INTERNATIONAL STANDARD

ISO 2495

Second edition
1995-03-15

Iron blue pigments — Specifications and methods of test

Pigments de bleu de Prusse — Spécifications et méthodes d'essai

Reference number
ISO 2495:1995(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 2495 was prepared by Technical Committee ISO/TC 35, *Paints and varnishes*, Subcommittee SC 2, *Pigments and extenders*.

This second edition cancels and replaces the first edition (ISO 2495:1972), which has been technically revised.

Annex A of this International Standard is for information only.

International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland
Printed in Switzerland

Iron blue pigments — Specifications and methods of test

1 Scope

This International Standard specifies the requirements and the corresponding methods of test for iron blue pigments.

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 787-3:1979, *General methods of test for pigments and extenders — Part 3: Determination of matter soluble in water — Hot extraction method.*

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 787-16:1986, *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 842:—[1], *Raw materials for paints and varnishes — Sampling.*

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

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

ISO 8780-2:1990, *Pigments and extenders — Methods of dispersion for assessment of dispersion characteristics — Part 2: Dispersion using an oscillatory shaking machine.*

3 Definition

For the purposes of this International Standard, the following definition applies.

3.1 iron blue pigment: A pigment formed by the reaction of iron salts with cyanoferrate(II) or cyanoferrate(III) ions and followed, if necessary, by treatment with oxidizing agents.

---

[1] To be published. (Revision of ISO 842:1984)

4 Required characteristics and associated tolerances

4.1 Composition

The pigment shall be essentially an iron blue pigment as defined in clause 3. The pigment, after drying as described in 9.1 and examination as described in clauses 7 and 8, shall comply with the following requirements:

a) The sum of the iron(II) and iron(III) ions (basic iron content), together with the iron cyanoferrate complex content [expressed as $Fe(CN)_6$] shall be not less than 70 % (*m/m*).

b) The total iron content (expressed as Fe) shall be not less than 30 % (*m/m*).

The pigment, when examined by the procedure described in clause 6, shall be free from added colouring matter.

NOTE 1 The pigment may contain substances added during manufacture for the purpose of improving the quality or working properties, or both, of the pigment.

4.2 Appearance

The material shall be in the form of a soft dry powder or in such a condition that it may be readily reduced thereto by crushing under a palette knife, without grinding action.

4.3 Other characteristics

4.3.1 For iron blue pigments complying with this International Standard, the essential requirements are specified in table 1 and the conditional requirements are listed in table 2. The conditional requirements shall be specified by agreement between the interested parties.

4.3.2 The agreed reference pigment referred to in table 2 shall comply with the requirements of table 1.

5 Sampling

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

6 Identification

6.1 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.

6.1.1 Sodium hydroxide, 50 g/l solution.

6.1.2 Hydrochloric acid, diluted 1 + 1.

Add 1 part by volume of concentrated hydrochloric acid [approximately 37 % (*m/m*), $\rho \approx 1{,}19$ g/ml] to 1 part by volume of water.

6.2 Apparatus

Use ordinary laboratory apparatus and glassware.

6.3 Procedure

Place approximately 0,1 g of the pigment in a 50 ml beaker and add 15 ml of the sodium hydroxide solution (6.1.1). Heat the beaker until the liquid boils and continue to heat for 5 min. Check that the blue coloration has been completely destroyed by examining the liquid in the beaker against an illuminated white background. Ignore a reddish-brown precipitate which may be formed as this only indicates the presence of iron(III) hydroxide.

NOTE 2 If the blue coloration is not completely destroyed, this indicates the presence of an added pigment that is not iron blue as defined in clause 3.

Cool the liquid in the beaker and slowly add the hydrochloric acid (6.1.2) until the liquid is faintly acid to litmus. Examine the liquid for reformation of the characteristic iron blue coloration.

6.4 Assessment of results

Record whether the pigment sample is completely decolorized by treatment with alkali and is reformed on acidification.

Table 1 — Essential requirements

Characteristic	Unit	Requirement	Method of test
Volatile matter w(VM)	% (m/m)	$2,0 < w(\mathrm{VM}) < 6,0$	See clause 9
Matter soluble in water (hot extraction method)	% (m/m)	max. 2	ISO 787-3
Acidity or alkalinity of the aqueous extract	ml of 0,1 mol/l solution per 100 g of pigment	max. 20	ISO 787-4

Table 2 — Conditional requirements

Characteristic	Requirement	Method of test
Colour	Equal to that of the agreed reference pigment (see 4.3.2) 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 4.3.2)	See clause 10
Oil absorption value	Shall not differ by more than 10 % from the value agreed between the interested parties	ISO 787-5

7 Determination of sum of iron(II) and iron(III) ions (basic iron content) and of hexacyanoferrate complex content

For the determination of the basic iron content and hexacyanoferrate complex content, two methods are provided. Method A (7.1) shall be used as the referee method in cases of dispute.

NOTE 3 It is recommended that mercury be removed from the waste solutions before discharge to effluent drains. A suggested procedure is given in annex A.

7.1 Method A

7.1.1 Principle

A test portion is decomposed by cold aqueous potassium hydroxide solution to form insoluble iron hydroxide and soluble potassium hexacyanoferrate(II).

The iron hydroxide is dissolved in hydrochloric acid. The iron(III) is reduced to iron(II) with tin(II) chloride solution and the iron(II) is titrated with potassium dichromate solution, using barium diphenylamine sulfonate as indicator.

The potassium cyanoferrate(II) is oxidized to cyanoferrate(III) with potassium permanganate solution. Then potassium iodide solution is added and the iodine liberated is titrated with sodium thiosulfate solution.

7.1.2 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.

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

7.1.2.2 Hydrochloric acid, diluted 1 + 1.

Add 1 part by volume of the concentrated hydrochloric acid (7.1.2.1) to 1 part by volume of water.

7.1.2.3 Mixture of sulfuric and orthophosphoric acids.

Mix 310 ml of concentrated sulfuric acid [approximately 96 % (m/m), $\rho \approx 1,84$ g/ml)] carefully with 250 ml of concentrated orthophosphoric acid (H_3PO_4) [approximately 85 % (m/m), $\rho \approx 1,70$ g/ml]. Add the resultant mixture slowly to about 400 ml of water and then dilute with water to 1 litre.

7.1.2.4 Potassium hydroxide solution, 10 % *(m/m)*.

7.1.2.5 Potassium iodide solution, 10 % *(m/m)*.

7.1.2.6 Sodium acetate solution.

Dissolve 500 g of crystalline sodium acetate in 1 000 ml of water.

7.1.2.7 Mercury(II) chloride, saturated solution (60 g/l to 100 g/l).

7.1.2.8 Tin(II) chloride, 100 g/l solution.

Dissolve 50 g of $SnCl_2 \cdot 2H_2O$ in 300 ml of concentrated hydrochloric acid (7.1.2.1) and dilute with water to 500 ml.

Keep the solution clear by storing it in a hermetically closed flask containing a little metallic tin.

7.1.2.9 Sodium thiosulfate, standard volumetric solution, $c(Na_2S_2O_3) = 0{,}1$ mol/l.

7.1.2.10 Potassium permanganate, standard volumetric solution, $c(1/5\ KMnO_4) = 0{,}1$ mol/l.

7.1.2.11 Potassium dichromate, standard volumetric solution, $c(1/6\ K_2Cr_2O_7) = 0{,}1$ mol/l.

7.1.2.12 Zinc sulfate solution.

Dissolve 25 g of $ZnSO_4 \cdot 7H_2O$ in 100 ml of water.

7.1.2.13 Barium diphenylamine sulfonate indicator.

Prepare a 0,4 % *(m/m)* aqueous solution of barium diphenylamine sulfonate and filter the solution.

7.1.2.14 Starch, dissolved in hot water to give a 10 g/l solution, or powdered starch soluble in cold water (for example, that known as Zulkowsky starch is soluble).

7.1.3 Apparatus

Use ordinary laboratory apparatus and glassware complying with the requirements of ISO 385-1, together with the following:

7.1.3.1 Filter funnel.

7.1.3.2 Stoppered flask or bottle, of capacity 1 500 ml.

7.1.4 Procedure

7.1.4.1 Preliminary treatment of the test portion

Carry out the determination in duplicate.

Weigh, to the nearest 1 mg, approximately 1 g of the sample (see clause 5) into a 100 ml beaker. Add 20 ml of the potassium hydroxide solution (7.1.2.4). Stir the mixture carefully with a glass rod with a flattened end and allow to stand for 2 h. From time to time lightly grind any residue on the bottom of the beaker with the glass rod. When all the iron blue pigment is decomposed, filter the mixture through the filter funnel (7.1.3.1), containing a medium filter paper, and wash the beaker, glass rod and residue repeatedly with water until the washings are colourless and neutral. Add the washings to the filtrate.

Use the residue obtained for the determination of the sum of iron(II) and iron(III) ions (basic iron content) (7.1.4.2) and the filtrate for the determination of the hexacyanoferrate complex content (7.1.4.3).

7.1.4.2 Determination of sum of iron(II) and iron(III) ions (basic iron content)

Extract the residue on the filter paper with hot 1 + 1 hydrochloric acid (7.1.2.2), collecting the extract in a conical flask and keeping the total volume as small as possible.

NOTE 4 If the acid extract is coloured blue, it is an indication that the residue has not been properly washed and the determination should be repeated with another 1 g of the sample.

Heat the solution to boiling and add the tin(II) chloride solution (7.1.2.8) drop by drop until the solution is just colourless; then add two drops in excess. Keep the solution hot during the addition of the tin(II) chloride. Cool the solution rapidly to room temperature, add 5 ml of the mercury(II) chloride solution (7.1.2.7), mix well and wash down the sides of the flask with cold water. Allow the solution to stand for 1 min and add 40 ml of the sulfuric/orthophosphoric acid mixture (7.1.2.3). Dilute the solution with water to 100 ml to 200 ml according to the amount of iron present. Add 0,2 ml to 0,4 ml of the barium diphenylamine sulfonate indicator (7.1.2.13) and titrate with the potassium dichromate solution (7.1.2.11) until the solution begins to darken. Complete the titration by adding the potassium dichromate solution drop by drop until a permanent violet colour is obtained. Subtract 0,01 ml from the titration figure for each 0,1 ml of the indicator solution used.

7.1.4.3 Determination of hexacyanoferrate complex content

Transfer the filtrate obtained as described in 7.1.4.1 to the stoppered flask or bottle (7.1.3.2), dilute to 800 ml with water and add 30 ml of the hydrochloric acid (7.1.2.1) and 90 ml of the sodium acetate solution (7.1.2.6). Add the potassium permanganate solution (7.1.2.10) in excess, i.e. until a distinct red-brown colour is obtained and the turbidity first formed has disappeared. Then add 20 ml of the potassium iodide solution (7.1.2.5) and allow the mixture to stand for 4 min. Titrate the iodine liberated by the excess of permanganate with the sodium thiosulfate solution (7.1.2.9).

Add to the solution 20 ml of the hydrochloric acid (7.1.2.1), 20 ml of the potassium iodide solution (7.1.2.5) and 20 ml of the zinc sulfate solution (7.1.2.12) and allow the turbid mixture to stand for 3 min. Add either 50 mg of the cold-water-soluble starch or 3 to 4 drops of the starch solution (7.1.2.14) and titrate the liberated iodine with the sodium thiosulfate solution (7.1.2.9). Record the volume of sodium thiosulfate solution used (V_2).

7.1.5 Expression of results

7.1.5.1 Sum of iron(II) and iron(III) ions (basic iron content)

7.1.5.1.1 Calculation

Calculate the sum of the iron(II) and iron(III) ions (basic iron content) $w(\mathrm{Fe})$ of the pigment, expressed as a percentage by mass, using the equation

$$w(\mathrm{Fe}) = \frac{V_1 \times 0{,}558\,5 \times 10^{-2}}{m_1} \times 100$$

where

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

V_1 is the volume, in millilitres, of the potassium dichromate solution (7.1.2.11) used in the determination;

$0{,}558\,5 \times 10^{-2}$ is the factor for the conversion of millilitres of potassium dichromate solution to grams of Fe.

Calculate the mean of the two results and report the final result to the nearest 0,1 % (*m/m*).

7.1.5.1.2 Precision

No precision data are currently available.

7.1.5.2 Hexacyanoferrate complex content

7.1.5.2.1 Calculation

Calculate the hexacyanoferrate complex content $w[\mathrm{Fe(CN)_6}]$ of the pigment, expressed as a percentage by mass, using the equation

$$w[\mathrm{Fe(CN)_6}] = \frac{V_2 \times 2{,}119 \times 10^{-2}}{m_1} \times 100$$

where

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

V_2 is the volume, in millilitres, of the sodium thiosulfate solution (7.1.2.9) used in the second titration (see note 5);

$2{,}119 \times 10^{-2}$ is the factor for the conversion of millilitres of sodium thiosulfate solution to grams of $[\mathrm{Fe(CN)_6}]$.

NOTE 5 The volume of sodium thiosulfate solution required in the second titration is used in the calculation. If the volume required in the first titration (i.e. the difference between the permanganate and thiosulfate figures) is used, the calculated result may be higher than the true value due to interference by traces of organic matter present, the oxidation of which by the permanganate would increase the apparent hexacyanoferrate complex content.

Any excess of thiosulfate in the first titration will cause an opposite error in the second titration.

Calculate the mean of the two results and report the final result to the nearest 0,1 % (*m/m*).

7.1.5.2.2 Precision

No precision data are currently available.

7.2 Method B

7.2.1 Determination of sum of iron(II) and iron(III) ions (basic iron content)

7.2.1.1 Principle

The test portion is decomposed by aqueous sodium hydroxide solution to form insoluble iron hydroxide.

The iron hydroxide is dissolved in hydrochloric acid. The iron(III) is reduced to iron(II) with tin(II) chloride solution and the iron(II) is titrated with potassium permanganate solution.

7.2.1.2 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.

7.2.1.2.1 Sodium hydroxide solution, c(NaOH) approximately 2 mol/l.

7.2.1.2.2 Hydrochloric acid, approximately 25 % (*m/m*), $\rho \approx 1{,}125$ g/ml.

7.2.1.2.3 Tin(II) chloride, 100 g/l solution.

Dissolve 50 g of $SnCl_2 \cdot 2H_2O$ in 300 ml of concentrated hydrochloric acid [approximately 37 % (*m/m*), $\rho \approx 1{,}19$ g/ml] and dilute with water to 500 ml.

Keep the solution clear by storing it in a hermetically closed flask containing a little metallic tin.

7.2.1.2.4 Mercury(II) chloride, saturated solution (60 g/l to 100 g/l).

7.2.1.2.5 Mixture of manganese sulfate/sulfuric acid/orthophosphoric acid.

Dissolve 45 g of $MnSO_4 \cdot H_2O$ in water. Add to the solution 138 ml of orthophosphoric acid [approximately 85 % (*m/m*), $\rho \approx 1{,}70$ g/ml] and 130 ml of sulfuric acid [approximately 96 % (*m/m*), $\rho \approx 1{,}84$ g/ml] and dilute with water to 1 litre.

7.2.1.2.6 Potassium permanganate, standard volumetric solution, $c(1/5\ KMnO_4) = 0{,}1$ mol/l.

7.2.1.3 Apparatus

Use ordinary laboratory apparatus and glassware complying with the requirements of ISO 385-1.

7.2.1.4 Procedure

Carry out the determination in duplicate.

Weigh, to the nearest 1 mg, approximately 1 g of the sample (see clause 5) into a 100 ml beaker. Add 10 ml of the sodium hydroxide solution (7.2.1.2.1) and heat to boiling. Boil for a few minutes.

Filter through a wide-textured filter paper and wash the residue on the filter with hot water until it is free from alkali. Reject the filtrate and washings.

Dissolve the residue on the filter in the hydrochloric acid (7.2.1.2.2). Heat the solution to boiling and add the tin(II) chloride solution (7.2.1.2.3) drop by drop until the solution is just colourless. Dilute with water and, after approximately 2 min, add about 15 ml of the mercury(II) chloride solution (7.2.1.2.4).

The precipitate formed should be white. If it is grey or black due to reduction of the mercury(II) chloride to metallic mercury by excess tin(II) chloride, repeat the determination, using a smaller volume of the tin(II) chloride solution.

Dilute to about 600 ml with water and add 15 ml of the manganese sulfate/orthophosphoric acid/sulfuric acid mixture (7.2.1.2.5). Titrate the solution with the potassium permanganate solution (7.2.1.2.6) until a slight pink coloration persists. Record the volume of potassium permanganate solution used (V_3).

7.2.1.5 Expression of results

7.2.1.5.1 Calculation

Calculate the sum of the iron(II) and iron(III) ions (basic iron content) w(Fe) of the pigment, expressed as a percentage by mass, using the equation

$$w(\mathrm{Fe}) = \frac{V_3 \times 0{,}558\,5 \times 10^{-2}}{m_2} \times 100$$

where

m_2 is the mass, in grams, of the test portion;

V_3 is the volume, in millilitres, of the potassium permanganate solution (7.2.1.2.6) used in the determination;

$0{,}558\,5 \times 10^{-2}$ is the factor for the conversion of millilitres of potassium permanganate solution to grams of Fe.

Calculate the mean of the two results and report the final result to the nearest 0,1 % (*m/m*).

7.2.1.5.2 Precision

No precision data are currently available.

7.2.2 Determination of hexacyanoferrate complex content

WARNING — The method described in this subclause is intended to be carried out by qualified chemists or suitably trained and/or supervised personnel. The substances and procedures used in this method may be injurious to health if adequate precautions are not taken. Particular attention is drawn to the hazards of hydrocyanic acid and its salts.

7.2.2.1 Principle

A test portion is decomposed by aqueous sodium hydroxide solution to form insoluble iron hydroxide and soluble sodium hexacyanoferrate(II).

The sodium hexacyanoferrate(II) is subsequently transformed to mercury(II) cyanide, hydrocyanic acid and sodium cyanide, and the sodium cyanide is titrated with silver nitrate, using potassium iodide as indicator.

7.2.2.2 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.

7.2.2.2.1 Sodium hydroxide solution, $c(\mathrm{NaOH})$ approximately 2 mol/l.

7.2.2.2.2 Sodium hydroxide solution, $c(\mathrm{NaOH})$ approximately 8 mol/l.

7.2.2.2.3 Magnesium chloride solution, $c(\mathrm{MgCl_2})$ approximately 1,5 mol/l.

7.2.2.2.4 Mercury(II) chloride solution, $c(\mathrm{HgCl_2})$ approximately 0,05 mol/l.

7.2.2.2.5 Sulfuric acid, $c(\mathrm{H_2SO_4})$ approximately 2 mol/l.

7.2.2.2.6 Potassium iodide solution, $c(\mathrm{KI})$ approximately 0,25 mol/l.

7.2.2.2.7 Silver nitrate, standard volumetric solution, $c(\mathrm{AgNO_3}) = 0{,}1$ mol/l.

7.2.2.3 Apparatus

Use ordinary laboratory apparatus and glassware complying with the requirements of ISO 385-1 or ISO 1042 as appropriate, together with the following:

7.2.2.3.1 Distillation flask, of capacity 500 ml, with ground-glass neck.

7.2.2.3.2 Distillation apparatus, with ground-glass joints, consisting of a distillation head with a dropping funnel, an Allihn condenser and a distillation receiver (Volhard absorption flask or equivalent).

7.2.2.4 Procedure

Carry out the determination in duplicate.

Weigh, to the nearest 1 mg, approximately 1 g of the sample (see clause 5) into a 100 ml beaker. Add 10 ml of the sodium hydroxide solution (7.2.2.2.1) and heat to boiling. Boil for a few minutes.

Filter through a wide-textured filter paper and wash the residue on the filter with hot water until it is free from alkali. Collect the filtrate and washings in a 250 ml one-mark volumetric flask and make up to the mark with water.

Pipette 25 ml of the solution into the distillation flask (7.2.2.3.1) and add 15 ml of the sodium hydroxide solution (7.2.2.2.2), 100 ml of hot water and 30 ml of the magnesium chloride solution (7.2.2.2.3).

Boil the solution (which now contains precipitated magnesium hydroxide) for 5 min and add 100 ml of the mercury(II) chloride solution (7.2.2.2.4). Continue boiling for 5 min to 10 min. After that time, the sodium hexacyanoferrate(II) has been transformed to mercury(II) cyanide.

Connect the flask to the distillation apparatus (7.2.2.3.2). From the dropping funnel add slowly 50 ml of the sulfuric acid (7.2.2.2.5). Distil the liberated hydrocyanic acid into the distillation receiver which contains the sodium hydroxide solution (7.2.2.2.1), so that sodium cyanide is formed.

Titrate the solution in the distillation receiver with the silver nitrate solution (7.2.2.2.7), using potassium iodide solution (7.2.2.2.6) as indicator, until a slight yellowish-brown coloration persists. Record the volume of silver nitrate solution used (V_4).

7.2.2.5 Expression of results

7.2.2.5.1 Calculation

Calculate the hexacyanoferrate complex content $w[\mathrm{Fe(CN)_6}]$ of the pigment, expressed as a percentage by mass, using the equation

$$w[\mathrm{Fe(CN)_6}] = \frac{V_4 \times 3{,}53 \times 10^{-2}}{m_3} \times 100$$

where

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

V_4 is the volume, in millilitres, of the silver nitrate solution (7.2.2.2.7) used in the determination;

$3{,}53 \times 10^{-2}$ is the factor for the conversion of millilitres of silver nitrate solution to grams of $[Fe(CN)_6]$.

Calculate the mean of the two results and report the final result to the nearest 0,1 % *(m/m)*.

7.2.2.5.2 Precision

No precision data are currently available.

8 Determination of total iron content

For the determination of the total iron content, two methods are provided. Method A (8.1) shall be used as the referee method in cases of dispute.

8.1 Method A

8.1.1 Principle

The test portion is decomposed by ignition at about 300 °C.

The residue is dissolved in hydrochloric acid. The iron(III) is then reduced to iron(II) with tin(II) chloride solution, followed by titration of the iron(II) with potassium dichromate solution, as described in 7.1.4.2.

8.1.2 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.

8.1.2.1 Nitric acid, concentrated, approximately 65 % *(m/m)*, $\rho \approx 1{,}40$ g/ml.

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

8.1.2.3 Hydrochloric acid, diluted 1 + 1.

Add 1 part by volume of the hydrochloric acid (8.1.2.2) to 1 part by volume of water.

8.1.2.4 Ammonia solution, approximately 33 % *(m/m)*, $\rho \approx 0{,}88$ g/ml.

8.1.2.5 Tin(II) chloride, 100 g/l solution.

Dissolve 50 g of $SnCl_2 \cdot 2H_2O$ in 300 ml of the hydrochloric acid (8.1.2.2) and dilute with water to 500 ml. Keep the solution clear by storing it in a hermetically closed flask containing a little metallic tin.

8.1.3 Apparatus

Use ordinary laboratory apparatus and glassware complying with the requirements of ISO 385-2, together with the following:

8.1.3.1 Muffle furnace.

8.1.4 Procedure

Carry out the determination in duplicate.

Weigh, to the nearest 1 mg, approximately 1 g of the sample (see clause 5) and ignite at about 300 °C in the muffle furnace (8.1.3.1). Moisten the ash with the nitric acid (8.1.2.1), carefully evaporate to dryness and re-ignite to ensure complete decomposition of the hexacyanoferrate complex.

Dissolve the residue in approximately 20 ml of the concentrated hydrochloric acid (8.1.2.2) and dilute the solution to 150 ml with water. Add 2 ml of the nitric acid and boil the mixture to ensure oxidation of the iron salts.

Add the ammonia solution (8.1.2.4) in excess to precipitate all the iron as iron(III) hydroxide, filter through a wide-textured filter paper and wash thoroughly. Dissolve the residue on the filter in hot 1 + 1 hydrochloric acid (8.1.2.3), collecting the solution in a conical flask and keeping the total volume as small as possible.

Heat the solution to boiling, add the tin(II) chloride solution (8.1.2.5) drop by drop until the solution is just colourless and continue as described in 7.1.4.2.

8.1.5 Expression of results

8.1.5.1 Calculation

Calculate the total iron content of the pigment using the equation given in 7.1.5.1.1.

8.1.5.2 Precision

No precision data are currently available.

8.2 Method B

8.2.1 Principle

A test portion is decomposed by ignition in the presence of potassium hydrogen sulfate.

The melt is dissolved in water and hydrochloric acid. The iron(III) is then reduced to iron(II) with tin(II) chloride solution, followed by titration of the iron(II) with potassium permanganate solution as described in 7.2.1.4.

8.2.2 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.

8.2.2.1 Potassium hydrogen sulfate.

8.2.2.2 Hydrochloric acid, approximately 25 % (*m/m*), $\rho \approx 1{,}125$ g/ml.

8.2.2.3 Tin(II) chloride, 100 g/l solution.

Dissolve 50 g of $SnCl_2 \cdot 2H_2O$ in 300 ml of hydrochloric acid [approximately 37 % (*m/m*), $\rho \approx 1{,}19$ g/ml] and dilute with water to 500 ml.

Keep the solution clear by storing it in a hermetically closed flask containing a little metallic tin.

8.2.3 Apparatus

Use ordinary laboratory apparatus and glassware complying with the requirements of ISO 385-1.

8.2.4 Procedure

Carry out the determination in duplicate.

Weigh, to the nearest 1 mg, approximately 0,5 g of the sample (see clause 5) into a crucible, add approximately 3 g of the potassium hydrogen sulfate (8.2.2.1) and ignite with a small flame in a fume cupboard. After cooling, dissolve the melt in water in a 1 000 ml beaker, whilst gently heating, and add some hydrochloric acid (8.2.2.2).

Heat the solution to boiling, add the tin(II) chloride solution (8.2.2.3) drop by drop until the solution is just colourless and continue as described in 7.2.1.4.

8.2.5 Expression of results

8.2.5.1 Calculation

Calculate the total iron content of the pigment using the equation given in 7.2.1.5.1.

8.2.5.2 Precision

No precision data are currently available.

9 Determination of volatile matter

For the determination of the volatile-matter content, two methods are provided. Method A (9.1) shall be used as the referee method in cases of dispute.

9.1 Method A: Determination at 60 °C for 16 h

9.1.1 Procedure

Carry out the determination in duplicate.

Into a weighing bottle of about 65 mm diameter, weigh, to the nearest 1 mg, a test portion of the pigment such that, when spread in a uniform layer, the depth of the layer does not exceed 5 mm.

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

9.1.2 Expression of results

Calculate the volatile-matter content $w(\text{VM})$ of the pigment, expressed as a percentage by mass, using the equation

$$w(\text{VM}) = \frac{m_5 - m_6}{m_4} \times 100$$

where

m_4 is the mass, in grams, of the test portion;

m_5 is the mass, in grams, of the weighing bottle and test portion before heating;

m_6 is the mass, in grams, of the weighing bottle and test portion after heating.

If the two results (duplicates) differ by more than 10 % of the higher value, repeat the procedure described in 9.1.1.

Calculate the mean of two valid results (replicates) and report the final result to the nearest 0,1 % (*m/m*).

9.2 Method B: Determination at 105 °C for 2 h

Carry out the determination by the method described in ISO 787-2 but heat the weighing bottle and contents for 2 h at (105 ± 2) °C, without repeating the heating.

10 Ease of dispersion

Carry out the test in accordance with ISO 8780-2, using an average milling concentration of 20 % (*m/m*) of pigment.

If the pigment under test requires more than 30 min to obtain a fineness of grind of 5 µm, the target of the test should be a fineness of grind of 10 µm.

11 Test report

The test report shall contain at least the following information:

a) all details necessary to identify the product tested;

b) a reference to this International Standard (ISO 2495);

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 from the methods of test specified;

e) the date of the tests.

Annex A
(informative)

Decontamination of the waste solutions from the analysis

To avoid discharging to the environment the mercury contained in the waste solutions from the determination of iron, such solutions should be collected and processed for the removal of their mercury content.

A suitable treatment system may be constructed as follows:

Three 10-litre plastic bottles are connected as illustrated in figure A.1 (the expansion chamber incorporated in the connecting lines minimizes the risk of pressure increase in the case of a vigorous reaction). The first two bottles each contain 3 kg of aluminium or iron rods to induce electrochemically mercury precipitation.

The solution discharged from the third bottle is fed to a neutralization vessel before final discard. Accumulated mercury sludge is removed from time to time and concentrated by decantation before transfer to a recovery plant. The aluminium or iron rods are replaced as necessary.

Reference: ANSMANN, W., *Arch. Eisenhüttenw.* **53**(10), 1982, p. 390.

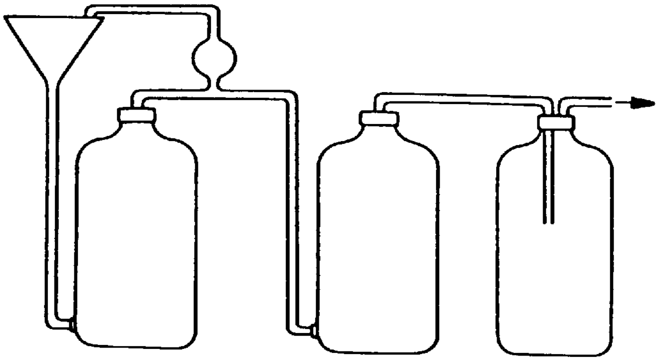

Figure A.1 — Apparatus for removal of mercury from waste solutions

ICS 87.060.10

Descriptors: paints, pigments, blue pigments, specifications, tests.

Price based on 11 pages