

ICS 87.060.10

G 54

GB

National Standard of the People's Republic of China

GB/T 1706—2006

Replaces GB 1706—1993

二氧化钛颜料

Titanium Dioxide Pigments

*(ISO 591-1:2000, Titanium dioxide pigments for paints —
Part 1: Specifications and methods of test, MOD)*

Issued on: 2006-09-01

Implemented on: 2007-02-01

**Issued by: General Administration of Quality Supervision, Inspection and
Quarantine of the People's Republic of China
Standardization Administration of the People's Republic of China**

Foreword

This Standard modifies and adopts the international standard ISO 591-1:2000 “Titanium dioxide pigments for paints — Part 1: Specifications and methods of test” (English version).

This Standard has been re-drafted in accordance with ISO 591-1:2000 “Titanium dioxide pigments for paints — Part 1: Specifications and methods of test”.

When adopting the international standard, certain technical differences have been made. These differences are indicated by a vertical single line in the margin of the relevant clauses. A summary of the technical differences and the reasons therefor is given in Annex A for reference.

Compared with ISO 591-1:2000, the main technical differences in this Standard are as follows:

- Method B — Chromium(II) reduction method for the determination of TiO_2 content has been deleted;
- The test methods adopted in this Standard are the current Chinese national standards. The great majority of these methods are technically equivalent to the corresponding international standards;
- Clauses 8 “Judgment of test results” and 9 “Marking, packaging, transport and storage” have been added;
- The foreword of the international standard has been deleted.

This Standard replaces GB 1706—1993 “Titanium dioxide pigments”, and constitutes a technical revision and editorial modification thereof.

Compared with GB 1706—1993, the main technical differences are as follows:

- The products in this Standard are divided into 2 types and 5 grades (the 1993 edition classified products into 2 types and 3 grades, with each grade further divided into 3 classes);
- This Standard adopts an agreed “reference sample” (the 1993 edition used a selected “standard sample”);
- The A1 and R1 grades in this Standard do not control the “aqueous extract resistivity” item (the 1993 edition required this item for all 3 grades);
- The indicators for “color”, “scattering power”, “pH of aqueous suspension”, “oil absorption” and “aqueous extract resistivity” in this Standard are all “agreed” (the 1993 edition specified concrete values);
- Method B — Zinc amalgam reduction method for the determination of TiO_2 content has been deleted;
- The “color determination — colorimetric method” has been added;
- The item “scattering power” replaces the item “tinting strength”.

Annex A of this Standard is an informative annex.

This Standard was proposed by the China Petroleum and Chemical Industry Association.

This Standard is under the jurisdiction of the National Technical Committee on Paints and Pigments Standardization.

Main drafting organizations of this Standard: CNOOC Changzhou Paint & Coatings Chemical Research Institute, Panzhihua Iron and Steel Group Co., Ltd. Titanium Industry Company, Sichuan Longxing Titanium Industry Co., Ltd., Zhenjiang Titanium Dioxide Powder Co., Ltd., Changzhou Wujin Shenwu Titanium Dioxide Plant.

Organizations participating in the drafting: Chongqing Yuhong Titanium Dioxide Powder Co., Ltd., Nanjing Titanium Dioxide Chemical Co., Ltd., Shanghai Coking & Chemical Co., Ltd. Titanium Dioxide Branch, Changzhou Changjiang Titanium Dioxide Plant, Zibo Zibo Iron and Steel Co., Ltd., Guangzhou Titanium Dioxide Plant, Shanghai Research Institute of Coatings.

Main drafters of this Standard: Shen Surong, Zhao Ling, Huang Guoxin, Qin Yuming, Li Hui, Guo Jianhua.

This Standard was first issued in 1979, first revised in 1988, second revised in 1993, and this is the third revision.

1 Scope

This Standard specifies the product classification, requirements, test methods, judgment of test results, and marking, packaging, transport and storage of titanium dioxide pigments.

This Standard applies to titanium dioxide pigments produced by the sulfate process or the chloride process. These products are mainly used in paints, rubber, plastics, inks, papermaking and other industries.

2 Normative References

The following documents contain provisions which, through reference in this Standard, constitute provisions of this Standard. For dated references, subsequent amendments (excluding corrections) or revisions do not apply; however, parties who reach an agreement based on this Standard are encouraged to investigate the possibility of applying the most recent editions of the documents. For undated references, the latest edition applies.

- **GB/T 1250** Rules for the expression and judgment of limiting values
- **GB/T 1717—1986** Determination of pH value of an aqueous suspension of pigments (eqv ISO 787-9:1981)
- **GB/T 1864—1989** Comparison of colour of pigments (eqv ISO 787-1:1982)
- **GB/T 3186** Paints, varnishes and raw materials for paints and varnishes — Sampling (GB/T 3186—2006, ISO 15528:2000, IDT)
- **GB/T 5211.2—2003** Determination of matter soluble in water of pigments — Hot extraction method (ISO 787-3:2000, General methods of test for pigments and extenders — Part 3: Determination of matter soluble in water — Hot extraction method, IDT)
- **GB/T 5211.3—1985** Determination of matter volatile at 105 °C of pigments (eqv ISO 787-2:1981)
- **GB/T 5211.12** Determination of resistivity of aqueous extract of pigments (GB/T 5211.12—1986, eqv ISO 787-14:1973)
- **GB/T 5211.14—1988** Determination of residue on sieve of pigments — Mechanical flushing method (eqv ISO 787-18:1983)
- **GB/T 5211.15—1988** Determination of oil absorption of pigments (eqv ISO 787-5:1980)
- **GB/T 5211.20—1999** Comparison of the colour of white, black and coloured pigments on a pure substrate — Colorimetric method (eqv ISO 787-25:1993)
- **GB/T 13451.2—1992** Determination of the relative tinting strength and the relative scattering power of coloured pigments and white pigments — Photometric method (eqv ISO 787-24:1985)

3 Terms and Definitions

This Standard adopts the following terms and definitions.

3.1 titanium dioxide pigments

Pigments composed of titanium dioxide (TiO₂) whose crystal structure, as determined by X-ray diffraction, is mainly anatase or rutile.

4 Product Classification

4.1 Type

This Standard covers the following two types of titanium dioxide pigments:

Type A: anatase;

Type R: rutile.

4.2 Grade

Pigments are further divided into the following grades:

Type A: A1, A2.

Type R: R1, R2, R3.

5 Requirements and Tolerance Limits

5.1 Appearance

The product shall be a soft dry powder, or be easily crushed with a spatula without grinding.

5.2 Other characteristics

5.2.1 Titanium dioxide pigments complying with this Standard shall meet the basic requirements given in Table 1, and the conditional requirements given in Table 2. The conditional requirements shall be agreed between the parties concerned.

5.2.2 The reference sample specified in Table 2 shall conform to the requirements in Table 1.

Table 1 — Basic Requirements

	Requirements				
	Type A		Type R		
Characteristic	A1	A2	R1	R2	R3
TiO ₂ content (mass fraction) / % ≥	98	92	97	90	80
Volatile matter at 105 °C (mass fraction) / % ≤	0.5	0.8	0.5	agreed	agreed
Matter soluble in water (mass fraction) / % ≤	0.6	0.5	0.6	0.5	0.7
Residue on 45 µm sieve (mass fraction) / % ≤	0.1	0.1	0.1	0.1	0.1

Table 2 — Conditional Requirements

	Requirements				
	Type A		Type R		
Characteristic	A1	A2	R1	R2	R3
Colour ^a	Similar to the agreed reference sample (see 5.2.2)				
Scattering power	agreed				
Volatile matter at 105 °C (mass fraction) / % ≤ (after 24 h preconditioning at (23 ± 2) °C and (50 ± 5) % RH) ^b	0.5	0.8	0.5	1.5	2.5
pH of aqueous suspension	agreed				
Oil absorption	agreed				

Aqueous extract resistivity	agreed	—	agreed		

^a The reference sample used for the determination shall be a sample agreed by the parties concerned.

^b See 5.2.1.

6 Sampling

Take representative samples of the product under test in accordance with the provisions of GB/T 3186.

7 Test Methods

7.1 Determination of TiO₂ content — Aluminum reduction method

7.1.1 Principle

The dried test portion is dissolved in sulfuric acid containing ammonium sulfate. Under a carbon dioxide atmosphere, metallic aluminum is used to reduce tetravalent titanium to trivalent titanium. Ammonium thiocyanate is then used as the indicator, and the resulting solution is titrated with ferric ammonium sulfate standard volumetric solution.

7.1.2 Reagents

All reagents used shall be of analytical grade. Water shall conform to the requirements of grade 3 water specified in GB/T 6682.

WARNING: Use reagents in accordance with appropriate health and safety regulations.

7.1.2.1 Concentrated hydrochloric acid: mass fraction about 37 %, $\rho \approx 1.19$ g/mL.

7.1.2.2 Concentrated sulfuric acid: mass fraction about 96 %, $\rho \approx 1.84$ g/mL.

7.1.2.3 Ammonium sulfate.

7.1.2.4 Sodium bicarbonate: saturated solution. Add about 10 g sodium bicarbonate to 90 mL water.

7.1.2.5 Ammonium thiocyanate indicator. Dissolve 24.5 g ammonium thiocyanate in 80 mL hot water, filter, cool to room temperature, and dilute to 100 mL. Store in a sealed, dark-colored bottle.

7.1.2.6 Ferric ammonium sulfate standard volumetric solution: 1 mL equivalent to 0.004 8 g TiO₂.

7.1.2.6.1 Preparation

Weigh 30 g ferric ammonium sulfate [FeNH₄(SO₄)₂·12H₂O] into a 1 000 mL graduated volumetric flask, add 300 mL water containing 15 mL sulfuric acid (7.1.2.2) to dissolve it. Add potassium permanganate solution (7.1.2.7) dropwise until the solution turns pink. Dilute to the mark with water and mix well. If turbid, filter.

7.1.2.6.2 Standardization

Weigh 190 mg to 210 mg of a titanium dioxide standard reference material, dried to constant mass at (105 ± 2) °C, and standardize the above solution in accordance with the procedure described in 7.1.4.3. Calculate the titanium dioxide equivalent of the solution, T₁, expressed in grams of TiO₂ per millilitre, using Formula (1):

$$T_1 = (m_1 \times w(\text{TiO}_2)) / (V_1 \times 100)$$

..... (1)

m_1 — the mass of the titanium dioxide standard reference material used, in grams (g);

$w(\text{TiO}_2)$ — the titanium dioxide content of the standard reference material, expressed as a mass fraction.

If a spectroscopically pure titanium dioxide is used, $w(\text{TiO}_2)$ is taken as 100 %;

V_1 — the volume of ferric ammonium sulfate standard volumetric solution consumed in the titration, in millilitres (mL).

7.1.2.7 Potassium permanganate solution, $c(1/5 \text{ KMnO}_4) = 0.1 \text{ mol/L}$. Dissolve 3.16 g potassium permanganate in 500 mL water, place in a 1 000 mL graduated volumetric flask, dilute to the mark with water and mix well.

7.1.2.8 Metallic aluminum: electrolytic grade, in the form of aluminum foil, aluminum sheet or aluminum wire, with a content (mass fraction) of not less than 99.5 %.

7.1.3 Apparatus

Use ordinary laboratory apparatus and the following:

7.1.3.1 Glass liquid-sealing tube: see Figure 1; other suitable absorption vessels may also be used.

7.1.3.2 Weighing bottle: wide-mouthed, with a suitable cover, not larger than required for the test.

7.1.3.3 Oven: capable of maintaining a temperature of $(105 \pm 2) ^\circ\text{C}$.

7.1.3.4 Desiccator: containing a suitable desiccant, e.g. silica gel.

Figure 1 — Glass liquid-sealing tube (unit: mm)

*[Figure 1 — Glass liquid-sealing tube]
Total height $\approx 110 \text{ mm}$ | Bulb diameter $\approx 50 \text{ mm}$ |
Stem length $\approx 100 \text{ mm}$*

(Schematic only — see original figure for details.)

7.1.4 Procedure

7.1.4.1 General

Carry out the determination in duplicate in parallel.

7.1.4.2 Test portion

Take 10 g of the sample (see Clause 6) into a wide-mouthed weighing bottle (7.1.3.2) and dry to constant mass in the oven (7.1.3.3) at $(105 \pm 2) ^\circ\text{C}$. Cover, place in the desiccator (7.1.3.4) and cool to room temperature.

Weigh, to the nearest 0.1 mg, 190 mg to 210 mg of the above test portion (m_2).

7.1.4.3 Determination

Transfer the test portion to a dry 500 mL conical flask. Add 7 g to 8 g ammonium sulfate (7.1.2.3) and 20 mL sulfuric acid (7.1.2.2). Place on a hot plate, first heat gently with a small flame until strong white fumes are evolved, then continue to heat more strongly until dissolution is complete. Allow to cool, then add 120 mL water and 20 mL hydrochloric acid (7.1.2.1).

Add about 2.5 g metallic aluminum foil (7.1.2.8) to the conical flask. Fit the glass liquid-sealing tube, tighten the rubber stopper, and add sodium bicarbonate solution (7.1.2.4) to the liquid-sealing tube.

After the aluminum foil is completely dissolved, heat the solution to a gentle boil and keep it boiling for 3 min to 5 min. Cool to about 60 °C; it is best to immerse the lower part of the conical flask in cold water. During cooling, the sodium bicarbonate solution in the liquid-sealing tube will be drawn into the flask; above the reduced titanium solution, carbon dioxide gas will be evolved. During this process, sodium bicarbonate solution shall be replenished in time. Remove the stopper, pour the sodium bicarbonate solution in it into the conical flask, and rinse the stopper with a small amount of water. Collect the rinsings in the conical flask.

Add 2 mL ammonium thiocyanate indicator (7.1.2.5) and titrate immediately with ferric ammonium sulfate standard volumetric solution (7.1.2.6) to a light orange endpoint. It is recommended to add the major part of the ferric ammonium sulfate solution at the beginning, shake thoroughly, and then add the remainder drop by drop to complete the titration. Record the volume of ferric ammonium sulfate solution consumed (V_2).

7.1.5 Expression of results

7.1.5.1 Calculation

Calculate the titanium dioxide content, $w(\text{TiO}_2)$, expressed as mass fraction, using Formula (2):

$$w(\text{TiO}_2) = (V_2 \times T_1 \times 100) / m_2 \quad \dots\dots\dots (2)$$

m_2 — the mass of the test portion dried to constant mass, in grams (g);

V_2 — the volume of ferric ammonium sulfate standard volumetric solution consumed in the titration, in millilitres (mL);

T_1 — the titanium dioxide equivalent of each millilitre of ferric ammonium sulfate standard volumetric solution (see 7.1.2.6.2), in grams per millilitre (g/mL).

Take the arithmetic mean of two parallel determinations as the result, and report the result to 0.1 %.

NOTE: *The result of the calculation includes traces of substances that can be reduced by aluminum and then re-oxidized by ferric iron, such as tin and antimony. However, in titanium dioxide pigments commonly used for paints and related products, the amount of these interfering substances is very small.*

7.1.5.2 Permissible deviation

The absolute difference between the results of two parallel determinations shall not exceed 0.4 %.

7.2 Determination of matter volatile at 105 °C. Carry out in accordance with the provisions of GB/T 5211.3—1985.

7.3 Determination of matter soluble in water. Carry out in accordance with the provisions of GB/T 5211.2—2003, using a test portion of 10 g.

NOTE: *If the pigment is highly dispersed, the following procedure may be used: weigh 5 g of the test portion into a beaker, add 100 mL water, heat to boiling for 5 min, cool, then add 40 g/L sodium carbonate solution 25 mL under stirring, place on a water bath for 5 min, transfer the solution to a 250 mL volumetric flask, dilute to the mark with water and mix well. Filter, then carry out the remaining operations in accordance with GB/T 5211.2—2003, performing a blank test at the same time.*

The relative error of two parallel determinations shall not exceed 8 %.

7.4 Determination of residue on sieve. Carry out in accordance with the provisions of GB/T 5211.14—1988.

The test portion shall be 20 g, the dispersing agent shall be sodium hexametaphosphate with a mass concentration of 100 g/L, and the dosage shall be 3 mL to 5 mL.

7.5 Determination of colour

7.5.1 General

Two methods are provided: Method A — colorimetric method, and Method B — visual method. Method A is used as the routine method; Method B is used only as an agreed method. In case of dispute, Method A shall be used.

7.5.2 Method A — Colorimetric method

Carry out in accordance with the provisions of GB/T 5211.20—1999. The alkyd resin vehicle used shall be composed of linseed oil (mass fraction 63 %), phthalic anhydride (mass fraction 23 %) and trimethylolpropane (mass fraction 14 %).

7.5.3 Method B — Visual method

Carry out in accordance with the provisions of GB/T 1864—1989. The test portion shall be 2.00 g, and the dosage of refined linseed oil shall be 0.8 mL to 1.1 mL.

7.6 Determination of scattering power. Carry out in accordance with the provisions of GB/T 13451.2—1992.

The composition and properties of the alkyd resin vehicle used shall be the same as in 7.5.2.

7.7 Determination of matter volatile at 105 °C after 24 h preconditioning at (23 ± 2) °C and (50 ± 5) % RH. Place about 30 g of the test portion into a wide-mouthed container, e.g. a Φ 70 mm weighing bottle or evaporating dish (sample thickness not exceeding 2 cm). After standing for 24 h at (23 ± 2) °C and (50 ± 5) % RH, carry out the determination in accordance with 7.2.

7.8 Determination of pH of an aqueous suspension. Carry out in accordance with the provisions of GB/T 1717—1986.

7.9 Determination of oil absorption. Carry out in accordance with the provisions of GB/T 5211.15—1988. The agreed routine test portion shall be 5 g; in case of dispute, the test portion shall be 10 g.

7.10 Determination of resistivity of the aqueous extract. Carry out in accordance with the provisions of GB/T 5211.12.

8 Judgment of Test Results

The judgment shall be carried out by the rounded-value comparison method in accordance with GB/T 1250.

9 Marking, Packaging, Transport and Storage

9.1 Marking

Product packaging bags shall be printed with firm and clear markings, including the manufacturer's name and address, product name, registered trademark, standard code, type, production batch number, net content, date of manufacture and the prescribed “moisture-proof” mark.

9.2 Packaging

The product may be packed in plastic woven bags with plastic film liners, or in other suitable packaging materials.

9.3 Transport

During transport and loading/unloading, the packages shall be handled with care, lifted and lowered gently, to prevent contamination and damage. The product shall be protected from rain and exposure to direct sunlight during transport.

9.4 Storage

Products shall be sorted, batched and stored in a ventilated and dry place. They shall be kept strictly away from substances with which they may react, and shall be protected from moisture.

Annex A

(Informative)

Technical Differences between this Standard and ISO 591-1:2000 and the Reasons Therefor

Table A.1 gives a summary of the technical differences between this Standard and ISO 591-1:2000 and the reasons therefor.

Table A.1 — Technical differences between this Standard and ISO 591-1:2000 and the reasons therefor

Clause No. of this Standard	Technical difference	Reason
1	Added to the scope the wording “product classification, judgment of test results, and marking, packaging, transport and storage.” Added the wording “This Standard applies to titanium dioxide pigments produced by the sulfate process or the chloride process. These products are mainly used in paints, rubber, plastics, inks, papermaking and other industries.”	To make the scope of the Standard clearer and to suit national practice.
2	Chinese national standards are cited, instead of the international standard.	To suit national conditions.
Table 2	The three items “pH of aqueous suspension,” “oil absorption” and “aqueous extract resistivity” are specified as “agreed” instead of “similar to the agreed reference sample.”	User requirements; easier to implement.
Table 1 Table 2	Deleted the “Test method” column of Table 1 and Table 2 in ISO 591-1:2000.	Test methods are described in detail separately in Clause 7.
7.1	Deleted “7.1 General” of ISO 591-1:2000. Deleted “7.3 Method B — Chromium(II) reduction method” of ISO 591-1:2000.	Method B has been deleted, so this clause becomes redundant. This method is difficult to implement domestically.
7.1.2.7	Deleted the word “standard” of ISO 591-1:2000. “3.16 g” replaces “3.160 7 g”.	This solution is only used as a general solution; a general solution does not need to be accurate to 0.000 1 g.
7.1.2.8	Added the wording “with a content (mass fraction) of not less than 99.5 %”.	The provision is clearer and easier to implement.
7.1.3	Deleted the burette, pipette and volumetric flask.	These are ordinary laboratory apparatus, so they are not listed again.
7.1.4.3	The dosage of metallic aluminum foil is changed from “1 g” to “2.5 g”. Deleted the “burette” of ISO 591-1:2000; this Standard uses only the glass liquid-sealing tube (i.e. absorber). The wording of the procedure has been modified. Deleted the wording of ISO 591-1:2000 “or until obvious residues consisting of silicon dioxide (SiO ₂) or silicate	To ensure the reduction reaction is sufficient and complete. To suit national conditions; easier to implement. Clearer expression; easier to operate.

	substances are present”; the wording of the procedure has been modified.	
7.1.5.2	“Permissible deviation” replaces “precision.”	Easier to implement.
7.3 ~ 7.7, 7.9	Specific supplementary provisions have been added to the test conditions.	Easier to operate.
8 ~ 9	Added “8 Judgment of test results” and “9 Marking, packaging, transport and storage.” Deleted the “8 Test report” of ISO 591-1:2000.	To suit national conditions and user requirements.

Bibliography

- [1] GB 1706—1993, Titanium dioxide pigments (neq ISO 591:1977).
 - [2] GB/T 6682, Water for analytical laboratory use — Specification and test methods (GB/T 6682—1992, neq ISO 3696:1987).
 - [3] HG/T 2457, General rules for inspection, marking, packaging, transport and storage of pigment products (HG/T 2457—1993).
-

— End of Standard —

GB/T 1706—2006

All rights reserved. Reproduction is prohibited.

Book No.: 155066-1-28769 Price: 10.00 RMB



中华人民共和国国家标准

GB/T 1706—2006
代替 GB 1706—1993

二氧化钛颜料

Titanium dioxide pigments

(ISO 591-1:2000, Titanium dioxide pigments for paints—
Part 1: Specifications and methods of test, MOD)

2006-09-01 发布

2007-02-01 实施



中华人民共和国国家质量监督检验检疫总局
中国国家标准化管理委员会

发布

前 言

本标准修改采用国际标准 ISO 591-1:2000《色漆用二氧化钛颜料 第1部分:规格和试验方法》(英文版)。

本标准根据国际标准 ISO 591-1:2000《色漆用二氧化钛颜料 第1部分:规格和试验方法》重新起草。

本标准在采用国际标准时进行了修改,这些技术性差异用垂直单线标识在它们所涉及的条款的页边空白处。在附录 A 中给出了技术性差异及其原因的一览表以供参考。

本标准与国际标准 ISO 591-1:2000 相比,主要技术差异为:

- 本标准删除了“TiO₂ 含量测定中 B 法氯化铬(Ⅱ)还原法”;
- 本标准中所用试验方法均采用现行国家标准,其中大部分方法系等效采用相应国际标准;
- 本标准增加了“8 检验结果的判定”和“9 标志、包装、运输和贮存”;
- 本标准删除了国际标准的前言。

本标准代替 GB 1706—1993《二氧化钛颜料》,并作了技术上的修订和编辑性修改。

本标准与 GB 1706—1993 相比,主要技术差异为:

- 本标准中产品分 2 种类型 5 个品种(1993 版分 2 种类型 3 个品种,每个品种又分 3 个等级);
- 本标准采用商定的“参比样”(1993 版采用选定的“标准样”);
- 本标准中 A1 和 R1 2 个品种不控制“水萃取液电阻率”项目(1993 版 3 个品种均对此项目作了要求);
- 本标准中“颜色”、“散射力”、“水悬浮液 pH 值”、“吸油量”、“水萃取液电阻率”等项目指标均为商定(1993 版均规定了具体指标);
- 本标准删除了“二氧化钛含量测定中 B 法锌汞齐法”;
- 本标准增加了“颜色测定——色度法”;
- 本标准中用“散射力”代替了“消色力”项目。

本标准的附录 A 为资料性附录。

本标准由中国石油和化学工业协会提出。

本标准由全国涂料和颜料标准化技术委员会归口。

本标准主要起草单位:中国化工建设总公司常州涂料化工研究院、攀枝花钢铁有限责任公司钛业公司、四川龙蟒钛业有限责任公司、镇江钛白粉股份有限公司、常州市武进区申武钛白粉厂。

本标准参加起草单位:重庆渝港钛白粉股份有限公司、南京钛白化工有限责任公司、上海焦化有限公司钛白分公司、常州市长江钛白粉厂、淄博钛业股份有限公司、广州钛白粉厂、上海市涂料研究所。

本标准主要起草人:沈苏江、赵玲、黄国鑫、晏育刚、奉辉、郭建华。

本标准于 1979 年首次发布,1988 年第一次修订,1993 年第二次修订,本次为第三次修订。

二氧化钛颜料

1 范围

本标准规定了二氧化钛颜料的产品分类、要求、试验方法、检验结果的判定及标志、包装、运输和贮存。

本标准适用于硫酸法或氯化法生产的二氧化钛颜料。该产品主要用于涂料、橡胶、塑料、油墨及造纸等行业。

2 规范性引用文件

下列文件中的条款通过本标准的引用而成为本标准的条款。凡是注日期的引用文件,其随后所有的修改单(不包括勘误的内容)或修订版均不适用于本标准,然而,鼓励根据本标准达成协议的各方研究是否可使用这些文件的最新版本。凡是不注日期的引用文件,其最新版本适用于本标准。

GB/T 1250 极限数值的表示方法和判定方法

GB/T 1717—1986 颜料水悬浮液 pH 值的测定(eqv ISO 787-9:1981)

GB/T 1864—1989 颜料颜色的比较(eqv ISO 787-1:1982)

GB/T 3186 色漆、清漆和色漆与清漆用原材料 取样(GB/T 3186—2006,ISO 15528:2000,IDT)

GB/T 5211.2—2003 颜料水溶物测定 热萃取法(ISO 787-3:2000,General methods of test for pigments and extenders—Part 3:Determination of matter soluble in water-Hot extraction method,IDT)

GB/T 5211.3—1985 颜料在 105℃挥发物的测定(eqv ISO 787-2:1981)

GB/T 5211.12 颜料水萃取液电阻率的测定(GB/T 5211.12—1986,eqv ISO 787-14:1973)

GB/T 5211.14—1988 颜料筛余物的测定 机械冲洗法(eqv ISO 787-18:1983)

GB/T 5211.15—1988 颜料吸油量的测定(eqv ISO 787-5:1980)

GB/T 5211.20—1999 在本色体系中白色、黑色和着色颜料颜色的比较 色度法(eqv ISO 787-25:1993)

GB/T 13451.2—1992 着色颜料相对着色力和白色颜料相对散射力的测定 光度计法(eqv ISO 787-24:1985)

3 术语和定义

本标准采用下列术语和定义:

3.1

二氧化钛颜料 titanium dioxide pigments

由 X-射线法测定的晶体结构主要为锐钛型或金红石型的二氧化钛(TiO_2)组成的颜料。

4 产品分类

4.1 型号

本标准包括以下两种类型的二氧化钛颜料:

A 型:锐钛型;

R 型:金红石型。

4.2 品种

颜料可进一步分为下列品种:

A 型:A1、A2。
R 型:R1、R2、R3。

5 特性和容许范围

5.1 外观

应为软质干燥粉末或在不加研磨下用调刀易于碾碎。

5.2 其他特性

5.2.1 符合本标准的二氧化钛颜料,其基本要求规定于表 1,条件要求规定于表 2。条件要求将由有关双方规定。

表 2 中预处理后 105℃挥发物为商定项目,只有当有关方面明确规定或有合同约定时才进行。

5.2.2 表 2 中所指商定的参比样应符合表 1 中要求。

表 1 基 本 要 求

特 性		要 求				
		A 型		R 型		
		A1	A2	R1	R2	R3
TiO ₂ 的质量分数/%	≥	98	92	97	90	80
105℃挥发物的质量分数/%	≤	0.5	0.8	0.5	商定	
水溶物的质量分数/%	≤	0.6	0.5	0.6	0.5	0.7
筛余物(45 μm)的质量分数/%	≤	0.1	0.1	0.1	0.1	0.1

表 2 条 件 要 求

特 性	要 求				
	A 型		R 型		
	A1	A2	R1	R2	R3
颜色 ^a	与商定的参比样相近(见 5.2.2)				
散射力 ^a	商定				
在(23±2)℃和相对湿度(50±5)%下预处理 24 h 后 105℃挥发物的质量分数/% ^b ≤	0.5	0.8	0.5	1.5	2.5
水悬浮液 pH 值	商 定				
吸油量					
水萃取液电阻率		商定	—	商定	
^a 测定时所用的参比样为有关双方商定的样品。					
^b 见 5.2.1。					

6 取样

按 GB/T 3186 的规定取受试产品的代表性样品。

7 试验方法

7.1 二氧化钛含量的测定——铝还原法

7.1.1 原理

经干燥的试样溶解在含有硫酸铵的硫酸中。在二氧化碳气氛下用金属铝将四价钛还原成三价钛。然后以硫氰酸铵作指示剂,用硫酸铁铵标准滴定溶液滴定上述溶液。

7.1.2 试剂

所用试剂均应采用分析纯试剂,并使用符合 GB/T 6682 规定的纯度至少为 3 级的水。

警告:应按照适当的健康和安全规则使用试剂。

7.1.2.1 浓盐酸:质量分数约为 37%, $\rho \approx 1.19$ g/mL。

7.1.2.2 浓硫酸:质量分数约为 96%, $\rho \approx 1.84$ g/mL。

7.1.2.3 硫酸铵。

7.1.2.4 碳酸氢钠:饱和溶液

将约 10 g 碳酸氢钠加入到 90 mL 水中。

7.1.2.5 硫氰酸铵指示剂

将 24.5 g 硫氰酸铵溶于 80 mL 热水中,过滤,冷却至室温并稀释至 100 mL,贮存于密闭深色瓶中。

7.1.2.6 硫酸铁铵标准滴定溶液:1 mL 相当于 0.004 8 g TiO_2 。

7.1.2.6.1 制备

称取 30 g 硫酸铁铵 $[\text{FeNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}]$ 置于 1 000 mL 单刻度容量瓶中,加入 300 mL 含 15 mL 硫酸(7.1.2.2)的水溶解。滴加高锰酸钾溶液(7.1.2.7)直至溶液呈粉红色。用水稀释至刻度并摇匀。如溶液浑浊则过滤。

7.1.2.6.2 标定

称取经 $(105 \pm 2)^\circ\text{C}$ 下干燥至恒重的二氧化钛标准参比物质 190 mg~210 mg,按 7.1.4.3 中所述步骤标定上述溶液。

用式(1)计算溶液的二氧化钛相当量 T_1 ,以每毫升相当 TiO_2 克数表示:

$$T_1 = \frac{m_1 \times w(\text{TiO}_2)}{V_1 \times 100} \dots\dots\dots (1)$$

式中:

m_1 ——所用二氧化钛标准参比物质的质量,单位为克(g);

$w(\text{TiO}_2)$ ——标准参比物质的二氧化钛含量,以质量分数表示(如用光谱纯二氧化钛,则 $w(\text{TiO}_2)$ 以 100%计);

V_1 ——滴定消耗硫酸铁铵标准溶液的体积,单位为毫升(mL)。

7.1.2.7 高锰酸钾溶液 $c\left(\frac{1}{5}\text{KMnO}_4\right) = 0.1$ mol/L

将 3.16 g 高锰酸钾溶于 500 mL 水中,盛于 1 000 mL 单刻度容量瓶,加水稀至刻度,混匀。

7.1.2.8 金属铝:电解级,以铝箔、铝片和铝线形式存在,含量(质量分数)不低于 99.5%。

7.1.3 仪器

使用普通实验室仪器,以及下列仪器:

7.1.3.1 玻璃液封管:见图 1,其他合适的吸收器也可使用。

7.1.3.2 称量瓶:广口,带合适盖子,尺寸不大于试验所需。

7.1.3.3 烘箱:能维持 $(105 \pm 2)^\circ\text{C}$ 。

7.1.3.4 干燥器:内盛合适干燥剂,如硅胶。

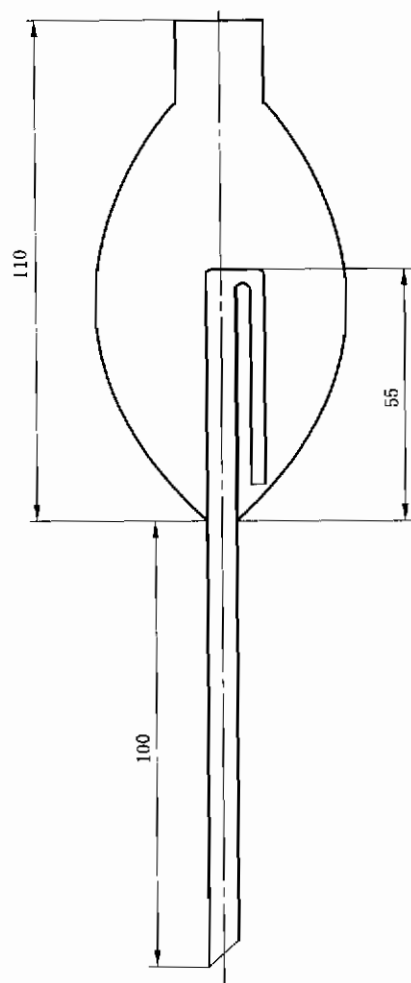


图 1 玻璃液封管

7.1.4 步骤

7.1.4.1 总则

平行测定两次。

7.1.4.2 试样

取 10 g 试样(见第 6)于敞口称量瓶(7.1.3.2)中在 $(105 \pm 2)^\circ\text{C}$ 下干燥至恒重,加盖并置于干燥器(7.1.3.4)中冷却至室温。

称取 190 mg~210 mg 上述试样(m_2),精确至 0.1 mg。

7.1.4.3 测定

将试样转移至干燥的 500 mL 锥形瓶中。加入 7 g~8 g 硫酸铵(7.1.2.3)和 20 mL 硫酸(7.1.2.2)。摇匀置于电热板上先小火缓慢加热,直至产生强烈白烟,再继续加强热直至溶解完全。小心冷却,加入 120 mL 水和 20 mL 盐酸(7.1.2.1)。

加入约 2.5 g 金属铝片(7.1.2.8)于锥形瓶中,装上液封管,塞紧胶塞,并在液封管中加入碳酸氢钠溶液(7.1.2.4)。

待铝片溶解完全,加热溶液至微沸并保持 3 min~5 min。冷却至约 60°C ,最好将锥形瓶部分浸入盛水容器中,冷却过程中碳酸氢钠溶液会吸入瓶中在还原后的钛溶液上方产生二氧化碳气体,在这个过程中应随时补加碳酸氢钠溶液。取下塞子,将其中的碳酸氢钠溶液倒入锥形瓶中并用少量水淋洗塞子,将淋洗液收集于锥形瓶中。

加入 2 mL 硫氰酸铵指示剂(7.1.2.5),立即用硫酸铁铵标准溶液(7.1.2.6)滴定至终点淡橙色。最好开始时加入大部分硫酸铁铵溶液,充分摇动,再逐滴加入完成滴定过程。记录所耗硫酸铁铵溶液的体积(V_2)。

7.1.5 结果的表示

7.1.5.1 计算

用式(2)计算二氧化钛含量 ω ,以质量分数表示:

$$\omega(\text{TiO}_2) = \frac{V_2 \times T_1 \times 100}{m_2} \dots\dots\dots(2)$$

式中:

m_2 ——经干燥至恒重的试样的质量,单位为克(g);

V_2 ——滴定消耗硫酸铁铵标准溶液的体积,单位为毫升(mL);

T_1 ——与每毫升硫酸铁铵标准溶液相当的二氧化钛克数(见 7.1.2.6.2),单位为克每毫升(g/mL)。

计算两次平行测定值之平均值,报告结果至 0.1%。

注:计算结果包括铬、砷和其他一些能被铝还原后又被三价铁氧化的物质。然而,通常用于色漆和相关产品的二氧化钛颜料中这些干扰物质的量似乎极少。

7.1.5.2 允许差

两次平行测定值之绝对差不得大于 0.4%。

7.2 105℃挥发物的测定

按 GB/T 5211.3—1985 中的规定进行。

7.3 水溶物的测定

按 GB/T 5211.2—2003 中的规定进行,试样量为 10 g。

注:如颜料高度分散,可按下法进行测定:称取试样 5 g,置于一烧杯中,加入 100 mL 水,加热煮沸 5 min,冷却,然后在搅拌下加入 40 g/L 碳酸铵溶液 25 mL,放置片刻,将该溶液转移至 250 mL 容量瓶中,用水稀释至刻度,摇匀。过滤,其余操作仍按 GB/T 5211.2—2003 进行,同时进行空白试验。

两次平行测定的相对误差不得大于 8%。

7.4 筛余物的测定

按 GB/T 5211.14—1988 中的规定进行。试样量为 20 g,分散剂为六偏磷酸钠,质量浓度为 100 g/L,用量 3 mL~5 mL。

7.5 颜色的测定

7.5.1 总则

提供了两种方法:A 法——色度法和 B 法——目视法。A 法用作常规方法,B 法仅作商定方法,仲裁时选用 A 法。

7.5.2 A 法——色度法

按 GB/T 5211.20—1999 中的规定进行。所用醇酸树脂组成的质量分数为 63%亚麻仁油、23%邻苯二甲酸酐和 14%三羟甲基丙烷。

7.5.3 B 法——目视法

按 GB/T 1864—1989 中的规定进行。试样量为 2.00 g,精制亚麻仁油加量为 0.8 mL~1.1 mL。

7.6 散射力的测定

按 GB/T 13451.2—1992 的规定进行。所用醇酸树脂其组成及性能与 7.5.2 中一致。

7.7 在(23±2)℃和相对湿度(50±5)%下预处理 24 h 后 105℃挥发物的测定

将试样约 30 g 放入敞口容器中,如 $\phi 70$ mm 称量瓶或蒸发皿(样品厚度不超过 2 cm),在(23±2)℃和相对湿度(50±5)%下放置 24 h 后按 7.2 规定进行。

7.8 水悬浮液 pH 值的测定

按 GB/T 1717—1986 中的规定进行。

7.9 吸油量的测定

按 GB/T 5211.15—1988 中的规定进行。规定日常检测称量为 5 g,仲裁时称量为 10 g。

7.10 水萃取液电阻率的测定

按 GB/T 5211.12 中的规定进行。

8 检验结果的判定

按 GB/T 1250 中修约值比较法进行。

9 标志、包装、运输和贮存

9.1 标志

产品包装袋上应印有牢固、清晰的标志,包括生产厂名称和地址、产品名称、注册商标、标准代号、型号、生产批号、净含量、生产日期及规定的“防潮”标志。

9.2 包装

产品可用塑料编织袋内衬塑料薄膜袋包装,也可用其他适宜的包装材料包装。

9.3 运输

运输、装卸时要轻装、轻卸,防止包装污染和破损。产品在运输中应防止雨淋和日光曝晒。

9.4 贮存

产品应按分类、分批存放在通风干燥处,严禁与产品可发生反应的物品接触,并注意防潮。

附 录 A
(资料性附录)

本标准与 ISO 591-1:2000 技术性差异及其原因

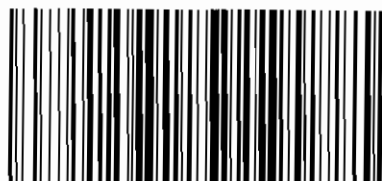
表 A.1 给出了本标准与 ISO 591-1:2000 的技术性差异及其原因的一览表。

表 A.1 本标准与 ISO 591-1:2000 技术性差异及其原因

本标准的 章条编号	技 术 性 差 异	原 因
1	在范围中增加了“产品分类、检验结果的判定及标志、包装、运输和贮存。” 增加了“本标准适用于硫酸法或氯化法生产的二氧化钛颜料。该产品主要用于涂料、橡胶、塑料、油墨及造纸等行业”	使标准适用范围更明确,符合我国习惯
2	引用了采用国际标准的我国标准,而非国际标准	适合我国国情
表 2	“水悬浮液 pH 值”、“吸油量”、“水萃取液电阻率”三项中要求以“商定”代替“与商定参照颜料相近”	用户需求,实施更方便
表 1 表 2	删除 ISO 591-1:2000 表 1、表 2 中“试验方法”一栏	已在第 7 中分别详细描述
7.1	删除 ISO 591-1:2000 中“7.1 通则”。 删除 ISO 591-1:2000 中“7.3 B 法氯化铬(Ⅱ)还原法”	已删除 B 法,此条解释多余。 此法在国内实施有困难
7.1.2.7	删除 ISO 591-1:2000 中“标准”二字。 以“3.16 g”代替“3.160 7 g”	此溶液用途只相当于一般溶液。 一般溶液无需精确至 0.000 1 g
7.1.2.8	增加“含量(质量分数)不低于 99.5 %”	规定更明确,便于标准的实施
7.1.3	删除滴定管、移液管和容量瓶	均属普通实验室仪器,故不再重复
7.1.4.3	金属铝片用量以“2.5 g”代替“1 g”。 删除 ISO 591-1:2000 中“导管”,本标准中仅选用液封管(即吸收器),操作步骤稍有变动。 删除 ISO 591-1:2000 中“或直至显然残余物由二氧化硅(SiO ₂)或含硅物质组成”,文字表述稍作变动	保证还原反应充分、完全。 适合我国国情,实施方便。 表述更清晰,便于操作
7.1.5.2	以“允许差”代替“精密度”	便于标准的实施
7.3~7.7、7.9	对试验条件作了具体补充规定	便于操作
8~9	增加“8 检验结果的判定”和“9 标志、包装、运输和贮存”。 删除 ISO 591-1:2000 中“8 试验报告”	适合我国国情,用户需求

参 考 文 献

- [1] GB 1706—1993 二氧化钛颜料(neq ISO 591:1977)
 - [2] GB/T 6682 分析实验室用水规格和试验方法(GB/T 6682—1992, neq ISO 3696:1987)
 - [3] HG/T 2457 颜料产品检验、标志、包装、运输和贮存通则(HG/T 2457—1993)
-



GB/T 1706-2006

版权专有 侵权必究

*

书号:155066·1-28769

定价: 10.00 元