

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 473

LITHOPONE

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BRIEF HISTORY

The ISO Recommendation R 473, *Lithopone*, was drawn up by Technical Committee ISO/TC 35, *Paints, Varnishes and Related Products and their Raw Materials*, the Secretariat of which is held by the Netherlands Normalisatie-instituut (NNI).

Work on this question by the Technical Committee began in 1950, taking into account the studies which had been made by the former International Federation of the National Standardizing Associations (ISA), and led in 1960 to the adoption of a Draft ISO Recommendation.

In April 1962, this Draft ISO Recommendation (No. 484) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies:

Austria	Italy	Romania
Burma	Japan	Spain
Canada	Netherlands	Sweden
Chile	New Zealand	Switzerland
Czechoslovakia	Portugal	United Kingdom
Germany	Republic of	U.S.S.R.
India	South Africa	Yugoslavia

Two Member Bodies opposed the approval of the Draft:

Belgium
France

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in February 1966, to accept it as an ISO RECOMMENDATION.

LITHOPONE

1. SCOPE

This ISO Recommendation establishes the more important requirements for lithopones and the methods of test for these requirements.

2. DESCRIPTIONS

2.1 Lithopone 30%. A white pigment consisting of zinc sulphide (ZnS) and barium sulphate (BaSO₄) in approximately molecular proportions. The material is a calcined co-precipitate.

2.2 Lithopone 60%.* A white pigment which contains approximately 60% zinc sulphide (ZnS) and a balance consisting mainly of barium sulphate (BaSO₄). The material is a calcined co-precipitate.

3. REQUIRED CHARACTERISTICS AND THEIR TOLERANCES

The lithopones should have the following characteristics:

Properties	Lithopone 30 %	Lithopone 60 %	Clause describing test method
Total zinc, calculated as zinc sulphide, min. %	28	58	5.1
Zinc oxide, max. %	1	1	5.2
Sum of total zinc, calculated as zinc sulphide and barium sulphate, min. %	99	99	5.1 and 5.2
Barytes	absent	absent	5.3
Matter volatile at 105 °C, max. %	0.5	0.5	5.4
Matter soluble in water, max. %	0.5	0.5	5.5
Alkalinity of the aqueous extract	equal to an agreed reference sample		5.6
Residue on sieve (63 µm), max. %	0.1	0.1	5.7
Colour	equal to an agreed reference sample		5.8
Lightening power	as agreed between purchaser and vendor		5.9
Hiding power			5.10
Oil absorption value			5.11

* There are on the market

- (1) lithopones with a content of about 40 or 50 per cent zinc sulphide. These products should be marked so as to indicate the zinc sulphide content,
- (2) 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.

4. SAMPLING

See ISO Recommendation R , *Sampling Raw Materials for Paints and Varnishes*.*

5. TEST METHODS

5.1 Determination of barium sulphate and total zinc content

5.1.1 Reagents

5.1.1.1 Potassium cyanoferrate (II),** standard volumetric solution, approximately 0.05 M (standardization factor *F*).

Dissolve 21.0 g of potassium cyanoferrate(II), 300 mg of potassium cyanoferrate(III)*** and 2 g of anhydrous sodium carbonate (to stabilize the solution) in distilled water and dilute with distilled water to 1000 ml in a graduated flask.

5.1.1.2 Zinc chloride standard solution, approximately 5 g of zinc per litre (concentration *c*). Weigh about 5 g of chemically pure zinc to the nearest 0.1 mg, dissolve in 300 ml of hydrochloric acid 4 M and dilute the solution obtained with distilled water to 1000 ml in a graduated flask.

5.1.1.3 Diphenylamine ethanolic solution, 5 g per 100 ml.

5.1.1.4 Congo paper.

5.1.1.5 Hydrochloric acid 4 M.

5.1.1.6 Sulphuric acid 2 M.

5.1.1.7 Ammonia solution ($d = 0.9$).

5.1.1.8 Ammonia solution 4 M.

5.1.1.9 Lead acetate paper.

5.1.2 Standardization of the potassium cyanoferrate (II) solution

Take 25.0 ml of the zinc chloride solution by means of a pipette and add ammonia solution 4 M until a piece of Congo paper, touched onto the solution, just turns to a pure red colour. Then carefully neutralize the solution with hydrochloric acid from a dropping bottle. Add a few drops in excess until the Congo paper turns to a lasting red-blue or blue-red colour (pH 1.5 to 3.0).

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

* At present second Draft ISO Recommendation No. 731.

** IUPAC name, formerly called potassium ferrocyanide.

*** IUPAC name, formerly called potassium ferricyanide.

Immediately titrate the solution with the potassium cyanoferrate (II) solution until the colour turns to a lasting yellow or yellowish green (V_1 millilitres being used).

Then backtitrate the solution with the zinc chloride solution until the colour just turns to blue again (V_2 millilitres being used). The standardization factor F expressed in grammes of zinc per millilitre of the potassium cyanoferrate (II) solution is

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

where c = concentration of the standard zinc chloride solution, in grammes of zinc per millilitre;

V_1 = volume of the potassium cyanoferrate (II) solution required for the titration, in millilitres;

V_2 = volume of the standard zinc chloride solution required for the back-titration, in millilitres.

5.1.3 Procedure

Weigh to the nearest 0.1 mg about 0.6 g of 30% lithopone (dried at $105 \pm 2^\circ\text{C}$) or about 0.3 g of 60% lithopone (dried at $105 \pm 2^\circ\text{C}$) (m_1 grammes) into a beaker and add 25 ml of hydrochloric acid 4 M. Immediately cover with a watch glass and boil until evolution of hydrogen sulphide has ceased (test with lead acetate paper). Dilute with 100 ml of distilled water, add 5 ml of sulphuric acid and boil again.

Allow the precipitate to settle in the heat and filter the supernatant solution through a fine filter paper. Transfer the precipitate to the filter and wash with hot distilled water containing some sulphuric acid until a drop of the washings shows no reaction with potassium cyanoferrate (II) solution. Fold the filter paper over the precipitate. Transfer while still wet to a weighed porcelain crucible and ignite in contact with air to a constant mass (the mass of the residue being m_2 grammes). The residue is assumed to be barium sulphate.* On adding a few drops of sulphuric acid to the contents of the crucible, no trace of hydrogen sulphide should be noticeable; otherwise the sulphuric acid should be driven off and the residue re-ignited.

Combine the washings with the filtrate. Add a slight excess of ammonia solution (5.1.1.7) (verify on Congo paper), followed by hydrochloric acid from a dropping bottle until a small piece of Congo paper added to 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 distilled water, heat the solution to boiling, add 10 drops of the diphenylamine solution and immediately titrate the solution in a similar manner as described for the standardization of the potassium cyanoferrate (II) solution in clause 5.1.2 (V_3 millilitres of the potassium cyanoferrate (II) solution and V_4 millilitres of the zinc chloride solution being required).

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

5.1.4 Calculation of results

The barium sulphate content of the lithopone in per cent by mass is

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

The total zinc content of the lithopone in per cent by mass, calculated as zinc sulphide, is

$$1.490 (F V_3 - c V_4) \frac{100}{m_1}$$

where

- m_1 = mass of the test portion, in grammes;
- m_2 = mass of the residue, in grammes;
- F = standardization factor of the potassium cyanoferrate (II) solution, in grammes of zinc per millilitre;
- c = concentration of the standard zinc chloride solution, in grammes of zinc per millilitre;
- V_3 = volume of the potassium cyanoferrate (II) solution required for the titration in millilitres;
- V_4 = volume of the standard zinc chloride solution required for the backtitration in millilitres.

5.2 Determination of the zinc oxide content

5.2.1 Reagents

5.2.1.1 *Potassium cyanoferrate (II)*, standard volumetric solution approximately 0.05 M (standardization factor F) prepared as described in clause 5.1.1.1.

5.2.1.2 *Zinc chloride*, approximately 5 g of zinc per litre standard solution (concentration c) prepared as described in clause 5.1.1.2.

5.2.1.3 *Diphenylamine*, ethanolic solution, 5 g per 100 ml.

5.2.1.4 *Congo paper*.

5.2.1.5 *Hydrochloric acid* 4 M.

5.2.1.6 *Ammonia solution* 4 M.

5.2.1.7 *Ammonium chloride*.

5.2.2 Standardization of the potassium cyanoferrate (II) solution

Standardize the potassium ferrocyanide solution as described in clause 5.1.2.

5.2.3 Procedure

Weigh to the nearest 1 mg about 10 g of lithopone (dried at $105 \pm 2^\circ\text{C}$) (m grammes) into a 500 ml graduated flask. Add 4 g of ammonium chloride and 100 ml of ammonia. Allow the suspension to stand for one hour in the cold with shaking at intervals. After this period make the suspension up to the mark with distilled water, shake and filter through an absolutely dry filter and funnel. Discard the first 10 to 20 ml of the filtrate and collect the remainder in a dry beaker. Remove 250 ml of this filtrate to a beaker by means of a pipette and add to it 10 ml of the zinc chloride solution followed by hydrochloric acid from a dropping bottle until a piece of Congo paper added to 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 and titrate the solution immediately as described for the standardization of the potassium cyanoferrate (II) solution in clause 5.1.2 (V_1 millilitres of potassium cyanoferrate (II) solution and V_2 millilitres of zinc chloride solution being required).