INTERNATIONAL STANDARD 591

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Titanium dioxide pigments for paints

Pigments de dioxyde de titane pour peintures

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FOREWORD

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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 591 was drawn up by Technical Committee ISO/TC 35, *Paints and varnishes*, and was circulated to the member bodies in May 1975.

It has been approved by the member bodies of the following countries :

Austria	Israel	Sweden
Brazil	Mexico	Switzerland
Bulgaria	New Zealand	Turkey
France	Poland	United Kingdom
Germany	Portugal	Yugoslavia
India	Romania	
Iran	South Africa, Rep. of	

The member body of the following country expressed disapproval of the document on technical grounds :

Netherlands

This International Standard cancels and replaces ISO Recommendation R 591-1967, of which it constitutes a technical revision.

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Titanium dioxide pigments for paints

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1 SCOPE AND FIELD OF APPLICATION

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

2 REFERENCES

ISO 787, *General methods of test for pigments.*

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

3 DESCRIPTION

The material shall consist essentially of titanium dioxide (TiO_2) of the anatase or the rutile crystal structure, as determined by X-ray examination. The material shall be in the form of a soft dry powder or in such a condition that it may be readily reduced thereto by crushing under a palette knife, without any grinding action.

4 CLASSIFICATION

4.1 Types

This International Standard covers two types of titanium dioxide pigments as follows :

Type A : Anatase type

Type R : Rutile type

4.2 Grades

The pigments are further classified into the following grades :

Grade A1 **Grade A2**	Type A
Grade R1 **Grade R2** **Grade R3**	Type R

5 REQUIRED CHARACTERISTICS AND THEIR TOLERANCES

Titanium dioxide pigments shall have the characteristics indicated for their type and grade in the table. The requirement for matter volatile at 105 °C after preconditioning shall only apply if this characteristic is specified by the interested parties or in a contract.

6 SAMPLING

A representative sample of the pigment to be tested shall be taken as specified in ISO 842.

TABLE – **Required characteristics**

Characteristic	Requirements for Type A		Type R			Test method
	A1	A2	R1	R2	R3	
TiO_2 content, % min.	98	92	97	90	80	Clause 7 or other agreed method*
Colour	Closely matching that of an agreed sample					ISO 787 Part I**
Lightening power	Similar to that of an agreed sample					ISO 787 Part XVII**
Matter volatile at 105 °C at point of acceptance, % max.	0,5	0,8	0,5	To be agreed		ISO 787 Part II
Matter volatile at 105 °C after 24 h preconditioning at 23 ± 2 °C and (50 ± 5) % RH, % max.	0,5	0,8	0,5	1,5	2,5	ISO 787 Part II
Matter soluble in water, % max.	0,6	0,5	0,6	0,5	0,7	ISO 787 Part III***
pH of aqueous suspension	Similar to that of an agreed sample					ISO 787 Part IX
Oil absorption	Similar to that of an agreed sample					ISO 787 Part V
Residue on sieve, 45 μm, % max.	0,10	0,10	0,10	0,10	0,10	ISO 787 Part XVIII or other agreed method
Resistivity of aqueous extract	–	Similar to that of an agreed sample	–	Similar to that of an agreed sample		ISO 787 Part XIV

* Other methods which may be used by agreement are, for example, the modified Nakazono method, the cadmium reductor method, and the Jones reductor method.

** A palette knife of plastics material should be used. This is to be included as an alternative in the revision of ISO 787, Parts I and VII.

*** If necessary, a coagulating agent should be used.

7 DETERMINATION OF TITANIUM DIOXIDE CONTENT

7.1 Interferences

Chromium, vanadium, molybdenum and niobium impurities may affect the results of this determination; these impurities may be present in commercial pigments, but normally in very small quantities only.

7.2 Reagents

All reagents shall be of analytical grade. The water used shall be distilled water or water of equivalent purity.

7.2.1 Ammonium sulphate.

7.2.2 Carbon dioxide or **nitrogen.**

7.2.3 Sulphuric acid, concentrated (ρ 1,84 g/ml).

7.2.4 Sulphuric acid, 100 g/l solution.

7.2.5 Sulphuric acid, 40 g/l solution.

7.2.6 Sulphuric acid, 20 g/l solution.

7.2.7 Potassium thiocyanate, 100 g/l solution.

7.2.8 Zinc amalgam, 3 % (*m/m*) prepared as follows :

CAUTIONARY NOTE – The following operations shall be carried out in a fume cupboard.

Place 50 ml of mercury in a small porcelain dish on a steam bath and cover the surface of the mercury with the sulphuric acid solution (7.2.6). Add 20 to 30 g of zinc in small granules. Stir from time to time and replenish the dilute acid with water as required. When all the solid zinc has disappeared, allow the amalgam to cool and stand for several hours.

Finally, filter through a Gooch crucible with no asbestos pad. Preserve the amalgam in a small bottle under the sulphuric acid solution (7.2.6). 50 ml of it will serve for many reductions and, when exhausted, may be reactivated by adding further quantities of zinc in the same way.

7.2.9 Ammonium iron(III) sulphate, 0,062 5 N standard volumetric solution, standardized against a sample of known titanium dioxide content (TiO_2) by the procedure given in 7.4.

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Dimensions in millimetres

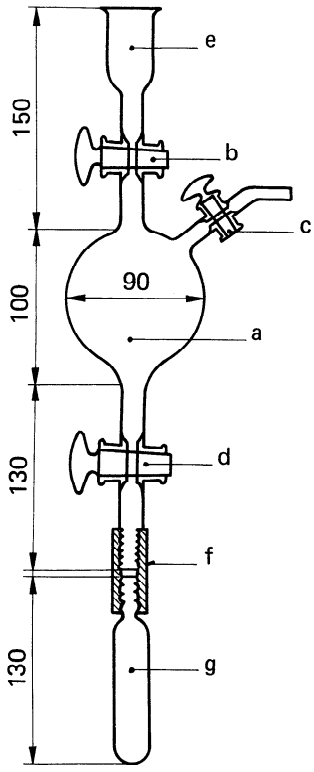

FIGURE — **Nakazono reductor**

7.3 Apparatus

Nakazono reductor (see figure), consisting of a bulb (a) with a capacity of about 350 ml with three stopcocks (b, c, d) attached. Stopcocks (b) and (d) are diametrically opposite and the extension (e) is so shaped as to facilitate the pouring of solutions into the bulb. To the lower stopcock (d) can be attached, by means of thick rubber tubing (f), a small flask (g) of capacity about 50 ml. The third stopcock (c), which is smaller in bore than either of the other two, is attached to the central sphere near the upper stopcock (b) and serves to admit carbon dioxide or nitrogen (7.2.2).

7.4 Procedure

Weigh, into a 250 ml beaker to the nearest 0,1 mg, approximately 0,2 g of the sample, previously dried as for the determination of volatile matter at 105 °C. Add 20 ml of the sulphuric acid (7.2.3) and 10 g of the ammonium sulphate (7.2.1). Mix carefully and cover with a watch glass, then heat on a heating plate until copious fumes are evolved.

Continue heating at a low heat until the pigment is completely dissolved (generally this is completed after boiling for a few minutes) or until it is clear that the remainder is composed of silica or siliceous material. Cool the solution, dilute with 100 ml of water, stir and filter if necessary. Wash the insoluble residue with the cold sulphuric acid solution (7.2.4).

Attach the flask (g) to the stopcock (d) and, with the stopcocks (b) and (d) open, pour into the extension (e) sufficient of the sulphuric acid solution (7.2.5) to fill the flask (g) and leave no air space below the stopcock (d).

Close the stopcock (d) and add 20 ml of the zinc amalgam (7.2.8) to the bulb (a) through the extension (e) and the stopcock (b). Heat the solution or filtrate, which should not exceed 300 ml in volume, to 50 °C and pour it into the extension (e). Attach the stopcock (c) to a source of the carbon dioxide or nitrogen (7.2.2). Pass the inert gas for 3 min and close stopcocks (b) and (c).

Remove the whole apparatus from the stand after disconnecting the inert gas supply and shake vigorously for 5 min, holding the apparatus in such a way that the stopcocks are kept shut.

The characteristic violet coloration of trivalent titanium is formed. Then, holding the apparatus upright, open the stopcock (d) and allow the zinc amalgam to flow into the flask (g). Do this cautiously, closing the stopcock (d) as soon as the amalgam has passed. Remove the flask (g) and the rubber tubing (f) and retain the amalgam for another determination.

Re-connect the inert gas supply, open the stopcock (c) and then the stopcock (b). Add 10 ml of the potassium thiocyanate solution (7.2.7) to the bulb (a) and titrate with the ammonium iron(III) sulphate solution (7.2.9) until a faint pink coloration remains for 1 min.

Facilitate the titration by attaching a piece of very narrow glass tubing to the tip of the burette with a piece of rubber tubing. The extension passes through the stopcock (b) into the bulb (a).

7.5 Expression of results

The titanium dioxide content (TiO_2), expressed as a percentage by mass, is given by the formula

$$\frac{0{,}50\ V}{m}$$

where

V is the volume, in millilitres, of ammonium iron(III) sulphate solution (7.2.9) required for the titration;

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

8 TEST REPORT

The test report shall contain at least the following information :

a) a reference to this International Standard or to a corresponding national standard;

b) the type and identification of the product tested;

c) the results of the tests and whether or not the product tested complies with the relevant specification limits;

d) any deviation, by agreement or otherwise, from the procedure specified;

e) the date of the tests.

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