INTERNATIONAL
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Zinc dust pigments for paints — Specifications and test methods

Pigments à base de poussière de zinc pour peintures — Spécifications et méthodes d'essai

Reference number
ISO 3549:1995(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 3549 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 3549:1976), which has been technically revised.

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Zinc dust pigments for paints — Specifications and test methods

1 Scope

This International Standard specifies the requirements and corresponding test methods for zinc dust pigments suitable for use in protective coatings.

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 565:1990, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings.*

ISO 594-1:1986, *Conical fittings with a 6 % (Luer) taper for syringes, needles and certain other medical equipment — Part 1: General requirements.*

ISO 842:1984, *Raw materials for paints and varnishes — Sampling.*

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

3 Definition

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

3.1 zinc dust pigment: A fine grey powder of essentially spheroidal particles, mainly consisting of metallic zinc.

NOTE 1 Zinc dust pigments for paints may vary in their metallic zinc content, chemical purity, particle shape, particle size distribution, mean and maximum diameter, etc. These variations are all likely to have an influence on the zinc dust behaviour in paints with regard to parameters such as dispersibility, fineness of grind, reactivity, electrical conductivity and packing properties.

4 Required characteristics and tolerances

4.1 For zinc dust pigments complying with this International Standard, the essential requirements are specified in tables 1 and 2.

Table 1 — Composition of zinc dust pigment

Characteristic	Unit	Requirement	Test method
Total zinc content	% (*m/m*)	min. 98	See clause 7
Metallic zinc content	% (*m/m*)	min. 94	See clause 8
Lead (Pb) content	% (*m/m*)	max. 0,2	See clause 9
Cadmium (Cd) content	% (*m/m*)	max. 0,1	See clause 9
Iron (Fe) content	% (*m/m*)	max. 0,05	See clause 9
Arsenic (As) content	% (*m/m*)	max. 0,000 5	See clause 10
Chloride (Cl) content)	% (*m/m*)	max. 0,005	See clause 11
Matter insoluble in acid	% (*m/m*)	max. 0,05	See clause 12

NOTE — If the zinc oxide content is required, this can be calculated by multiplying the difference between the total zinc content and the metallic zinc content by 1,244 7.

Table 2 — Residue on sieve

Nominal size of sieve aperture µm	Residue on sieve % (*m/m*)	Test method
125	max. 0,01	See clause 6
90	max 0,1.	
45	max. 5	

4.2 Requirements for other physical properties (surface area, particle size distribution, etc.) and the choice of reference pigment to which these properties refer shall be the subject of agreement between the interested parties.

4.3 The reference pigment shall also comply with the requirements given in tables 1 and 2.

5 Sampling

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

WARNING — The sample shall on no account be dried before testing, and any portion of the sample not used shall not be returned to the sample container after having been manipulated.

6 Determination of residue on sieve

6.1 Principle

A suitable test portion of the sample is passed through an air-jet sieve apparatus having sieves with nominal mesh apertures of 45 µm, 90 µm and 125 µm. The residue on each of these sieves is determined.

6.2 Apparatus

6.2.1 Sieves, circular, with a sieving area of diameter 200 mm and with nominal mesh apertures of 45 µm, 90 µm and 125 µm, complying with ISO 565. A transparent lid shall be provided to cover the sieve during use.

6.2.2 Air-jet sieve apparatus (see figure 1), consisting of a cylindrical casing which contains a sieve (see 6.2.1). The base of the casing has an outlet (to which an extractor fan is connected) and an air inlet to permit the injection of air.

The air inlet is connected to a jet rotating at 20 r/min to 25 r/min and consists of a slot-shaped nozzle located beneath and very close to the sieve (see figure 1). When the jet rotates, it blows air continuously through the sieve, preventing the powder particles from settling. The air is extracted through the outlet, drawing the finer particles through the sieve. The flow of air is controlled by adjusting a slot at the outlet.

The vacuum obtained shall be 1 250 Pa or better.

6.2.3 Timer (for example a stopwatch), recording to the nearest 1 s or better. It may be equipped with a switch for stopping the motor of the sieve apparatus (6.2.2).

6.2.4 Analytical balance, capable of weighing at least 50 g to the nearest 1 mg.

6.2.5 Mallet, of light construction, with a plastic head, suitable for tapping the apparatus to dislodge powder adhering to the lid and sieve.

6.2.6 Clean brush.

6.2.7 Stainless-steel boat.

6.3 Checking and cleaning the sieve

Check that the sieve is clean and undamaged and is not blocked by material used in a previous determination. A magnifying glass of sufficient magnification is recommended to aid this inspection.

If cleaning of the sieve is necessary, an ultrasonic system is recommended for this purpose. It is also possible to clean the sieve by turning it upside down on a clean sheet of paper and tapping vigorously to eliminate any residual particles.

6.4 Procedure

Carry out the determination in duplicate.

6.4.1 Weigh, to the nearest 0,01 g, a test portion of approximately 50 g (m_0).

6.4.2 Secure the 45 µm sieve (6.2.1) in position in the sieve apparatus (6.2.2) and transfer the test portion to the sieve.

6.4.3 Cover the sieve with the transparent lid, switch on the extractor fan and sieve apparatus (6.2.2) and tap the lid and the sieve lightly from time to time with the mallet (6.2.5) to distribute the material and dislodge adhering particles.

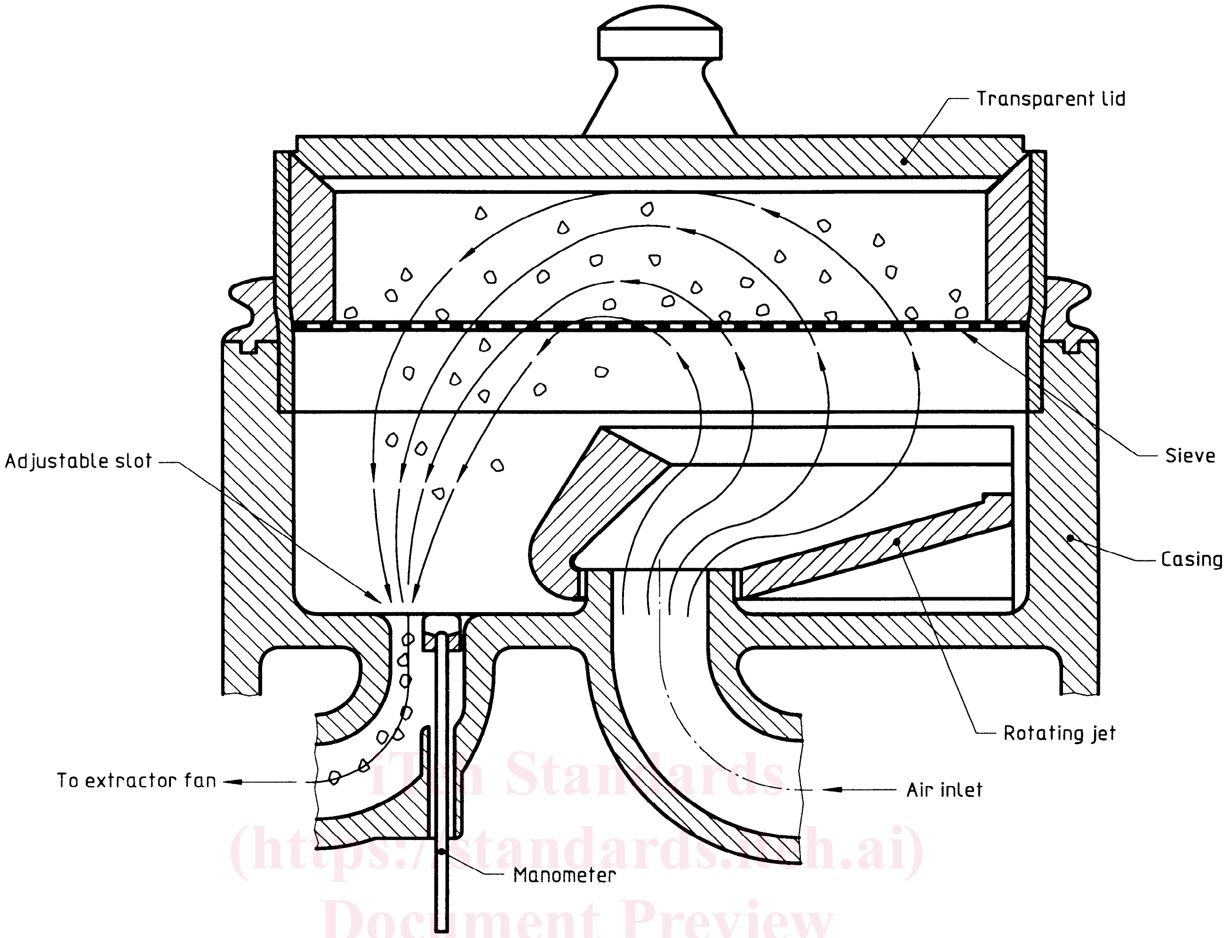

Figure 1 — Air-jet sieve apparatus

6.4.4 After 120 s, switch off the apparatus and remove the transparent lid and sieve. Recover the sieve residue by turning the sieve upside down on a clean sheet of paper and tapping vigorously to ensure that all particles are recovered.

6.4.5 Weigh the stainless-steel boat (6.2.7) to the nearest 1 mg (m_1). Transfer the sieve residue to it and reweigh to the nearest 1 mg (m_2).

6.4.6 Repeat the operations described in 6.4.1 to 6.4.5 with the 90 µm sieve and then with the 125 µm sieve on fresh 50 g portions of the sample.

6.5 Expression of results

Calculate the residue on each sieve, R, expressed as a percentage by mass, using the equation

$$R = \frac{(m_2 - m_1)}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the steel boat;

m_2 is the mass, in grams, of the boat and the sieve residue.

If the two results (duplicates) for each sieve differ by more than one tenth of the maximum limit for that sieve in table 2, repeat the procedure.

Calculate the mean of two valid results (replicates) and report the result to three decimal places for the 125 µm sieve, two decimal places for the 90 µm sieve and one decimal place for the 45 µm sieve.

7 Determination of total zinc content

7.1 Principle

The zinc is titrated against EDTA solution.

7.2 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.

7.2.1 Hydrochloric acid, concentrated, approximately 38 % (*m/m*), $\rho \approx 1{,}19$ g/ml.

7.2.2 Hydrochloric acid, diluted 1 + 4.

Dilute 200 ml of concentrated hydrochloric acid (7.2.1) to 1 litre with water.

7.2.3 Sulfuric acid, diluted 1 + 1.

Slowly add, whilst cooling, 1 part by volume of concentrated sulfuric acid [approximately 96 % (*m/m*), $\rho \approx 1{,}84$ g/ml] to 1 part by volume of water.

7.2.4 Nitric acid, concentrated, approximately 68 % (*m/m*), $\rho \approx 1{,}42$ g/ml.

7.2.5 Ammonia solution, approximately 25 % (*m/m*), $\rho \approx 0{,}81$ g/ml.

7.2.6 Buffer solution.

Dissolve 200 g of hydroxylammonium chloride ($NH_2OH{\cdot}HCl$) in approximately 300 ml of water. Dissolve 28 g of sodium hydroxide in approximately 300 ml of water. Combine the two solutions, cool and dilute to 1 litre with water.

7.2.7 (Ethylenedinitrilo)tetraacetic acid (EDTA), disodium salt dihydrate, standard volumetric solution.

7.2.7.1 Preparation

Weigh, to the nearest 1 mg, about 20 g of disodium EDTA dihydrate, dissolve in water and make up to 1 litre. Store in a polyethylene bottle.

7.2.7.2 Standardization

Weigh, to the nearest 1 mg, approximately 1,5 g of 99,99 % pure zinc metal (mass m_0) into a 250 ml beaker. Dissolve in 20 ml of hydrochloric acid (7.2.1) and add 1 ml or 2 ml of nitric acid (7.2.4). After complete dissolution, transfer quantitatively into a 500 ml volumetric flask, make up to the mark with water and mix thoroughly.

Pipette 50 ml of the zinc solution into a 500 ml conical flask. Add 200 ml of water and 3 drops of bromothymol blue indicator (7.2.9), followed by ammonia solution (7.2.5), drop by drop, until the colour changes to blue. Then add dilute hydrochloric acid (7.2.2) until the yellow colour returns. Finally add 20 ml of buffer solution (7.2.6) and 3 drops of xylenol orange indicator (7.2.8) and titrate with EDTA solution (7.2.7.1) until the red colour changes to pale yellow.

Calculate the zinc equivalent of the EDTA solution, ρ(Zn), expressed in grams per millilitre (i.e. the mass, in grams, of zinc forming a complex with 1 ml of EDTA solution), using the equation

$$\rho(\mathrm{Zn}) = \frac{m_0 \times 50}{500 \times V_0} = \frac{m_0}{10 \times V_0}$$

where

m_0 is the mass, in grams, of zinc metal used;

V_0 is the volume, in millilitres, of EDTA solution used for the titration.

Carry out the titration three times and calculate the mean value of the zinc equivalent ρ(Zn), in grams per millilitre.

7.2.8 Xylenol orange, 1g/l indicator solution of the sodium salt in water.

7.2.9 Bromothymol blue, 1 g/l indicator solution in 96 % (*V/V*) ethanol.

7.3 Procedure

7.3.1 Test portion

Weigh, to the nearest 1 mg, 1,5 g of the sample (m_1) into a 250 ml beaker.

7.3.2 Determination

Carry out the determination in duplicate.

Dissolve the test portion in 20 ml of concentrated hydrochloric acid (7.2.1) and add 1 ml or 2 ml of nitric acid (7.2.4) to ensure that any lead present is dissolved. Add 5 ml of sulfuric acid (7.2.3) and evaporate until dense white fumes appear. Cool, then add 100 ml of water, boil for a short time and allow to stand until the following day.

Filter the solution through a sintered-glass or silica filter crucible (P40 porosity); wash the beaker and the crucible, collecting the filtrate in a 500 ml one-mark volumetric flask. Make the solution up to the mark and mix thoroughly.

Pipette 50 ml of the solution into a 500 ml conical flask. Add 200 ml of water and 3 drops of bromothymol blue indicator (7.2.9), followed by ammonia solution (7.2.5), drop by drop, until the colour changes to blue. Then add dilute hydrochloric acid (7.2.2) until the yellow colour returns. Finally add 20 ml of buffer solution (7.2.6) and 3 drops of xylenol orange indicator (7.2.8) and titrate with EDTA solution (7.2.7) until the red colour changes to pale yellow.