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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 275

ZINC OXYDE FOR PAINTS — Specifications and methods of test

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For each individual country the only valid standard is the national standard of that country.

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BRIEF HISTORY

The ISO Recommendation R 275, *Zinc Oxide for Paints*, was drawn up by Technical Committee ISO/TC 35, *Raw Materials for Paints, Varnishes and Similar Products*, the Secretariat of which is held by the Nederlands Normalisatie-Instituut (NNI).

Work on this question by the Technical Committee began in 1953 and led in 1958 to the adoption of a Draft ISO Recommendation.

In December 1958, this Draft ISO Recommendation (No. 266) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to some modifications of an editorial nature, by the following Member Bodies:

India	Poland
Israel	Romania
Italy	Spain
Japan	United Kingdom
Netherlands	U.S.S.R.

Six Member Bodies opposed the approval of the Draft:

Austria	Germany
Belgium	New Zealand
France	Sweden

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in November 1962, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 275 November 1962

ZINC OXYDE FOR PAINTS

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1. SCOPE

This ISO Recommendation defines the important requirements for zinc oxide for paints and the methods of test for these requirements. *

2. DEFINITION

Zinc oxide for paints is a white pigment which consists mainly of zinc oxide (ZnO).

3. REQUIRED CHARACTERISTICS AND THEIR TOLERANCES

Zinc oxide for paints should have the following characteristics:

Property	Type I	Type II	Type III	Type IV	Type V	Clause describing test method
Zinc oxide (ZnO) content %	min. 99.0	94.0 to 99.0	87.0 to 94.0	68.0 to 87.0	62.0 to 68.0	5.1
Lead content (lead compounds, expressed as lead, Pb) %	max. 0.3	0.3 to 3.0	3.0 to 8.0	8.0 to 22.0	22.0 to 27.0	5.2
Residue on sieve, max. %	0.1	0.1	0.3	0.3	0.3	5.3
Matter volatile at 105 °C, max. %	0.3	0.3	0.3	0.3	0.3	5.4
Loss on ignition at 500 °C, max. %	0.5	1.0	1.0	1.0	1.0	5.5
Matter soluble in water, max. %	0.5	1.0	1.5	1.5	1.5	5.6
Neutrality of the aqueous extract	Neutral to methyl red					5.7
Colour	To match that of the agreed sample					5.8
Lightening power	To be agreed between purchaser and vendor					5.9
Oil absorption						5.10
Hiding power						5.11

* For painting building interiors Convention No. 13 of the International Labour Organization limits the use of zinc oxides for paints to pigments containing less than 2% of lead.

4. SAMPLING

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

5. TEST METHODS

All reagents should be of analytical grade. Water should be distilled water or water of at least equal purity.

5.1 Zinc oxide content

5.1.1 *Reagents*

5.1.1.1 Aqueous ammonia, concentrated ($d = 0.9$).

5.1.1.2 Aqueous ammonia 4N.

5.1.1.3 Hydrochloric acid, concentrated ($d = 1.19$).

5.1.1.4 Hydrochloric acid 4N.

5.1.1.5 Hydrogen peroxide solution, 3%.

5.1.1.6 Hydrogen sulphide, saturated aqueous solution.

5.1.1.7 Potassium hexacyanoferrate (II) **, standard solution, approximately 0.05 molar.
Dissolve 21.0 g of potassium hexacyanoferrate (II), 300 mg of potassium hexacyanoferrate (III) *** and 2 g of anhydrous sodium carbonate (to stabilize the solution) in water, and dilute with water to 1000 ml.

5.1.1.8 Zinc chloride, standard solution, containing 5 g of zinc per litre.
Weigh accurately 5.0 g of chemically pure zinc, dissolve in 300 ml of hydrochloric acid 4N, and dilute with water to 1000 ml in a volumetric flask.

Alternatively, it may be more convenient to weigh accurately a quantity of zinc which is not exactly 5.0 g, in which case appropriate adjustment should be made in the calculations to take account of the fact that the zinc chloride solution does not contain exactly 5 g of zinc per litre.

5.1.1.9 Diphenylamine solution in ethanol, 5 g per 100 ml.

5.1.1.10 Congo paper.

5.1.1.11 Lead acetate paper.

5.1.2 *Standardization of the potassium hexacyanoferrate (II) solution*

Pipette 25.0 ml of the zinc chloride solution into a flask and add ammonia 4N until a piece of Congo paper placed in contact with the solution just turns to a pure red colour. Then carefully neutralize the solution by adding the hydrochloric acid 4N from a dropping bottle and add a few drops in excess until the Congo paper turns to a permanent blueish-red or reddish-blue colour (pH 3.0 to 1.5).

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

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* At present Draft ISO Recommendation No. 731.

** International Union of Pure and Applied Chemistry (IUPAC) name for potassium ferrocyanide, $K_4Fe(CN)_6$.

*** International Union of Pure and Applied Chemistry (IUPAC) name for potassium ferricyanide, $K_3Fe(CN)_6$.

Immediately titrate the solution with the potassium cyanoferrate solution until the colour turns to a permanent yellow or yellowish-green (V_1 ml being used).

Then backtitrate the solution with the zinc chloride solution until the colour just turns to blue again (V_2 ml being used).

The standardization factor F of the potassium cyanoferrate solution expressed in grammes of zinc per ml, is given by the following formula:

$$F = \frac{0.005\ (25 + V_2)}{V_1} \quad *$$

5.1.3 *Procedure*

Accurately weigh about 1.0 g of the zinc oxide (dried at 105 ± 2 °C) (m grammes). Mix with 15 ml of the concentrated hydrochloric acid and 30 ml of water and evaporate to dryness.

Dissolve the residue, by gently heating if necessary, in 7 ml of concentrated hydrochloric acid and 30 ml of water. Then add 75 ml of saturated hydrogen sulphide solution, heat the suspension to a temperature of 40 °C and allow to stand for one hour at this temperature.

When the lead sulphide has settled, filter the liquid into a 500 ml volumetric flask and wash the filter thoroughly with a mixture of 25 ml of the saturated hydrogen sulphide solution, 5 ml of the concentrated hydrochloric acid and 75 ml of water. Boil the filtrate and washings to expel hydrogen sulphide (test on lead acetate paper).

After cooling ** the solution, make up to 500 ml with water and shake. Pipette 100 ml of this solution into a flask and add the aqueous ammonia 4N until a piece of Congo paper, added to the solution, just turns to a pure red colour. Carefully add the hydrochloric acid 4N from a dropping bottle to neutralize the solution and add a few drops in excess until the Congo paper turns to a blueish-red or reddish-blue colour (pH 3.0 to 1.5). Heat the solution to boiling, add 10 g of diphenylamine solution and titrate at once, in a similar manner as described for the standardization of the potassium cyanoferrate solution in clause 5.1.2 (V_3 ml of the potassium cyanoferrate solution and V_4 ml of the zinc chloride solution being required).

5.1.4 *Expression of results*

The zinc oxide content (ZnO) of the sample in per cent by mass is given by the following formula:

$$\mathrm{ZnO} = 6.223\ (FV_3 - 0.005\ V_4)\ \frac{100}{m} \quad *$$

where

F = the standardization factor of the potassium cyanoferrate (II) solution,

V_3 = the volume in millilitres of the potassium cyanoferrate (II) solution used,

V_4 = the volume, in millilitres, of the standard zinc chloride solution,

m = the mass, in grammes of the test sample.

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* If the zinc content of solution 5.1.1.8 is not exactly 0.005 g/ml an appropriate correction should be applied to the factor 0.005.

** If the solution contains iron or manganese, add, after cooling, about 1 ml of 3% hydrogen peroxide and 60 ml of concentrated aqueous ammonia. Make up to 500 ml with water and allow the solution to stand for two hours. Then filter through an absolutely dry filter and funnel. Discard the first 10 to 20 ml of the filtrate. Collect the remainder in a dry flask, remove 100 ml by means of a pipette and boil this to expel ammonia. Then acidify this solution with hydrochloric acid 4N until a piece of Congo paper turns to a blueish red or a reddish blue (pH 3.0 to 1.5). 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 solution in clause 5.1.2 (V_3 ml of the potassium cyanoferrate solution and V_4 ml of the zinc chloride solution being used). For the calculation, see under clause 5.1.4.

5.2 Lead content

5.2.1 *Reagents*

5.2.1.1 Acetic acid solution, 2 g per 100 ml.

5.2.1.2 Hydrochloric acid 4N.

5.2.1.3 Ammonium acetate solution 2N.

5.2.1.4 Potassium dichromate solution, 5 g per 100 ml, freshly prepared.

5.2.1.5 Congo paper.

5.2.2 *Procedure*

Accurately weigh about 5.0 g (m grammes) of zinc oxide of type I or II or about 1.5 g of zinc oxide of type III, IV or V (always dried at 105 ± 2 °C), transfer to an approximately 300 ml beaker, and dissolve by heating in 35 ml of the hydrochloric acid. Add to this solution the ammonium acetate solution until the liquid gives no acid reaction to Congo paper.

Filter off any insoluble matter present, and wash thoroughly with the ammonium acetate solution 2N. Dilute the filtrate, combined with the washings, to about 200 ml with water, and then heat to boiling.

Precipitate the lead from the boiling solution as lead chromate, by adding an excess of the potassium dichromate solution. Keep the liquid boiling until the precipitate has turned to dark orange-red. Then keep the liquid hot for 1½ to 2 hours on a water bath.

After cooling, filter the precipitate through a weighed filter crucible (porosity 5 to 10 μm). Wash with 2% acetic acid, then with water, and dry in an oven at a temperature of 105 ± 2 °C or in a vacuum desiccator to constant mass.

5.2.3 *Expression of results*

The lead content (Pb) of the test sample, in per cent by mass, is given by the following formula:

$$\mathrm{Pb} = \frac{63.75\,p}{m}$$

where

p = the mass in grammes of the dried precipitate,

m = the mass in grammes of the test sample.

5.3 Residue on sieve

Refer to the ISO Recommendation R , * *General Methods of Test for Pigments.*

5.4 Matter volatile at 105 °C

Refer to the ISO Recommendation R , * *General Methods of Test for Pigments.*

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* At present Draft ISO Recommendation No. 832.

5.5 Loss on ignition at 500 °C

5.5.1 *Procedure*

Accurately weigh about 2.5 g (m grammes) of zinc oxide, previously dried at 105 ± 2 °C, into a weighed porcelain crucible and ignite at a maximum temperature of 500 °C (dark red heat). Let the crucible cool in a desiccator and weigh.

5.5.2 *Expression of results*

The loss on ignition at 500 °C (L) of the test sample in per cent by mass is given by the following formula:

$$L = \frac{q}{m} \times 100$$

where

q = the loss in mass in grammes of the test sample after ignition at 500 °C,

m = the mass in grammes of the test sample.

5.6 **Matter soluble in water** (hot extraction method)

Refer to ISO Recommendation R ..., * *General Methods of Test for Pigments.*

5.7 Test for neutrality

Add methyl red to the filtrate mentioned under clause 5.6, and observe the colour.

5.8 Comparison of colour

5.9 Lightening power

5.10 Oil absorption

Refer to ISO Recommendation R , * *General Methods of Test for Pigments.*

5.11 Hiding power

Under consideration.

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* At present Draft ISO Recommendation No. 832.

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