BRITISH STANDARD

BS 318:1988
ISO 4621:1986

Specification for

Chrome oxide green pigments for paints

[ISO title: Chrome oxide green pigments — Specifications and methods of test]

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Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Pigments, Paints and Varnishes Standards Committee (PVC/-) to Technical Committee PVC/1 upon which the following bodies were represented:

Aluminium Powder and Paste Association
British Colour Makers' Association
British Precast Concrete Federation Ltd.
Cement Makers' Federation
Chemical Industries Association
Oil and Colour Chemists' Association
Paintmakers' Association of Great Britain Ltd.
Red Lead and Litharge Manufacturers' Association
Titanium Pigment Manufacturers' Technical Committee

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Contents

National foreword

This British Standard has been prepared under the direction of the Pigments, Paints and Varnishes Standards Committee. It is identical with ISO 4621:1986 "*Chrome oxide green pigments — Specifications and methods of test*", published by the International Organization for Standardization (ISO). This revision supersedes BS 318:1968, which is withdrawn.

BS 318 was first published in 1928 and this fourth revision brings the standard into line with international agreements by implementing the International Standard as an identical British Standard.

The main differences from the 1968 edition of this British Standard are as follows:

a) the requirements for a type suitable for rubbers and plastics are omitted;

b) the grades are differentiated by particle size requirements specified with reference to a 45 µm sieve;

c) the sampling is carried out according to the standardized procedure for raw materials for paints;

d) oil absorption, colour, relative tinting strength and dispersibility are included in conditional requirements.

It has been assumed in the drafting of this standard that it will be used and applied by those who are appropriately qualified and experienced.

Terminology and conventions. The text of the International Standard has been approved as suitable for publication as a British Standard without deviation. Some terminology and certain conventions are not identical with those used in British Standards; attention is drawn especially to the following.

The comma has been used as a decimal marker. In British Standards it is current practice to use a full point on the baseline as the decimal marker.

The symbol "l" has been used to denote litre (and in its submultiples). In British Standards it is current practice to use the symbol "L" and to use the spelling "sulphur", etc., instead of "sulfur", etc.

Wherever the words "International Standard" appear, referring to this standard, they should be read as "British Standard" and "ISO 4621" should be read as "BS 318".

Cross-references

International Standard	Corresponding British Standard
	BS 3483 *Methods for testing pigments for paints*
ISO 787-1:1982	Part A1:1983 *Comparison of colour* (Identical)
ISO 787-2:1981	Part B6:1982 *Determination of matter volatile at 105 °C* (Identical)
ISO 787-3:1979	Part C1:1980 *Determination of matter soluble in water (hot extraction method)* (Identical)
ISO 787-5:1980	Part B7:1982 *Determination of oil absorption value* (Identical)
ISO 787-7:1981	Part B3:1982 *Determination of residue on sieve (water method, using manual procedure)* (Identical)
ISO 787-9:1981	Part C4:1982 *Determination of pH value of an aqueous suspension* (Identical)

International Standard	Corresponding British Standard
ISO 787-16:1986	Part A4:1988 *Determination of relative tinting strength and colour on reduction of coloured pigments using visual comparison*[a] (Identical)
ISO 787-20:1975	Part B5:1974 *Comparison of ease of dispersion (oscillatory shaking method)* (Technically equivalent)
ISO 842:1984	BS 4726:1986 *Methods for sampling raw materials for paints and varnishes* (Identical)
ISO 1042:1983	BS 1792:1982 *Specification for one-mark volumetric flasks* (Technically equivalent)
ISO 3696:1987[b]	BS 3978:1987 *Specification for water for laboratory use* (Identical)
	BS 3900 *Methods of test for paints*
ISO 3856-6:1984	Part B11:1986 *Determination of total chromium content of liquid matter* (Identical)
ISO 6713:1984	Part B5:1986 *Preparation of acid extracts from liquid paints or coating powders* (Identical)

[a] In preparation.

[b] Since ISO 3696 has been published the footnote to clause **2** of BS 318 is no longer valid.

The Technical Committee has reviewed the provisions of ISO 385-1:1984 and ISO 648:1977, to which reference is made in the text, and has decided that they are acceptable for use in conjunction with this standard. Related British Standards for these International Standards are BS 846:1985 "*Specification for burettes*" and BS 1583:1986 "*Specification for one-mark pipettes*" respectively.

Additional information. In order to carry out some of the tests described in this standard it is necessary for a reference sample to be supplied or agreed by the parties concerned (see Table 2).

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i to iv, pages 1 to 6, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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1 Scope and field of application

This International Standard specifies the requirements and corresponding methods of test for chrome oxide green pigments suitable for general use.

2 References

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

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

ISO 787, *General methods of test for pigments and extenders — Part 1: Comparison of colour of pigments — Part 2: Determination of matter volatile at 105 °C — Part 3: Determination of matter soluble in water — Hot extraction method — Part 5: Determination of oil absorption value — Part 7: Determination of residue on sieve — Water method — Manual procedure — Part 9: Determination of pH value of an aqueous suspension — Part 16: Determination of relative tinting strength (or equivalent colouring value) and colour on reduction of coloured pigments — Visual comparison method — Part 20: Comparison of ease of dispersion (Oscillatory shaking method).*

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

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

ISO 3696, *Water for laboratory use — Specifications*[1].

ISO 3856-6, *Paints and varnishes — Determination of "soluble" metal content — Part 6: Determination of total chromium content of the liquid portion of the paint — Flame atomic absorption spectrometric method.*

ISO 6713, *Paints and varnishes — Preparation of acid extracts from paints in liquid or powder form.*

3 Definition

chrome oxide green pigment

an inorganic, coloured pigment consisting essentially of chromium(III) oxide (Cr_2O_3) in the form of a dry powder

4 Classification

This International Standard specifies the following three grades of chrome oxide green pigments, which shall not be in admixture with other colouring matter of organic or inorganic nature and shall be free from extenders or diluents (see also the note to clause **9**).

Grade 1: These pigments shall contain a residue on a sieve of 45 µm mesh aperture of not more than 0,01 % (*m*/*m*).

Grade 2: These pigments shall contain a residue on a sieve of 45 µm mesh aperture of more than 0,01 % (*m*/*m*) and not more than 0,1 % (*m*/*m*).

Grade 3: These pigments shall contain a residue on a sieve of 45 µm mesh aperture of more than 0,1 % (*m*/*m*) and not more than 0,5 % (*m*/*m*).

5 Required characteristics and their tolerances

5.1 For chrome oxide green pigments complying with this International Standard, the essential requirements are specified in Table 1 and the conditional requirements are referred to in Table 2. For the conditional requirements according to Table 2, agreement between the interested parties is necessary.

5.2 The agreed reference pigment, referred to in Table 2, shall conform with the requirements specified in Table 1.

6 Sampling

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

7 Determination of total chromium content as chromium(III) oxide

7.1 Reagents

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

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

7.1.1 *Sodium peroxide*

7.1.2 *Sulfuric acid,* 1 + 1.

Cautiously add 1 volume of sulfuric acid, 96 % (*m*/*m*), to 1 volume of water, while cooling.

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[1] At present at the stage of draft.

Table 1 — Essential requirements

Characteristic			Requirement	Test method
Chromium content, expressed as Cr_2O_3		% (*m*/*m*)	min. 96	Clause **7**
"Soluble" chromium content		% (*m*/*m*)	max. 0,02	Clause **8**
Matter volatile at 105 °C		% (*m*/*m*)	max. 0,3	ISO 787-2
Loss on ignition at 1 000 °C		% (*m*/*m*)	max. 1	Clause **9**
Matter soluble in water (hot extraction method)		% (*m*/*m*)	max. 0,2	ISO 787-3
Residue on sieve (45 μm)	grade 1	% (*m*/*m*)	max. 0,01	ISO 787-7[a]
	grade 2	% (*m*/*m*)	Greater than 0,01, max. 0,1	
	grade 3	% (*m*/*m*)	Greater than 0,1, max. 0,5	

[a] It will be necessary to break up loose agglomerates on the sieve by gently brushing, but care should be taken not to disrupt aggregates.

Table 2 — Conditional requirements

Characteristic	Requirement	Test method
Colour	Shall closely match that of the agreed reference pigment (**5.2**)	ISO 787-1
Relative tinting strengh	Shall closely match that of the agreed reference pigment (**5.2**)	ISO 787-16
Ease of dispersion	Shall not be inferior to that of the agreed reference pigment (**5.2**)	ISO 787-20, or other agreed method
pH of aqueous suspension	Shall not differ by more than 1 pH unit from that of the agreed reference pigment (**5.2**)	ISO 787-9
Oil absorption value	Shall not differ by more than 15 % from the value agreed between the interested parties	ISO 787-5
Resistance to acid and alkali	The colour change shall not be greater than that of the agreed reference pigment (**5.2**).	Clause **10**

7.1.3 *Ammonium iron(II) sulfate,* standard volumetric solution, $c[(NH_4)_2Fe(SO_4)_2 \, 6H_2O] \approx 0{,}2$ mol/l.

7.1.3.1 *Preparation*

Dissolve 78,4 g of ammonium iron(II) sulfate hexahydrate in about 200 ml of water and add 50 ml of the sulfuric acid (**7.1.2**). Dilute to the mark in a 1 000 ml one-mark volumetric flask, complying with the requirements of ISO 1042, and mix well.

7.1.3.2 *Standardization*

Immediately before use standardize the ammonium iron(II) sulfate solution (**7.1.3.1**) by the following procedure.

Weigh, to the nearest 0,1 mg, approximately 0,9 g of the potassium dichromate (**7.1.4**), dissolve it in 200 ml of water and acidify with 10 ml of the sulfuric acid solution (**7.1.2**).

Titrate the solution as described in **7.3.2**.

7.1.4 *Potassium dichromate,* dried at 150 to 200 °C.

7.1.5 *Ammonium persulfate*

7.1.6 *Silver nitrate*

7.2 Apparatus

Ordinary laboratory apparatus, and

7.2.1 *Crucible,* made of alumina or other suitable material such as zirconium, fitted with a lid.

7.2.2 *Muffle furnace,* capable of being maintained at 700 ± 25 °C.

7.2.3 *Burette,* of capacity 100 ml, complying with the requirements of ISO 385-1.

7.2.4 *pH meter,* with a range of 0 to 1 400 mV.

7.2.5 *Calomel electrode*

7.2.6 *Platinum electrode*

7.2.7 *Magnetic stirrer,* with a polytetrafluoroethylene (PTFE) coated rod.

7.3 Procedure

Carry out the determination, together with the blank determination, in duplicate.

7.3.1 *Test portion*

Weigh, to the nearest 0,1 mg, a test portion of approximately 0,5 g into the crucible (**7.2.1**), add 5 to 6 g of the sodium peroxide (**7.1.1**), and mix well.

7.3.2 ***Determination***

Cover the crucible and heat in the muffle furnace (**7.2.2**), maintained at 700 ± 25 °C, for 10 min. Allow the crucible and lid to cool for 10 min and then immerse them in about 200 ml of water in a 600 ml beaker and immediately cover the beaker with a watch-glass. Gently warm the beaker and contents until the vigorous reaction ceases. Carefully remove the crucible and lid and rinse them with water until free from the solution. Return the rinsings to the beaker.

Cool, add sufficient sulfuric acid (**7.1.2**) to change the colour from yellow to orange, and then add a further 10 ml of the sulfuric acid (**7.1.2**). Add 2,5 g of the ammonium persulfate (**7.1.5**) and 0,01 g of the silver nitrate (**7.1.6**), and gently boil for 30 min. Cool, stir the solution by means of the magnetic stirrer (**7.2.7**) and titrate it potentiometrically by adding the ammonium iron(II) solution (**7.1.3**) from the burette (**7.2.3**), using the pH meter (**7.2.4**) and electrodes (**7.2.5** and **7.2.6**) to determine the end-point.

NOTE The end-point of the titration may also be determined by addition of a redox indicator, for example sodium diphenylamine sulfonate. In this case, 15 ml of orthophosphoric acid [88 % (*m*/*m*)] and an additional 20 ml portion of the sulfuric acid (**7.1.2**) should be added to the solution before titration. The indicator should be added shortly before the end-point is reached.

7.4 Expression of results

7.4.1 ***Calculation***

Calculate the total chromium content of the pigment, using the equation

$$w(\mathrm{Cr_2O_3}) = \frac{51{,}67 \times V_1 \times m_2}{m_1 \times V_2}$$

where

m_1 is the mass, in grams, of the test portion;

m_2 is the mass, in grams, of the potassium dichromate (**7.1.4**) used for the standardization;

V_1 is the volume, in millilitres, of the ammonium iron(II) sulfate solution (**7.1.3**) required for the determination;

V_2 is the volume, in millilitres, of the ammonium iron(II) sulfate solution (**7.1.3**) required for the standardization;

$w(\mathrm{Cr_2O_3})$ is the chromium(III) oxide content of the pigment, expressed as a percentage by mass;

51,67 is the factor (× 100) for the conversion of grams of potassium chromate ($K_2Cr_2O_7$) to grams of chromium(III) oxide (Cr_2O_3).

Take the mean of the two determinations, but if the duplicate determinations differ by more than 0,2 % (*m*/*m*), repeat the procedure.

7.4.2 ***Precision***

No precision data are currently available.

8 Determination of "soluble" chromium content

8.1 Preparation of the hydrochloric acid extract

Prepare the hydrochloric acid extract of the pigment sample by the method described in ISO 6713, sub-clause **8.2**.

8.2 Determination of chromium

NOTE For the determination of acid-soluble chromium, two methods are provided. Method A (**8.2.1**) should be used as the referee method in cases of dispute. Other methods can be used by agreement between the interested parties. If a spectrophotometric method is agreed, method B (**8.2.2**) should be used.

8.2.1 ***Method A*** (Atomic absorption spectrometric method)

Use the method described in ISO 3856-6, taking as the test solution the hydrochloric acid extract (**8.1**). Calculate the soluble chromium content of the pigment, using the equation

$$w(\mathrm{Cr}) = \frac{100 \times m_0}{m_5}$$

where

m_0 is the mass, in grams, of soluble chromium in the hydrochloric acid extract (**8.1**)

m_5 is the mass, in grams, of the test portion taken to prepare the hydrochloric acid extract (**8.1**);

$w(\mathrm{Cr})$ is the "soluble" chromium content of the pigment, expressed as a percentage by mass.

8.2.2 ***Method B*** (Spectrophotometric method)

8.2.2.1 *Principle*

After addition of sodium hydroxide solution to the hydrochloric acid extract, oxidation of any trivalent chromium by hydrogen peroxide to hexavalent chromium. Spectrophotometric measurement of the hexavalent chromium at a wavelength of 366 nm.

8.2.2.2 *Reagents*

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

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

8.2.2.2.1 *Hydrochloric acid,* $c(\mathrm{HCl}) = 0{,}07$ mol/l.

8.2.2.2.2 *Sodium hydroxide,* approximately 40 g/l solution.

8.2.2.2.3 *Hydrogen peroxide,* 30 % (*m*/*m*) solution.

8.2.2.2.4 *Chromium,* standard solution containing 100 mg of Cr per litre.

Weigh, to the nearest 0,1 mg, 282,9 mg of dry potassium dichromate, dissolve in the hydrochloric acid (**8.2.2.2.1**) in a 1 000 ml one-mark volumetric flask, dilute to the mark with the same hydrochloric acid and mix well.

1 ml of this standard solution contains 100 µg of Cr.

8.2.2.3 *Apparatus*

Ordinary laboratory apparatus, and

8.2.2.3.1 *Spectrophotometer,* suitable for measurements at a wavelength of about 366 nm, fitted with cells of optical path length 10 mm.

8.2.2.3.2 *Pipette,* of capacity 25 ml, complying with the requirements of ISO 648.

8.2.2.3.3 *Burette,* of capacity 25 ml, complying with the requirements of ISO 385-1.

8.2.2.3.4 *One-mark volumetric flasks,* of capacity 100 ml and 1 000 ml, complying with the requirements of ISO 1042.

8.2.2.4 *Procedure*

8.2.2.4.1 *Preparation of standard colorimetric solutions*

Prepare these solutions on the day of use.

Into a series of six 100 ml one-mark volumetric flasks (**8.2.2.3.4**), introduce from the burette (**8.2.2.3.3**), respectively, the volumes of the standard chromium solution (**8.2.2.2.4**) shown in Table 3.

Using a graduated cylinder, add 50 ml of the sodium hydroxide solution (**8.2.2.2.2**) to each flask, dilute to the mark with water, and mix well.

Table 3 — Standard colorimetric solutions

Standard colorimetric solution No.	Volume of standard chromium solution (8.2.2.2.4)	Corresponding concentration of Cr in the standard colorimetric solution
	ml	µg/ml
0[a]	0	0
1	2,5	2,5
2	5	5
3	7,5	7,5
4	10	10
5	12,5	12,5

[a] Compensation solution.

8.2.2.4.2 *Spectrophotometric measurements*

Measure the absorbances of the standard colorimetric solutions (**8.2.2.4.1**) with the spectrophotometer (**8.2.2.3.1**) at the wavelength of maximum absorption (about 366 nm) against water in the reference cell. Before each measurement, rinse the cell with the standard colorimetric solution. Deduct the absorbance of the compensation solution from those of the other standard colorimetric solutions.

8.2.2.4.3 *Calibration graph*

Plot a graph having the masses, in micrograms, of Cr contained in 1 ml of the standard colorimetric solutions as abscissae and the corresponding values of absorbance as ordinates. If the procedure has been carried out correctly and if the light of the light source is sufficiently monochromatic, the calibration graph should be a straight line.

8.2.2.4.4 *Determination*

Measure with the pipette (**8.2.2.3.2**) into a 150 ml beaker a volume, not exceeding 50 ml, of the hydrochloric acid extract, obtained as described in **8.1**, such that the absorbance will be within the calibration range. Dilute with water to 50 ml. Add 25 ml of the sodium hydroxide solution (**8.2.2.2.2**) and 1 ml of the hydrogen peroxide solution (**8.2.2.2.3**) and boil the solution for 15 min.

After cooling to room temperature, transfer the solution together with any precipitate that may have been formed into a 100 ml one-mark volumetric flask (**8.2.2.3.4**). Rinse the beaker with the sodium hydroxide solution (**8.2.2.2.2**), transferring the rinsings to the flask. Dilute to the mark with the sodium hydroxide solution and then filter off any precipitate.

Measure the absorbance of the solution or filtrate as described in **8.2.2.4.2**.

8.2.2.5 *Expression of results*

8.2.2.5.1 *Calculation*

Calculate the "soluble" chromium content of the pigment, using the equation

$$w(\mathrm{Cr}) = \frac{a \times V_3}{m_5 \times V_4 \times 100}$$

where

a is the chromium concentration, in micrograms per millilitre, of the test solution, obtained from the calibration graph;

m_5 is the mass, in grams, of the test portion taken to prepare the hydrochloric acid extract in ISO 6713, sub-clause **8.2**;

V_3 is the volume, in millilitres, of the hydrochloric acid plus ethanol used for the extraction in ISO 6713, sub-clause **8.2**;

V_4 is the volume, in millilitres, of hydrochloric acid extract used in **8.2.2.4.4**;

w(Cr) is the "soluble" chromium content of the pigment, expressed as a percentage by mass.

8.2.2.5.2 *Precision*

No precision data are currently available.

9 Determination of loss on ignition at 1 000 °C

NOTE The determination of loss on ignition is generally carried out in order to determine physically or chemically bound water. Discoloration, as the result of heating generally indicates the presence of small amounts of organic substances in chromic oxide pigments.

9.1 Apparatus

9.1.1 *Muffle furnace,* capable of being maintained at 1 000 ± 50 °C.

9.1.2 *Platinum crucible*

9.2 Procedure

9.2.1 *Test portion*

Weigh, to the nearest 0,1 mg, approximately 5 g of the sample into the platinum crucible (**9.1.2**), heated immediately before to 1 000 ± 50 °C for 15 min, cooled and weighed.

9.2.2 *Determination*

Heat the crucible and pigment in the muffle furnace (**9.1.1**) at 1 000 ± 50 °C for 15 min. Allow to cool in a desiccator to room temperature and weigh.

9.3 Expression of results

9.3.1 *Calculation*

Calculate the loss on heating of the pigment, using the equation

$$w = \frac{100 \times (m_3 - m_4)}{m_3}$$

where

m_3 is the mass, in grams, of the test portion;

m_4 is the mass, in grams, of the residue;

w is the loss on heating of the pigment, expressed as a percentage by mass.

9.3.2 *Precision*

No precision data are currently available.

10 Resistance to acid and alkali

10.1 Reagents

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

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

10.1.1 *Hydrochloric acid,* 1 + 4.

Dilute 20 ml of hydrochloric acid, 37 % (*m*/*m*), to 100 ml with water.

10.1.2 *Sodium hydroxide,* 200 g/l solution.

10.2 Procedure

Shake about 2 g of the test sample in a test tube with a convenient volume of the hydrochloric acid (**10.1.1**). Allow to stand for 1 h. Treat the agreed reference pigment at the same time in the same manner. Note any differences of colour change in the suspensions of the pigments. Repeat the test using the sodium hydroxide solution (**10.1.2**) in place of the hydrochloric acid and again note any differences of colour change in the suspensions of the pigments.

11 Test report

The test report shall contain at least the following information:

a) the type and identification of the product tested;

b) a reference to this International Standard (ISO 4621);

c) the results of the tests, the method used where a choice is available 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 dates of the tests.

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Publications referred to

See national foreword.

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