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Ultramarine pigments

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 256, *Pigments, dyestuff and extenders*.

This second edition cancels and replaces the first edition (ISO 788:1974), which has been technically revised.

The main changes compared to the previous edition are as follows:

- the title of the standard has been changed from “Ultramarine pigments for paints” to “Ultramarine pigments”;
- the test method of free sulfur has been changed to iodometric method;
- the requirements for free sulfur, water soluble matter and sieve residue are stricter;
- the limitation of the contents of elements and the test methods have been added;
- the requirements and test methods for resistance to light and resistance to bleeding have been added;
- the normative references have been updated and the text has been editorially revised.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Ultramarine pigments

1 Scope

This document specifies the requirements and corresponding test methods for artificial ultramarine pigments in plastics, paints, rubbers, etc.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 385, *Laboratory glassware — Burettes*

ISO 648, *Laboratory glassware — Single-volume pipettes*

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

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

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

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

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

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

ISO 787-10, *General methods of test for pigments and extenders — Part 10: Determination of density — Pyknometer method*

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

ISO 787-16, *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 787-22, *General methods of test for pigments and extenders — Part 22: Comparison of resistance to bleeding of pigments*

ISO 787-24, *General methods of test for pigments and extenders — Part 24: Determination of relative tinting strength of coloured pigments and relative scattering power of white pigments — Photometric methods*

ISO 787-25, *General methods of test for pigments and extenders — Part 25: Comparison of the colour, in full-shade systems, of white, black and coloured pigments — Colorimetric method*

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

ISO 1248:2006, *Iron oxide pigments — Specifications and methods of test*

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

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

ISO 18451-1, *Pigments, dyestuffs and extenders — Terminology — Part 1: General terms*

EN 14469-4, *Pigments and extenders — Testing of colouring materials in plasticized polyvinyl chloride (PVC-P) — Part 4: Determination of bleeding of colouring materials*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18451-1 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at https://www.electropedia.org/

3.1
ultramarine pigments
inorganic pigments made from kaolin, sodium carbonate and sulfur, etc., by calcining at high temperature in closed kilns

4 Classification

According to the content of free sulfur, ultramarine pigments are classified into two types:

- Type A: maximum 0,05 % (mass fraction) free sulfur;
- Type B: maximum 0,20 % (mass fraction) free sulfur.

5 Requirements and test methods

5.1 Appearance

The pigments should be in the form of powder without any visible impurities.

5.2 Technical requirements

5.2.1 For the ultramarine pigments, the basic requirements shall be as specified in Table 1 and the conditional requirements shall be as specified in Table 2.

Table 1 — Basic requirements for ultramarine pigments

Characteristic	Unit	Requirement		Test method
		Type A	Type B	
Matter soluble in water (hot extraction method)	% (mass fraction)	max. 1,0	max. 1,5	ISO 787-3
Residue on sieve (mesh aperture 63 µm, water method)	% (mass fraction)	max. 0,1	max. 0,5	ISO 787-7
Free sulfur	% (mass fraction)	max. 0,05	max. 0,20	Clause 7
Matter volatile at 105 °C	% (mass fraction)	max. 1,0		ISO 787-2
Density at 23 °C	g/cm^3	2,23 to 2,40		ISO 787-10
Organic colouring matter	—	negative test		ISO 1248:2006, Clause 11

Table 2 — Conditional requirements for ultramarine pigments

Characteristic	Unit	Requirement	Test method
pH value of aqueous suspension	—	to be agreed between the interested parties	ISO 787-9
Colour	—	to be agreed between the interested parties	ISO 787-1 or ISO 787-25
Relative tinting strength	—	to be agreed between the interested parties	ISO 787-16 or ISO 787-24
Oil absorption value[a]	—	to be agreed between the interested parties	ISO 787-5
Resistance to light	—	to be agreed between the interested parties	ISO 787-15
Resistance to bleeding	—	to be agreed between the interested parties	ISO 787-22 or EN 14469-4

[a] The oil absorption value of the ordinary commercial grades is usually between 30 ml and 40 ml per 100 g of pigment.

5.2.2 The ultramarine pigments shall meet the requirements for the limitation of elements shown in Table 3.

Table 3 — Requirements for the limitation of elements[a]

Characteristic	Unit	Requirement	Test method
Hg content	mg/kg	max. 1	Clause 8
Cd content	mg/kg	max. 5	
Se content	mg/kg	max. 10	
As content	mg/kg	max. 10	
Cr content	mg/kg	max. 50	
Pb content	mg/kg	max. 50	
Sb content	mg/kg	max. 50	
Ba content	mg/kg	max. 200	

[a] When the requirements of this document are not stricter than the relevant national laws and regulations, it shall be subjected to the national laws and regulations.

6 Sampling

Take a representative sample of the product to be tested, as described in ISO 15528.

7 Determination of free sulfur

7.1 Principle

The free sulfur in the sample reacts with anhydrous sodium sulfite. The production of sodium thiosulfate is determined by the iodometric method.

7.2 Reagents and materials

Use only reagents of recognized analytical grade and water of at least grade 3 as specified in ISO 3696.

7.2.1 Sodium sulfite, anhydrous, CAS-No 7757-83-7.

7.2.2 Formaldehyde solution. Dissolve 8 ml formaldehyde solution [37,0 % to 40,0 % (mass fraction), CAS-No 50-00-0] and dilute to 100 ml.

7.2.3 Acetic acid solution, 30 % (volume fraction). Dissolve 30 ml acetic acid [99,5 % (mass fraction), $\rho = 1{,}42\,\text{g/ml}$, CAS-No 64-19-7] in water and dilute to 100 ml.

7.2.4 Iodine solution. Weigh 0,13 g iodine (CAS-No 7553-56-2) and 0,35 g potassium iodide (CAS-No 7681-11-0) and dissolve in 100 ml water, transfer into a brown bottle and dilute to 1 000 ml.

7.2.5 Sodium thiosulfate standard titration solution, $c\ (Na_2S_2O_3) = 0{,}1$ mol/l.

7.2.6 Soluble starch solution, 5 g/l.

7.2.7 Sodium thiosulfate standard titration solution, $c\ (Na_2S_2O_3) = 0{,}002$ mol/l. Dilute the sodium thiosulfate standard titration solution (7.2.5) with water.

7.3 Apparatus

Use ordinary laboratory apparatus and burettes, pipettes and one-mark volumetric flasks in accordance with the requirements of ISO 385, ISO 648 and ISO 1042, respectively, together with the following.

7.3.1 Conical flask, 250 ml with a narrow neck and equipped with a ground-glass stopper.

7.3.2 Volumetric flask, 250 ml.

7.3.3 Pipette, 20 ml, 25 ml, 50 ml.

7.3.4 Electric heating board.

7.3.5 Burette, 50 ml, graduated in 0,1 ml divisions.

7.3.6 Balance, with an accuracy of 0,1 mg.

7.4 Procedure

7.4.1 Preparation and determination of the test solution

Carry out the determination in duplicate.

Weigh about 7 g to 10 g (to the nearest 0,1 mg) of the sample into a 250 ml conical flask (7.3.1) and add 5 g of anhydrous sodium sulfite (7.2.1) and 70 ml water. Boil under reflux for about 15 min, then cool down to room temperature and transfer the mixture into a 250 ml volumetric flask (7.3.2). Dilute the solution to scale with water and mix well. Store the mixture for about 60 min or separate it using a centrifuge. Remove 25 ml of the supernatant fluid using a single-line pipette and pour it into a 250 ml conical flask. This is the test solution.

Add 50 ml formaldehyde solution (7.2.2) to the test solution, shake it for 5 min. Add 5 ml acetic acid solution (7.2.3), stir the mixture evenly and add a known volume of iodine solution (7.2.4) (usually 50 ml) to ensure the presence of excessive iodine. Gently shake the solution mix it completely, add soluble starch solution (7.2.6) and titrate the excess iodine with sodium thiosulfate standard titration solution (7.2.7). Record the volume of sodium thiosulfate standard titration solution as V_1.

7.4.2 Determination of a blank test solution

Except for adding samples, carry out a blank test exactly the same way as the sample test, using the same amount of reagent, by identical analysis procedures. Record the volume of sodium thiosulfate standard titration solution as V_2.

7.5 Calculation and expression of results

Calculate the free sulfur content ω, as mass fraction in per cent, according to Formula (1):

$$\omega = \frac{0{,}032\,0(V_2 - V_1) \times c}{m \times \frac{25}{250}} \times 100 \qquad (1)$$

where

V_2 is the volume, in millilitres, of sodium thiosulfate standard titration solution in the blank test;

V_1 is the volume, in millilitres, of sodium thiosulfate standard titration solution, which is consumed by the titration sample;

c is the exact value of the concentration, in moles per litre, of sodium thiosulfate standard titration solution;

m is the numerical value of the sample mass, in grams;

0,032 0 is the mass of sulfur equivalent to 1,00 ml iodine solution $c(1/2\ I_2) = 1\ 000$ mol/l.

8 Determination of the elements content

8.1 Reagents and materials

WARNING — Nitric acid and hydrofluoric acid are corrosive and toxic. The related operations shall be performed in fume hood. This document does not point out all possible safety problems, and it is the responsibility of the user to take proper safety and health measures, and determine the applicability of regulatory limitations prior to use.

Use only reagents of recognized analytical grade and water conforming to grade 3 as specified in ISO 3696.

8.1.1 Nitric acid, CAS-No 7697-37-2, about 65 % (mass fraction).

8.1.2 Hydrofluoric acid, CAS-No 7664-39-3, about 40,0 % (mass fraction).

8.1.3 Standard stock solutions of arsenic, cadmium, chromium, lead, mercury, antimony, barium, selenium, 100 mg/l or 1 000 mg/l of certificated substance.

8.2 Apparatus

Use ordinary laboratory apparatus together with the following:

8.2.1 Analytical instrument with an appropriate detection limit, for example, atomic absorption spectroscopy (AAS), inductively coupled plasma–optical emission spectroscopy (ICP-OES), etc.

8.2.2 Balance, with the accuracy of 0,1 mg.

8.2.3 Microwave digestion instrument, can be closed digestion, with temperature control device.

8.2.4 Volumetric flask, hydrofluoric acid resistant, 50 ml, 100 ml.

8.2.5 Filter membrane, with the aperture size of 0,45 µm.

8.2.6 Nitric acid solution, water and nitric acid (8.1.1) with a volume ratio of 1 to 1.

All glass containers should be soaked in nitric acid solution (8.2.6) for 24 h, then washed with water and dried before using.

8.3 Procedure

8.3.1 Preparation and determination of the test solution

Weigh about 0,1 g to 0,2 g (to the nearest 0,1 mg) of the sample into a microwave digestion tank and add 9 ml nitric acid (8.1.1), 1 ml hydrofluoric acid (8.1.2) dropwise, respectively. When no bubbles are produced in the solution, close the digestion tank and place it into the microwave digestion instrument (8.2.3). Set the appropriate digestion conditions. After the microwave digestion is completed, cool down the digestion tank to room temperature and then open it. Wash the inner wall and inner cover of the digestion tank with a small amount of water. Quantitatively transfer the digestion solution into a one-mark volumetric flask (8.2.4). Make up to the mark with water, stopper it and mix well. Filter the solution with a filter membrane (8.2.5). Test the solution within 24 h.

Select the analytical instrument with an appropriate detection limit to determine the content of elements in the test solution and the reagent blank solution.

Refer to the instrument's operation manual before the determination, and include the information of analytical instruments in the test report.

8.3.2 Determination of a blank test solution

Except for adding samples, carry out the blank test exactly the same way as the sample test, using the same amount of reagent, by identical analysis procedures.

8.4 Calculation and expression of results

Calculate the content of the elements, according to the Formula (2):

$$\omega = \frac{(\rho - \rho_0) \times V \times F}{m} \tag{2}$$

where

ω is the content, in milligrams per kilogram, of the element;

ρ is the concentration, in milligrams per litre, of the element in the test solution;

ρ_0 is the concentration, in milligrams per litre, of the element in the blank solution;

V is the total volume, in millilitres, of the test solution;

F is the dilution ratio of the test solution;

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

The result is the mean value of duplicate determination results.

8.5 Detection limit of the test method

The detection limit of the methods to determine the content of the elements should not exceed one tenth of limitation of the standard. The detection limit of the test methods is generally three times the standard deviation of sample blank values. The sample blank values shall be determined by laboratories.