydrogen sulfide.

6.1.2.3 Tartaric acid.

6.1.2.4 Nitric acid/bromine reagent.

Saturate nitric acid, $c(HNO_3) = 4$ mol/l, with bromine.

6.1.2.5 Hydrochloric acid, concentrated, approximately 37 % (m/m), $\rho \approx 1,19$ g/ml.

6.1.2.6 Sulfuric acid, concentrated, approximately 96 % (m/m), $\rho \approx 1,84$ g/ml.

6.1.2.7 Sulfuric acid, dilute.

Dilute 5 ml of the concentrated sulfuric acid (6.1.2.6) by adding it to water, with cooling, and making up to 100 ml with water.

6.1.2.8 Ethanol, 95 % (V/V).

6.1.2.9 Sodium sulfide, 50 g/l solution, freshly prepared.

6.1.2.10 Ammonium acetate, saturated solution.

6.1.2.11 Ammonia solution, approximately 25 % (m/m), $\rho \approx 0,91$ g/ml.

6.1.3 Apparatus

Use ordinary laboratory apparatus and glassware complying with the requirements of the relevant International Standards (see clause 2), together with the following.

6.1.3.1 Sintered-glass crucibles, grade P 16, complying with the requirements of ISO 4793.

6.1.3.2 Sintered-silica crucibles, grade P 16, complying with the requirements of ISO 4793.

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

6.1.3.4 Muffle furnace, capable of being maintained at 500 °C ± 25 °C.

6.1.4 Procedure

Carry out the determination in duplicate.

6.1.4.1 Test portion

Weigh out, to the nearest 0,1 mg, about 0,5 g of the sample (see clause 5).

6.1.4.2 Determination

Place the test portion (6.1.4.1) in a 600 ml beaker and add, as a reducing agent, 2 ml of ethanol (6.1.2.8), followed by 100 ml of water and 15 ml of hydrochloric acid (6.1.2.5). Cover the beaker with a watch-glass and heat to boiling. Boil gently until all odour of aldehyde has disappeared. 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 sodium sulfide solution (6.1.2.9). Combine the filtrate and washings and allow to cool to room temperature.

Slowly add ammonia solution (6.1.2.11), whilst stirring, until a faint permanent precipitate forms. Then add 0,5 g of tartaric acid (6.1.2.3) and hydrochloric acid (6.1.2.5), until the pH-value of the solution is between 1 and 2. Pass hydrogen sulfide (6.1.2.2) through the solution in a fume cupboard until it is saturated. Dilute the solution to 400 ml and again saturate with hydrogen sulfide. Allow the precipitated lead sulfide to settle, preferably overnight, and pour off the clear supernatant liquor through a sintered-glass crucible (6.1.3.1), using gentle suction. Wash the precipitate once by decantation with water saturated with hydrogen sulfide and transfer it to the crucible with a jet of the same water using a rubber-tipped glass rod to aid the transfer. Wash the precipitate on the crucible five times with water saturated with hydrogen sulfide and discard the filtrate and washings.

Place the sintered-glass crucible in the original 600 ml beaker and add nitric acid/bromine reagent (6.1.2.4) to dissolve the lead sulfide precipitate. Wash the crucible with hot water, collecting the washings in the same beaker. Cover the beaker, heat the contents in a fume cupboard, and filter through another sintered-glass crucible (6.1.3.1).

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 concentrated sulfuric acid (6.1.2.6), 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 until white fumes are again evolved. Cool the beaker again, add 40 ml of ethanol (6.1.2.8) and 75 ml of water and allow the whole to stand overnight.

Pour off the clear liquor through a tared sintered-silica crucible (6.1.3.2), wash the precipitate once by decantation with a mixture of equal parts of ethanol and dilute sulfuric acid (6.1.2.7) and transfer to the