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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION
R 591

TITANIUM DIOXIDE FOR PAINTS

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BRIEF HISTORY

The ISO Recommendation R 591, *Titanium Dioxide for Paints*, 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 Stichting Nederlands Normalisatie-instituut (NNI).

Work on this question by the Technical Committee began in 1950 and led, in 1963, to the adoption of a Draft ISO Recommendation.

In April 1965, this Draft ISO Recommendation (No. 798) 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:

Argentina	Germany	Netherlands
Austria	India	Portugal
Brazil	Ireland	Spain
Canada	Israel	Sweden
Chile	Italy	Turkey
Czechoslovakia	Japan	U.A.R.
Denmark	Korea, Rep. of	United Kingdom
France	Morocco	Yugoslavia

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in July 1967, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 591 July 1967

TITANIUM DIOXIDE FOR PAINTS

1. SCOPE

This ISO Recommendation relates to titanium dioxide pigments, for use in paints, of the following types:

Type A — Anatase type titanium dioxide,
Type R — Rutile type titanium dioxide.

2. DESCRIPTION

2.1 Type A — Anatase type titanium dioxide

The material should consist essentially of titanium dioxide (TiO_2) of the anatase crystal structure as determined by X-ray examination or, as an indication only, by the determination of relative density.

The relative density of pure titanium dioxide of anatase crystal structure is 3.9, but most commercial pigments have densities lower than this. The material should 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.

2.2 Type R — Rutile type titanium dioxide

The material should consist essentially of titanium dioxide (TiO_2) of the rutile crystal structure as determined by X-ray examination or, as an indication only, by the determination of relative density.

The relative density of pure titanium dioxide of rutile crystal structure is 4.2, but most commercial pigments have densities lower than this. The material should 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.

3. REQUIRED CHARACTERISTICS AND THEIR TOLERANCES

Both types of titanium dioxide should have the following characteristics:

TABLE

Characteristics	Requirements	Clause describing test method
Titanium dioxide content % min.	90	5.1
Colour	Equal to that of an agreed reference sample	5.2
Lightening power	Equal to that of an agreed reference sample	5.3
Matter volatile at 105 °C % max.	0.75	5.4
Matter soluble in water % max.	0.5, or not more than that of the agreed reference sample	5.5
Acidity or alkalinity of the aqueous extract, max.	20 ml of 0.1 N solution per 100 g of pigment	5.6
Oil absorption value, grammes per 100 g of pigment	Within 10% of that of an agreed reference sample or, alternatively, between 12 and 35 g	5.7
Residue on sieve (63 μm) % max.	0.1	5.8

4. SAMPLING

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

5. METHODS OF TEST

5.1 Determination of titanium dioxide content

5.1.1 *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.

5.1.2 *Reagents.* All reagents should be of analytical quality. The water used should be distilled water or water of equal purity.

5.1.2.1 *Ammonium sulphate.*

5.1.2.2 *Carbon dioxide or nitrogen.*

5.1.2.3 *Sulphuric acid*, concentrated ($d = 1.84$).

5.1.2.4 *Sulphuric acid*, 40 g per litre.

5.1.2.5 *Sulphuric acid*, 20 g per litre.

5.1.2.6 *Zinc amalgam*, 3 % m/m prepared as below:

Place 50 ml of mercury in a small porcelain dish on a steam bath, covering the surface of the mercury with sulphuric acid (5.1.2.5). Add 21 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 sulphuric acid (5.1.2.5). 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.

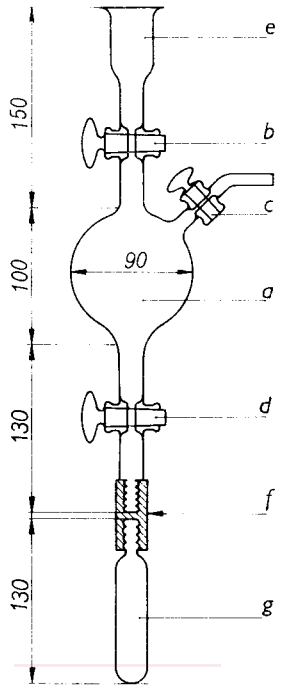
5.1.2.7 *Ammonium iron(III) sulphate*, 0.0625 N standard solution, standardized against a sample of known titanium dioxide content (TiO_2) by the procedure given in clause 5.1.4.

5.1.2.8 *Potassium thiocyanate solution*, 100 g per litre.

* At present Draft ISO Recommendation No. 731 (2).

5.1.3 *Apparatus*

Nakazono reductor (See Figure below) 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 bottom stopcock (*d*) can be attached, by means of thick rubber tubing (*f*), a small flask (*g*) of about 50 ml capacity. The third stopcock (*c*), which is smaller in bore than either of the other two, is attached to the central sphere near upper stopcock (*b*) and serves to admit carbon dioxide or nitrogen.



Dimensions in millimetres

FIGURE. — **Nakazono Reductor**

5.1.4 *Procedure*

Weigh, to the nearest 0.1 mg, approximately 0.2 g of the previously dried sample into a 250 ml beaker. Add 20 ml of the sulphuric acid (5.1.2.3) and 10 g of the ammonium sulphate (5.1.2.1). 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 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 distilled water, stir and filter if necessary.

Attach the flask (*g*) to stopcock (*d*) and with stopcocks (*b*) and (*d*) open, pour into the extension (*e*) sufficient of the sulphuric acid (5.1.2.4) to fill the flask (*g*) and leave no air space below stopcock (*d*).

Close the stopcock (*d*) and add 20 ml of the zinc amalgam (5.1.2.6) 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 into the extension (*e*). Attach stopcock (*c*) to a source of carbon dioxide or nitrogen. Pass the inert gas for three minutes and close stopcocks (*b*) and (*c*).