BRITISH STANDARD

BS 4313:1988
ISO 2040:1972

Specification for

Strontium chromate pigments for paints

[ISO title: Strontium chromate pigments for paints]

UDC 667.662:661.843:661.875.27:620.1.113

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

This British Standard, having been prepared under the direction of the Pigments, Paints and Varnishes Standards Committee, was published under the authority of the Board of BSI and comes into effect on 30 November 1988

First published April 1968
First revision

The following BSI references relate to the work on this standard:
Committee reference PVC/1
Draft for comment 76/50335 DC

ISBN 0 580 16979 0

Amendments issued since publication

Amd. No.	Date of issue	Comments

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 2040:1972 "*Strontium chromate pigments for paints*" published by the International Organization for Standardization (ISO) and confirmed in 1985. This revision supersedes BS 4313:1968 which is withdrawn.

BS 4313 was first published in 1968 and this first revision brings the standard in 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 requirement for total chromate content has been reduced;

b) the requirement for soluble chromate content has been reduced;

c) the sieve residue is carried out only by the water method.

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".

Wherever the words "International Standard" appear, referring to this standard, they should be read as "British Standard".

Cross-references

International Standard	Corresponding British Standard
	BS 3483 *Methods for testing pigments for paints*
ISO 787-2:1981[a]	Part B6:1982 *Determination of matter volatile at 105 °C* (Identical)
ISO 787-5:1980[b]	Part B7:1982 *Determination of oil absorption value* (Identical)
ISO 787-7:1981[c]	Part B3:1982 *Determination of residue on sieve (water method, using a manual procedure)* (Identical)
ISO 842:1984[d]	BS 4726:1986 *Methods for sampling raw materials for paints and varnishes* (Identical)

[a] Referred to in the text as ISO/R 787-II.
[b] Referred to in the text as ISO/R 787-V.
[c] Referred to in the text as ISO/R 787-VII.
[d] Referred to in the text as ISO/R 842.

The Technical Committee has reviewed the provisions of ISO/R 787-X[1)], to which reference is made in the text, and has decided that they are acceptable for use in conjunction with this standard. A related British Standard to ISO 787-10 is BS 3483 "*Methods for testing pigments for paints*" Part B8:1982 "*Determination of density (pyknometer method)*".

Since the publication of ISO 2040, ISO 787-VI has been withdrawn and the Technical Committee has reviewed the position and decided that for the purposes of this standard it is acceptable that only the sieve analysis by the water method given in ISO/R 787-VII[2)] should apply.

[1)] Now published as ISO 787-10:1981.
[2)] Now published as ISO 787-7:1981

Additional information. In clause **5**, water complying with grade 3 of BS 3978 is suitable. For the hydrochloric acid solution specified in **5.1.1** and **5.3.3.1**, and the sulphuric acid specified in **5.3.4.2**, the density unit is grams per millilitre.

The concentrations of a number of volumetric solutions are given as obsolete normalities and molarities. The concentrations in SI units are as follows.

0.1 N Sodium thiosulphate: $c(Na_2S_2O_3) = 0.1$ mol/L

2 N Hydrochloric acid: $c(HCl) = 2$ mol/L

1 M Potassium iodide: $c(KI) = 1$ mol/L

0.1 N Silver nitrate: $c(AgNO_3) = 0.1$ mol/L

5 N Sulphuric acid: $c(H_2SO_4) = 2.5$ mol/L

4 N Sodium hydroxide: $c(NaOH) = 4$ mol/L.

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 4, 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 lays down the requirements and test methods for strontium chromate pigments of approximate composition $SrCrO_4$ suitable for use in paints and corrosion-inhibiting coatings.

2 References

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

ISO/R 842, *Sampling raw materials for paints and varnishes.*

3 Required characteristics and their tolerances

Strontium chromate pigments for paints shall have the characteristics shown in the Table below.

4 Sampling

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

5 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.

5.1 Determination of strontium content

5.1.1 *Reagents*

1) *Acetic acid*, 10 % (V/V) solution.

2) *Hydrochloric acid*, d = 1.18.

3) *Sulphuric acid*, 50 g/l solution.

4) *Sulphamic acid*

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

5.1.2 *Apparatus*

Sintered silica crucible, porosity P 16 (maximum pore size 10 to 16 μm).

5.1.3 *Procedure*

5.1.3.1 *Test portion*

Weigh, to the nearest 0.1 mg, about 0.25 g of the sample.

5.1.3.2 *Determination*

Transfer the test portion into a 500 ml glass-stoppered flask. Add 50 ml of acetic acid 1) and shake or stir for 1 h at room temperature in such a manner that the pigment is kept in continuous suspension without increasing the temperature of the extracting liquid.

Filter the solution through an ash-free close-textured filter paper until a perfectly clear filtrate is obtained. Wash the residue on the filter with two 12.5 ml portions of acetic acid 1), combining the filtrate and washings. Add 2.5 ml of hydrochloric acid 2) and 70 ml of ethanol 5) and boil until the volume is reduced to about 35 ml.

Add 10 ml of ethanol 5) and 1.5 g of sulphamic acid 4) and heat the solution to near boiling. Allow to stand on a hot-water bath for 1 h with occasional stirring.

Allow the precipitate so formed to settle at room temperature. When the supernatant liquid is clear and the contents of the beaker cool, filter through the tared sintered silica crucible (**5.1.2**). Wash the precipitate with a mixture of 9 parts of ethanol 5) and 1 part of sulphuric acid solution 3) until the filtrate is colourless. Ignite the crucible at 800 °C for 30 min, cool in a desiccator and weigh.

5.1.4 *Expression of results*

Calculate the strontium content as a percentage by mass, expressed as SrO, by the formula

$$\frac{56.416 \times m_1}{m_0}$$

where

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

m_1 is the mass, in grams, of residue.

Report the result to one decimal place.

5.2 Determination of total chromate content

5.2.1 *Reagents*

1) *Sodium thiosulphate*, 0.1 N standard volumetric solution.

2) *Starch solution*, 1 % (m/m).

3) *Hydrochloric acid*, 2 N.

4) *Potassium iodide*, 1 M solution.

5) *Sodium hydrogen carbonate*

5.2.2 *Procedure*

5.2.2.1 *Test portion*

Weigh, to the nearest 0.1 mg, about 0.25 g of the sample.

Table — Required characteristics and their tolerances

Characteristic[a]		Requirement	Test method
Strontium content	% SrO	min. 48	clause **5.1**
Total chromate content	% CrO_3	min. 46	clause **5.2**
Water-soluble chloride content	% Cl	max. 0.1	clause **5.3.2**
Water-soluble nitrate content	% NO_3	max. 0.1	clause **5.3.3** or **5.3.4**
Chromate content in 100 ml of extract from 10 g of pigment	g CrO_3/100 ml	0.04 to 0.1	clause **5.3.5**
Volatile matter at 105 °C	%	max 1.0	ISO/R 787-II
Oil absorption value, compared with value agreed between the interested parties		within ± 15 %	ISO/R 787-V
Residue on sieve	oil method %	max. 0.5	ISO/R 787-VI
(63 μm)	water method %	max. 0.3	ISO/R 787-VII

[a] If a value for density is agreed between the interested parties, the method for determination shall be that given in ISO/R 787-X.

5.2.2.2 *Determination*

Dissolve the test portion in 30 ml of hydrochloric acid 3), in a conical stoppered flask. Make up to 100 ml with water, and add 2 g of sodium hydrogen carbonate 5). Add 10 ml of potassium iodide solution 4) and allow the flask to stand for 5 min in the dark. Afterwards, titrate with sodium thiosulphate solution 1). Towards the end of the titration add 5 ml of starch solution 2) as indicator and titrate until the colour changes to green or blue-green.

5.2.3 ***Expression of results***

Calculate the total chromate content as a percentage by mass, expressed as CrO_3, by the formula

$$\frac{3.33 \times V \times T}{m}$$

where

V is the volume, in millilitres, of 0.1 N sodium thiosulphate solution required;

T is the normality of the sodium thiosulphate solution;

m is the mass, in grams, of the test portion.

Report the result to one decimal place.

5.3 Determination of water-soluble chloride and nitrate contents, and water-soluble chromate content

The aqueous extract prepared according to **5.3.1** is used for the determination of

a) water-soluble chloride and nitrate contents;

b) water-soluble chromate content.

For the water-soluble nitrate content, two methods are provided:

Method A (**5.3.3**) for use when it is only required to determine whether the content is above or below the specified limit of 0.1 %;

Method B (**5.3.4**) for use when a precise determination of the content is required.

5.3.1 ***Preparation of aqueous extract***

5.3.1.1 *Apparatus*

Mechanical agitator or stirrer.

5.3.1.2 *Procedure*

5.3.1.2.1 *Test portion*

Weigh 30 ± 0.1 g of the sample in a chemically resistant glass flask.

5.3.1.2.2 *Preparation*

Agitate the test portion with 300 ml of water for 1 h at room temperature in such a manner that the pigment is kept in continuous suspension without increasing the temperature of the water. Filter the mixture and reserve the perfectly clear filtrate for the determinations according to **5.3.2** to **5.3.5**.

5.3.2 ***Determination of water-soluble chloride content***

5.3.2.1 *Reagents*

1) *Potassium chromate*, 50 g/l solution.

2) *Silver nitrate*, 0.1 N standard volumetric solution.

5.3.2.2 *Procedure*

Take 100 ml of the clear aqueous extract (**5.3.1**) and add 1 ml of potassium chromate solution 1). Titrate the solution with silver nitrate solution 2), slowly and with vigorous shaking, until a faint reddish brown colour persists.

Carry out a blank determination by adding 1 ml of potassium chromate solution 1) to 100 ml of water and titrating with silver nitrate solution 2) until the colour matches that of the previous titration, making due allowance for any opalescence or turbidity.

NOTE Alternatively, the end point of the titration may be determined by potentiometric indication.

5.3.2.3 *Expression of results*

Calculate the water-soluble chloride content as a percentage by mass, expressed as Cl, by the formula

$$0.035\ 4\ (V_1 - V_0)$$

where

V_0 is the volume, in millilitres, of 0.1 N silver nitrate solution required in the blank determination;

V_1 is the volume, in millilitres, of 0.1 N silver nitrate solution required by the test portion.

Report the result to two decimal places.

5.3.3 ***Determination of water-soluble nitrate content — Method A***

5.3.3.1 *Reagents*

1) *Hydrochloric acid*, d = 1.18.

2) *Sodium hydroxide* solution, 200 g/l.

3) *Ammonium chloride* solution, 17.2 mg/l.

4) *Devarda's alloy*, powdered.

5) *Ammonia-free water*

NOTE Ammonia-free water may be prepared by redistilling approximately 500 ml of distilled water to which has been added 1 g of anhydrous sodium carbonate and 1 g of potassium permanganate. Reject the first 100 ml of distillate and then collect about 300 ml.

6) *Nessler's reagent*, prepared by either of the following methods:

a) Dissolve 5 g of potassium iodide in 3.5 ml of water. Add cold saturated mercury (II) chloride ($HgCl_2$) solution, while stirring, until a faint red precipitate is formed. With continued stirring add 40 ml of 50 % potassium hydroxide solution, dilute to 100 ml with water, mix well, allow to settle, decant the clear supernatant liquid and store it in the dark.

or

b) Dissolve 3.5 g of potassium iodide and 1.25 g of mercury (II) chloride in 80 ml of water. Add cold saturated mercury (II) chloride solution, while stirring, until a slight red precipitate remains, then add 12 g of sodium hydroxide, stir until dissolved, and finally add a little more saturated mercury (II) chloride solution and dilute to 100 ml with water. Stir occasionally during several days, allow to stand, and use the clear supernatant liquid for the test.

5.3.3.2 *Apparatus*

1) *Distillation apparatus*

2) *Nessler cylinders*, 50 ml.

5.3.3.3 *Procedure*

Place 50 ml of the clear aqueous extract (**5.3.1**) into the distillation flask and dilute to 150 ml with ammonia-free water 5). Add 3 g of Devarda's alloy 4) and 30 ml of sodium hydroxide solution 2) and close the apparatus at once. Place 2 ml of hydrochloric acid 1) and 30 ml of ammonia-free water 5) in the receiver. Warm the flask gently until the reaction starts and then allow the reaction to proceed gently for about half an hour. Then distil about 70 ml of liquid, the receiver being kept cool with running water. Make up the distillate to 250 ml with ammonia-free water 5) and transfer 5 ml to a Nessler cylinder. Dilute to 50 ml with ammonia-free water 5). Transfer 5 ml of ammonium chloride solution 3) (equivalent to 0.1 % NO_3) into a similar Nessler cylinder and dilute to 50 ml with ammonia-free water. Add 1 ml of Nessler's reagent 6) to each cylinder and mix each thoroughly. Allow both cylinders to stand for 5 min and compare the intensity of colour of the two solutions.

5.3.3.4 *Expression of results*

Report the result as either greater than or less than 0.1 % NO_3.

5.3.4 ***Determination of water-soluble nitrate content — Method B***

5.3.4.1 *Principle*

The nitrate present in the test solution is used to nitrate salicylic acid in sulphuric acid medium. The nitro-compound formed is of an intense yellow colour in alkaline solution, and the colour is measured spectrophotometrically at a wavelength of 410 nm.

5.3.4.2 *Reagents*

1) *Sulphuric acid*, d = 1.84.

2) *Sulphuric acid*, 5 N.

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

4) *Sodium salicylate* solution, 5 g/l, freshly prepared.

5) *Sodium hydroxide* solution, 300 g/l.

6) *Sodium hydroxide* solution, 4 N.

7) *Potassium nitrate*, dried at 120 °C and cooled in a desiccator.

5.3.4.3 *Apparatus*

1) *Spectrophotometer* suitable for measurements at a wavelength of 410 nm.

2) *10 mm cells* for use with the spectrophotometer.

3) *pH meter*

4) *Volumetric flasks*, of capacity 50 ml, 100 ml, 250 ml and 500 ml.

5.3.4.4 *Preparation of calibration graph*

Standard solution I. Weigh 163 ± 0.1 mg of potassium nitrate 7), dissolve it in water in a 100 ml volumetric flask, make up to the mark and mix well.

Standard solution II. Dilute 10 ml of standard solution I to a volume of 500 ml.

Measure 2, 4, 6, 8 and 10 ml of standard solution II (corresponding to 0.04, 0.08, 0.12, 0.16 and 0.2 mg of NO_3 respectively) into separate 100 ml glass beakers.

Treat each beaker as follows: Add 1 ml of sodium salicylate solution 4), evaporate to dryness on a water bath and allow to cool in a desiccator. Moisten the whole of the dried residue with 1 ml of sulphuric acid 1) and allow to stand in the desiccator for 10 min. Afterwards, wash the contents into 50 ml volumetric flasks with water, add 10 ml of sodium hydroxide solution 5) and cool to room temperature. Make up to the mark with water and mix well. Determine and record the optical density at 410 nm in a 10 mm cell against a water blank.

Construct a graph of optical density against milligrams of NO_3.

5.3.4.5 *Procedure*

Place 50 ml of clear aqueous extract (**5.3.1**) into a 250 ml glass beaker and add 5 ml of sulphuric acid 2) and 2 ml of ethanol 3). Warm the solution until the chromate is reduced as indicated by a change of colour to green. Boil the solution vigorously to drive off organic matter, taking care to avoid losses by splashing, cool and add sodium hydroxide solution 6) until just alkaline. Cool again and adjust the pH to 8.0 ± 0.5 measured by the pH meter. Filter through filter paper and wash with hot water, collecting the filtrate and washings. Cool, make up the volume to 250 ml and mix.

Transfer 10 ml (see Note) of this solution to a 100 ml glass beaker. Add 1 ml of sodium salicylate solution 4) and proceed as described in **5.3.4.4** including the determination of the optical density at 410 nm.

From the known optical density of the test solution, determine from the calibration graph the corresponding mass of nitrate in milligrams.

NOTE If the nitrate content is more than 0.1 %, carry out a second determination, using 5 ml of solution.

5.3.4.6 *Expression of results*

Calculate the water-soluble nitrate content as a percentage by mass, expressed as NO_3, by the formula

$\frac{a}{2}$, when 10 ml of solution have been used;

a, when 5 ml of solution have been used (see Note to **5.3.4.5**);

where a is the mass, in milligrams, of NO_3 corresponding to the optical density of the test solution.

5.3.5 ***Determination of water-soluble chromate content***

5.3.5.1 *Reagents*

1) *Hydrochloric acid*, 2 N.

2) *Potassium iodide* solution, 1 M.

3) *Sodium thiosulphate*, 0.1 N standard volumetric solution.

4) *Starch solution*, 1 % (m/m).

5) *Sodium hydrogen carbonate*

5.3.5.2 *Procedure*

Transfer 50 ml of clear aqueous extract (**5.3.1**) to a conical stoppered flask. Add 50 ml of water and 2g of sodium hydrogen carbonate 5), followed by 30 ml of hydrochloric acid 1).

Then add 10 ml of potassium iodide solution 2) and allow the flask to stand for 5 min in the dark. Afterwards, titrate with sodium thiosulphate solution 3). Towards the end of the titration add 5 ml of starch solution 4) as indicator and titrate until the colour changes to green.

5.3.5.3 *Expression of results*

Calculate the water-soluble chromate content in grams per 100 ml, expressed as CrO_3, by the formula

$$0.066\,7 \times V \times T$$

where

V is the volume, in millilitres, of sodium thiosulphate solution required;

T is the normality of the sodium thiosulphate solution.

Report the result to two decimal places.

Publications referred to

See national foreword.

BSI — British Standards Institution

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