International Standard 473

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Lithopone pigments for paints — Specifications and methods of test

Lithopone pour peintures — Spécifications et méthodes d'essai

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 473 was developed by Technical Committee ISO/TC 35, *Paints and varnishes*.

This second edition was submitted directly to the ISO Council, in accordance with clause 6.11.2 of part 1 of the Directives for the technical work of ISO. It cancels and replaces the first edition (i.e. ISO 473-1976), which had been approved by the member bodies of the following countries :

Austria	Japan	Sweden
Canada	Netherlands	Switzerland
Chile	New Zealand	United Kingdom
Czechoslovakia	Portugal	USSR
Germany, F.R.	Romania	Yugoslavia
India	South Africa, Rep. of	
Italy	Spain	

The member bodies of the following countries had expressed disapproval of the document on technical grounds :

Belgium
France

Printed in Switzerland

Lithopone pigments for paints — Specifications and methods of test

1 Scope and field of application

This International Standard specifies the requirements and the corresponding test methods for two types of lithopone pigments, suitable for use in paints and related products.

2 References

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 4 : Determination of acidity or alkalinity of the aqueous extract.

Part 5 : Determination of oil absorption value.

Part 7 : Determination of residue on sieve — Water method — Manual procedure.

Part 17 : Comparison of lightening power of white pigments.

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

3 Definitions

3.1 lithopone 30 % : A white pigment consisting of zinc sulphide (ZnS) and barium sulphate ($BaSO_4$) in approximately equimolecular proportions. The material is a calcined co-precipitate.

3.2 lithopone 60 %[1] : A white pigment consisting of approximately 60 % zinc sulphide (ZnS), the balance being made up mainly of barium sulphate ($BaSO_4$). The material is a calcined co-precipitate.

4 Required characteristics and their tolerances

4.1 Lithopone pigments for paints shall have the characteristics shown in the table.

4.2 The sample agreed between the interested parties, to which reference is made at several points in the table, shall be one and the same and shall comply with all the requirements specified for the pigment under test.

5 Sampling

Take a representative sample of the product to be tested in accordance with ISO 842.

1) There are on the market

a) lithopones with a content of about 40 or 50 % of zinc sulphide. These products should be marked so as to indicate the zinc sulphide content;

b) barytes-reduced lithopones, which consist of mixtures of lithopones with higher zinc sulphide content and ground mineral barytes. These products should be marked so as to indicate the presence of barytes.

Table — Required characteristics and their tolerances

Characteristic	Requirement	Test method
Total zinc, calculated as zinc sulphide % (*m*/*m*) min. lithopone 30 % lithopone 60 %	 28 58	Clause 6
Zinc oxide, % (*m*/*m*) max.	1	Clause 7
Sum of total zinc, calculated as zinc sulphide, and barium sulphate, % (*m*/*m*) min.	99	Clause 6
Matter volatile at 105 °C % (*m*/*m*) max.	0,5	ISO 787/2
Matter soluble in water, % (*m*/*m*) max.	0,5	ISO 787/3
Residue on sieve (63 µm), % (*m*/*m*) max.	0,1	ISO 787/7
Colour	Closely matching that of the agreed sample	ISO 787/1
Alkalinity of the aqueous extract	Closely matching that of the agreed sample	ISO 787/4
Oil absorption value	To be agreed between the interested parties	ISO 787/5
Lightening power	To be agreed between the interested parties	ISO 787/17
Hiding power	To be agreed between the interested parties	To be agreed

Methods of test

During the analyses, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

6 Determination of barium sulphate and total zinc content

6.1 Reagents

6.1.1 Potassium hexacyanoferrate(II),[1] standard volumetric solution $c[K_4Fe(CN)_6] \approx 0{,}05$ mol/l (titre T expressed in grams of zinc per millilitre).

6.1.1.1 Preparation

Dissolve 21,0 g of potassium hexacyanoferrate(II), 300 mg of potassium hexacyanoferrate(III)[2] and 2 g of anhydrous sodium carbonate (to stabilize the solution) in water and dilute to 1 000 ml in a one-mark volumetric flask.

6.1.1.2 Standardization

Pipette 25,0 ml of the zinc chloride solution (6.1.2) into a flask and add ammonia solution (6.1.6) until a piece of Congo red paper (6.1.8), when touched with the solution, just turns to a pure red colour. Then carefully neutralize the solution with the hydrochloric acid solution (6.1.3) from a dropping bottle. Add a few drops in excess until the Congo red paper turns to a lasting red-blue or blue-red colour (pH 1,5 to 3,0).

Make up to 150 ml with water, heat the solution to boiling, and add 10 drops of the diphenylamine solution (6.1.7).

Immediately titrate the solution with the potassium hexacyanoferrate(II) solution (6.1.1.1) until the colour turns to a lasting yellow or yellowish green.

Then back-titrate the solution with the zinc chloride solution (6.1.2) until the colour just turns to blue again.

6.1.1.3 Calculation of titre

Calculate the titre T of the potassium hexacyanoferrate(II) solution, expressed in grams of zinc per millilitre, by using the formula

$$\frac{c\,(25 + V_2)}{V_1}$$

where

c is the concentration, in grams of zinc per millilitre, of the standard zinc chloride solution (6.1.2);

V_1 is the volume, in millilitres, of the potassium hexacyanoferrate(II) solution (6.1.1.1) required for the titration;

V_2 is the volume, in millilitres, of the standard zinc chloride solution (6.1.2) required for the back-titration.

6.1.2 Zinc chloride, standard reference solution, containing approximately 5 g of zinc per litre (concentration c, in grams of zinc per millilitre).

Weigh about 5 g of chemically pure zinc to the nearest 0,1 mg, dissolve in 300 ml of the hydrochloric acid solution (6.1.3) and dilute the solution obtained with water to 1 000 ml in a one-mark volumetric flask.

1) IUPAC name; formerly called potassium ferrocyanide, $K_4Fe(CN)_6$.

2) IUPAC name; formerly called potassium ferricyanide, $K_3Fe(CN)_6$.

6.1.3 Hydrochloric acid, 4 mol/l solution.

6.1.4 Sulphuric acid, 2 mol/l solution.

6.1.5 Ammonia, solution, ϱ = 0,9 g/ml.

6.1.6 Ammonia, 4 mol/l solution.

6.1.7 Diphenylamine, 50 g/l ethanolic solution.

6.1.8 Congo red paper.

6.1.9 Lead acetate paper.

6.2 Procedure

Weigh, to the nearest 0,1 mg, about 0,6 g of lithopone 30 % previously dried at 105 ± 2 °C or about 0,3 g of lithopone 60 % previously dried at 105 ± 2 °C into a beaker and add 25 ml of the hydrochloric acid solution (6.1.3). Immediately cover with a watch-glass and boil until the evolution of hydrogen sulphide has ceased [test with lead acetate paper (6.1.9)]. Dilute with 100 ml of water, add 5 ml of the sulphuric acid solution (6.1.4) and boil the solution again.

Allow the precipitate to settle whilst hot and filter the supernatant solution through a fine filter paper. Transfer the precipitate to the filter paper and wash with hot water containing some sulphuric acid until a drop of the washings shows no reaction with the potassium hexacyanoferrate(II) solution (6.1.1). Fold the filter paper over the precipitate. Transfer it whilst still wet to a weighed porcelain crucible and ignite it in contact with air to constant mass. The residue is assumed to be barium sulphate.[1] Add a few drops of the sulphuric acid solution to the contents of the crucible. No trace of hydrogen sulphide should be noticeable; otherwise, the sulphuric acid shall be driven off and the residue re-ignited.

Combine the washings with the filtrate. Add a slight excess of the ammonia solution (6.1.5) [verify on Congo red paper (6.1.8)], followed by the hydrochloric acid solution from a dropping bottle until a small piece of Congo red paper, when touched with the solution, just turns to a lasting red-blue or blue-red colour (pH 1,5 to 3,0).

If necessary, make up to 150 ml with water, heat the solution to boiling, add 10 drops of the diphenylamine solution (6.1.7) and immediately titrate the solution as specified for the standardization of the potassium hexacyanoferrate(II) solution in 6.1.1.2.

6.3 Expression of results

Calculate the barium sulphate content of the lithopone, expressed as a percentage by mass, by the formula

$$\frac{100\, m_2}{m_1}$$

Calculate the total zinc content of the lithopone, expressed as a percentage by mass, calculated as zinc sulphide, by the formula

$$1{,}490\,(TV_3 - cV_4)\,\frac{100}{m_1} = \frac{149}{m_1}(TV_3 - cV_4)$$

where

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

m_2 is the mass, in grams, of the residue;

T is the titre, expressed in grams of zinc per millilitre, of the potassium hexacyanoferrate(II) solution (6.1.1), as calculated in 6.1.1.3;

c is the concentration, in grams of zinc per millilitre, of the standard zinc chloride solution (6.1.2);

V_3 is the volume, in millilitres, of the potassium hexacyanoferrate(II) solution (6.1.1) required for the titration;

V_4 is the volume, in millilitres, of the standard zinc chloride solution (6.1.2) required for the back-titration.

7 Determination of the zinc oxide content

7.1 Reagents

7.1.1 Ammonium chloride.

7.1.2 Potassium hexacyanoferrate(II), standard volumetric solution $c[K_4Fe(CN)_6] \approx 0{,}05$ mol/l (titre T), prepared as specified in 6.1.1.1 and standardized as specified in 6.1.1.2.

7.1.3 Zinc chloride, standard reference solution containing approximately 5 g of zinc per litre (concentration c), prepared as specified in 6.1.2.

7.1.4 Hydrochloric acid, 4 mol/l solution.

7.1.5 Ammonia, 4 mol/l solution.

7.1.6 Diphenylamine, 50 g/l ethanolic solution.

7.1.7 Congo red paper.

7.2 Procedure

Weigh, to the nearest 1 mg, about 10 g of the lithopone previously dried at 105 ± 2 °C into a 500 ml one-mark volumetric flask. Add 4 g of the ammonium chloride (7.1.1) and 100 ml of the ammonia solution (7.1.5). Allow the suspension to stand for 1 h in the cold, shaking at intervals. After this period make the suspension up to the mark with water, shake

1) If desired, the actual barium sulphate content can be determined by fusing the residue with potassium sodium carbonate and converting the barium carbonate into barium sulphate.

and filter through an absolutely dry filter paper and funnel. Discard the first 10 to 20 ml of the filtrate and collect the remainder in a dry beaker. Transfer 250 ml of this filtrate to a beaker by means of a pipette and add to it 10 ml of the zinc chloride solution (7.1.3) followed by the hydrochloric acid solution (7.1.4) from a dropping bottle until a piece of the Congo red paper (7.1.7), when touched with the solution, turns to a lasting red-blue or blue-red colour (pH 1,5 to 3,0).

Heat the solution to boiling, add 10 drops of the diphenylamine solution (7.1.6) and immediately titrate the solution as specified for the standardization of the potassium hexacyanoferrate(II) solution in 6.1.1.2.

7.3 Expression of results

Calculate the zinc oxide content of the lithopone, expressed as a percentage by mass, by the formula

$$2{,}490\,(TV_5 - cV_6 - 10\,c)\,\frac{100}{m_3} = \frac{249}{m_3}\,(TV_5 - cV_6 - 10\,c)$$

where

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

T is the titre, expressed in grams of zinc per millilitre, of the potassium hexacyanoferrate(II) solution (7.1.2);

c is the concentration, in grams of zinc per millilitre, of the standard zinc chloride solution (7.1.3);

V_5 is the volume, in millilitres, of the potassium hexacyanoferrate(II) solution (7.1.2) required for the titration;

V_6 is the volume, in millilitres, of the standard zinc chloride solution (7.1.3) required for the back-titration.

8 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 473);

c) the results of the tests, 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 date of the tests.

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