ICS 87.060.10
G 54

National Standards of the People's Republic of China

GB/T 1706–2006
replace GB 1706—1993

Titanium dioxide pigment

Titanium dioxide pigments

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

2006-09-01 release **2007-02-01 Implementation**

Digital anti-counterfeiting

General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China
China National Standardization Administration

Publish

Preface

This standard is modified and adopted from the international standard ISO 591-1:2000 "Titanium dioxide pigments for paints – Part 1: Specifications and test methods" (English). (Chinese version).

This standard is revised based on the international standard ISO 591-1:2000 "Titanium dioxide pigments for paints – Part 1: Specifications and test methods". Drafting.

This standard has been modified in accordance with international standards. These technical differences are indicated by a vertical single line on the page of the clauses to which they pertain. Blank space. (See Appendix) A The table provides a summary of the technical differences and their reasons for reference.

This standard and international standards ISO 591-1:2000 The main technical differences are as follows:

—This standard removes the " Method B : Chromium Chloride (II) Reduction Method in the Determination of TiO2 Content";

—The test methods used in this standard are all based on current national standards, and most of the methods are equivalent to the corresponding international standards;

—This standard adds "8. Judgment of Inspection Results" and "9. Marking, Packaging, Transportation and Storage";

This standard removes the foreword of the international standard.

This standard replaces GB1706—1993 "Titanium Dioxide Pigment" and has been revised technically and edited. The main technical differences between this standard and GB1706-1993 are as follows:

—This standard classifies products into 2 types and 5 varieties (the 1993 version classified them into 2 types and 3 varieties, with each variety further divided into 3 grades);

- One standard uses an agreed-upon "reference sample" (the 1993 edition uses a selected "standard sample");

A1 in a standard and R1 Two varieties do not control the "resistivity of water extract" item (all three varieties in the 1993 version required this item);

—In this standard, "color", "scattering power", and "aqueous suspension" are used. pH The indicators such as "value" , "oil absorption", and "resistivity of water extract" are all... Agreed upon (specific indicators were stipulated in the 1993 version);

1. This standard has deleted "B in the determination of titanium dioxide content" Zinc amalgamation method;

—This standard adds "color measurement" - Colorimetric method;

—In this standard, the term “scattering power” has been replaced with “achromatic power”.

A of this standard This is an informational appendix.

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

This standard is under the jurisdiction of the National Technical Committee on Standardization of Coatings and Pigments.

The main drafting units of this standard are: Changzhou Coatings and Chemical Research Institute of China National Chemical Engineering Corporation, and Panzhihua Iron and Steel Co., Ltd. Titanium Industry Co., Ltd. The companies are Sichuan Longmang Titanium Industry Co., Ltd., Zhenjiang Titanium Dioxide Co., Ltd., and Changzhou Wujin District Shenwu Titanium Dioxide Factory.

The drafting units participating in this standard are: Chongqing Yugang Titanium Dioxide Co., Ltd., Nanjing Titanium Dioxide Chemical Co., Ltd., and Shanghai Coking Co., Ltd. The company's titanium dioxide division, Changzhou Changjiang Titanium Dioxide Plant, Zibo Cobalt Industry Co., Ltd. , Guangzhou Titanium Dioxide Plant, and Shanghai Coatings Research Institute.

The main drafters of this standard are: Shen Sujiang, Zhao Ling, Huang Guoxin, Yan Yugang, Feng Hui, and Guo Jianhua.

This standard was first published in 1979, revised for the first time in 1988, revised for the second time in 1993, and this is the third revision.

Titanium dioxide pigment

1 Scope

This standard specifies the product classification, requirements, test methods, judgment of test results, marking, packaging, and transportation of titanium dioxide pigments. Storage.

This standard applies to titanium dioxide pigments produced by the sulfuric acid process or the chloride process. This product is mainly used in the coatings, rubber , plastics, inks, and paper industries.

2 Normative references

The clauses in the following documents, through reference in this standard, become clauses of this standard. For dated references, subsequent references shall prevail. Amendments (excluding errata) or revisions are not applicable to this standard; however, parties agreeing to this standard are encouraged to study them. Can the latest versions of these documents be used? For undated references, the latest version applies to this standard.

GB /T 1250 Methods for representing and determining limit values

GB /T 1717-1986 pH of pigment aqueous suspension Determination of value (eqv) ISO 787-9:1981)

GB/T 1864–1989 Comparison of pigment colors (eqv IS O 787-1:1982)

GB /T 3186 Sampling of raw materials for paints, varnishes and paints and varnishes (GB /T3186—2006, ISO 15528:2000, IDT)

GB/T 5211.2—2003 Determination of water-soluble matter in pigments by thermal extraction (ISO 787-3:2000 , General methods of test for pig- ments and extensions —Part 3: Determination of matter soluble in water ~ Hot extraction method, I DT)

GB /T 5211.3—1985 Determination of volatiles in pigments at 105℃ (eqv) ISO 787-2: 1981)

GB /T 5211.12 Determination of resistivity of water extract of pigments (GB /T 5211.12—1986, eqv) ISO 787-14:1973)

GB /T 5211.14—1988 Determination of pigment sieve residue by mechanical rinsing method (eqv ISO 787-18:1983)

GB /T 5211.15—1988 Determination of pigment oil absorption (eqv) IS O 787-5:1980)

GB /T 5211.20—1999 Comparative colorimetric method for white, black, and coloring pigments in the natural color system (eqv) IS O 787-25: 1993)

GB /T 13451.2—1992 Determination of the relative tinting strength of colored pigments and the relative scattering strength of white pigments by photometric method (e qv) ISO 787-24:1985)

3 Terms and Definitions

The following terms and definitions are used in this standard:

3.1

Titanium dioxide pigment dioxide pigments

Depend on The crystal structure determined by X- ray diffraction is mainly anatase or rutile titanium dioxide (TiO_2). Pigments that make up the composition.

4 Product Categories

4.1 model

This standard includes the following two types of titanium dioxide pigments:

A : Anatase;

R type: Rutile type.

4.2 variety

Pigments can be further classified into the following categories:

A : A1 , A2 .
R type: R1 , R2 , R3 .

5 Characteristic requirements and allowable range

5.1 Appearance

It should be a soft, dry powder or easily crushed with a spatula without grinding.

5.2 Other features

5.2.1 The basic requirements for titanium dioxide pigments conforming to this standard are specified in Table 1, and the conditional requirements are specified in Table 2. The conditional requirements will be determined by the relevant parties.

The volatile matter at 105℃ after pretreatment in Table 2 is an agreed-upon item and will only be carried out when explicitly stipulated by the relevant parties or agreed upon in a contract .

5.2.2 The agreed reference samples referred to in Table 2 should meet the requirements in Table 1.

surface 1 base Book want beg

special sex	want beg				
	A type		R type		
	A1	A2	R1	R2	R3
TiO_2 Mass fraction/% ≥	98	92	97	90	80
Mass fraction of volatiles at 105℃ / %	0.5	0.8	0.5	Agreed	
Mass fraction of water-soluble matter / % ≤	0.6	0.5	0.6	0.5	0.7
Residue on sieve (45) mass fraction (μm) / % ≤	0.1	0.1	0.1	0.1	0.1

surface 2 strip Item want beg

special sex	want beg				
	A type		R type		
	Al	A2	R1	R2	R3
color [a]	Similar to the agreed reference sample (see 5.2.2)				
Scattering force [a]	Agreed				
Pretreatment at (23±2)℃ and (50=5)% relative humidity Mass fraction of volatiles at 105℃ after 24 hours / % [b] ≤	0.5	0.8	0.5	1.5	2.5
pH value of aqueous suspension	business Certainly				
Oil absorption					
Water extract resistivity		Agreed	—	Agreed	

a. The reference sample used in the determination is a sample agreed upon by both parties.

b See 5.2.1:

6 sampling

according to GB/T 3186 The regulations stipulate that representative samples of the tested products should be taken .

7 Test methods

7.1 Determination of titanium dioxide content—Aluminum reduction method

7.1.1 principle

The dried sample was dissolved in sulfuric acid containing ammonium sulfate. Tetravalent titanium was reduced to trivalent titanium with metallic aluminum under a carbon dioxide atmosphere. Then, using ammonium thiocyanate as an indicator, the above solution was titrated with a standard ammonium ferric sulfate titrant .

7.1.2 Reagents

All reagents used should be analytical grade and conform to GB /T standards. 6682 The water must have a purity level of at least 3.

Warning: The reagent should be used in accordance with appropriate health and safety rules.

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

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

7.1.2.3 Ammonium sulfate.

7.1.2.4 Sodium bicarbonate: saturated solution

Approximately 10 g Add sodium bicarbonate to 90 ml . In the water.

7.1.2.5 Ammonium thiocyanate indicator

24.5 g ammonium thiocyanate dissolved in 80 mL In 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 titration solution: 1 mL Equivalent to 0 .0048 gTiO_2 .

7.1.2.6.1 preparation

Weigh 30 g of ferric ammonium sulfate [$FeNH_4(SO_4)_2 \cdot I_2H_2O$] and place it in a 1000 mL container . ml . Add 300 mL to a single-mark volumetric flask Includes

15 mL Dissolve sulfuric acid (7.1.2.2) in water. Add potassium permanganate solution (7.1.2.7) dropwise until the solution turns pink. Dilute with water to the mark. Shake well. If the solution is cloudy, filter.

7.1.2.6.2 Calibration

mg to 210 mg of titanium dioxide standard reference material that has been dried to constant weight at (105±2)°C , and proceed according to 7.1.4.3. The steps described in

The above solution was then standardized.

The equivalent amount of titanium dioxide T1 in the solution is calculated using equation (1) . Expressed as grams of TiO_2 per milliliter :

$$T_1 = \frac{m_1 \times w(TiO_2)}{V_1 \times 100} \qquad \cdots\cdots (1)$$

…

In the formula:

m_1 —— The mass of the titanium dioxide standard reference material used is expressed in grams (g).

$w(TiO_2)$ —— The titanium dioxide content of the standard reference material is expressed as a mass fraction (e.g., if spectroscopically pure titanium dioxide is used, then...). $w(TiO_2)$

(100% calculation)

V_1 —— The volume of ferric ammonium sulfate standard solution consumed in the titration is expressed in milliliters (mL) .

7.1.2.7 potassium permanganate solution $c\left(\frac{1}{5}KMnO_4\right) = 0.1$ mol/L

3 . 16 g Potassium permanganate dissolved in 500 mL In water, containing 1000 mL For a single-mark volumetric flask, dilute with water to the mark and mix well. 7.1.2.8 Metallic aluminum: Electrolytic grade, existing in the form of aluminum foil, aluminum sheet and aluminum wire, with a content (mass fraction) of not less than 99.5%.

7.1.3 instrument

Use standard laboratory equipment, as well as the following instruments:

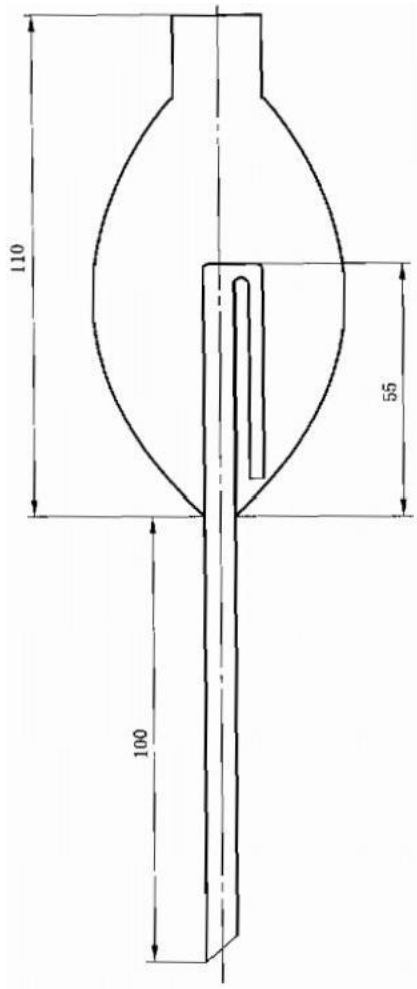
7.1.3.1 Glass liquid seal tube: See Figure 1. Other suitable absorbers can also be used.

7.1.3.2 Weighing bottle: wide-mouthed, with a suitable cap, and no larger than required for the test.

7.1.3.3 Oven: Can maintain (105±2)℃.

7.1.3.4 Dryer: Contains a suitable desiccant, such as silica gel.

The unit is millimeters.



picture 1 Glass liquid seal tube

7.1.4 step

7.1.4.1 General Principles

The measurements were performed twice in parallel.

7.1.4.2 Sample

Take 10 g The sample (see section 6) was dried to constant weight at (105±2)℃ in an open weighing bottle (7.1.3.2), then capped and placed in a desiccator. Cool to room temperature in (7.1.3.4).

Weigh 190 mg ~ 210 mg The above sample (m_2), Accurate to 0.1 mg .

7.1.4.3 Measurement

Transfer the sample to a dry 500°C environment. ml , cone shape bottle middle . add enter 7 g ~ 8g ammonium sulfate (7 . 1 .2 . 3) and 20 mL sulfuric acid (7.1.2.2). Shake well and place on a hot plate. Heat slowly over low heat until strong white smoke is produced, then increase the heat until completely dissolved. Carefully. Cool, then add 120 mL Water and 20 ml , hydrochloric acid (7.1.2.1).

Add approximately 2.5 g Aluminum sheet (7.1.2.8) is placed in an Erlenmeyer flask, fitted with a liquid seal tube, and the rubber stopper is tightened. Sodium bicarbonate is then added to the liquid seal tube. Solution (7.1.2.4).

Once the aluminum sheet has completely dissolved, heat the solution to a gentle boil and maintain this temperature for 3 to 5 minutes . Cool to approximately 60°C, ideally immersing part of the conical flask in the solution. In the water-filled container, during the cooling process, the sodium bicarbonate solution is drawn into the bottle , generating carbon dioxide gas above the reduced titanium solution. Sodium bicarbonate solution should be

added as needed during the process. Remove the stopper, pour the sodium bicarbonate solution into the conical flask, and rinse the stopper with a small amount of water. Collect the eluent in an Erlenmeyer flask.

Add 2 ml , Ammonium thiocyanate indicator (7.1.2.5) is immediately titrated with ferric ammonium sulfate standard solution (7.1.2.6) until the endpoint is a pale orange. It is best to add most of the ferric ammonium sulfate solution at the beginning, shake thoroughly, and then add it dropwise to complete the titration. Record the amount of ferric ammonium sulfate solution consumed.
Volume (V_2) .

7.1.5 Representation of results

7.1.5.1 calculate

The titanium dioxide content w is calculated using equation (2).
Expressed as a mass fraction:

$$\omega(TiO_2) = \frac{V_2 \times T_1 \times 100}{m_2} \quad \text{……(2)}$$

In the formula:

m_2 :—… the mass of the sample dried to constant weight, in grams (g);

V_2 — The volume of ferric ammonium sulfate standard solution consumed in the titration, in milliliters (mL):

T_1 --- The number of grams of titanium dioxide equivalent to one milliliter of ferric ammonium sulfate standard solution (see 7.1.2.6.2). The unit is grams per milliliter. (g/mL)

Calculate the average of the two parallel measurements and report the result to 0.1%.

Note: The calculation results include chromium, arsenic, and other substances that can be reduced by aluminum and then oxidized by ferric iron. However, the ferric iron commonly used in paints and related products... The amount of these interfering substances in titanium dioxide pigments appears to be extremely small.

7:1.5.2 Allowable difference

The absolute difference between two parallel measurements must not exceed 0.4%.

7.2 Determination of volatiles at 105℃

according to the provisions of GB /T 5211.3—1985 .

7.3 Determination of water-soluble substances

in accordance with the provisions of GB /T 5211.2—2003 , with a sample size of 10 g .

Note: If the pigment is highly dispersed, the determination can be performed as follows: Weigh 5 g of the sample, Place in a beaker and add 100 Add ml of water and heat to a boil for 5 minutes. Cool, then Add 40 g/L while stirring. 25 mL of ammonium carbonate solution After standing for a moment, transfer the solution to a 250 mL volumetric flask, dilute to the mark with water, and shake.

Mix well. Filter, and continue with the remaining operations as before. The test shall be conducted in accordance with GB/T 5211.2-2003 , and a blank test shall be performed simultaneously.

The relative error between two parallel measurements must not exceed 8%.

7.4 Determination of sieve residue

according to GB /T 5211.14-1988 The procedure was carried out according to the regulations. The sample size was 20 g, and the dispersant was sodium hexametaphosphate with a mass concentration of [missing information]. 100 g /L, dosage 3 mL ~ 5 mL .

7.5 Color measurement

7.5.1 General Principles

Two methods are provided: Method A – Colorimetric method and Method B Law — Japanese Visual Law. A The method is used as a conventional method, B The law is only an agreed method ; A shall be selected in arbitration. Law.

7.5.2 Method A – Colorimetric Method

According to GB /T 5211.20-1999 The process is carried out in accordance with the regulations . The alkyd resin used has a mass fraction of 63% linseed oil and 23% o-linseed oil. Phthalic anhydride and 14% trimethylolpropane.

7.5.3 Method B – Visual Inspection

according to GB /T The procedure shall be carried out in accordance with the provisions of 1864–1989 . The sample size is 2.00 g. The amount of refined flaxseed oil added is 0.8 ml to 1.1 ml .

7.6 Measurement of scattering force

according to GB /T The requirements of 13451.2—1992 shall apply. The composition and properties of the alkyd resin used shall conform to 7.5.2. Consistent with 7.7 Determination of volatiles at 105℃ after pretreatment at (23±2)℃ and (50±5)% relative humidity for 24 h.

Place approximately 30 g of the sample into an open container, such as a Φ 70 mm container . Weighing bottles or evaporating dishes (sample thickness not exceeding 2 cm) at (23 ± 2)℃ Placed at a relative humidity of (50±5)% for 24 days h Then proceed as specified in 7.2.

7.8 Aqueous suspension pH value measurement

according to GB/T The provisions of 1717–1986 shall apply.

7.9 Determination of oil absorption

according to GB /T The procedures outlined in 5211.15—1988 are to be followed. The daily weighing limit is specified as 5. g, The weight was 10 during arbitration. g . 7.10 **Determination of resistivity of water extract**

according to GB/T 5211.12 It shall be carried out in accordance with the provisions of the law.

8 Judgment of test results

according to The rounding value comparison method in GB /T1250 shall be used.

9 Marking, packaging, transportation and storage

9.1 logo

The product packaging should bear a strong and clear marking, including the manufacturer's name and address, product name, registered trademark, standard code, and model number. The product number, production batch number, net content, production date, and the prescribed "moisture-proof " label are required.

9.2 Package

The product can be packaged in a plastic woven bag lined with a plastic film bag, or in other suitable packaging materials.

9.3 transportation

Handle with care during transportation and loading/unloading to prevent packaging contamination and damage. Protect products from rain and direct sunlight during transportation.

9.4 Storage

The products should be stored in a well-ventilated and dry place according to their categories and batches. They must not come into contact with substances that may react with the products, and should be protected from moisture.

Appendix record A

(Informative Appendix)

This standard and ISO 591-1:2000 Technical differences and their causes

surface A.1 provides a comparison between this standard and ISO 591-1:2000. A list of technical differences and their causes.

surface A.1 This standard is consistent with ISO 591-1:2000 Technical differences and their causes

This standard Chapter Number	technology technique sex Difference different	Original because
l	The scope has been expanded to include "product classification, determination of inspection results and marking, packaging , transportation and storage". The standard now applies to titanium dioxide produced by the sulfuric acid process or the chlorination process. Pigments. This product is mainly used in coatings, rubber, plastics, inks, and papermaking. industry "	This makes the scope of application of the standard clearer and conforms to Chinese customs.
2	It cites Chinese standards that adopt international standards, rather than international standards.	Suitable for my country's national conditions
Table 2	Among the three items: "pH value of aqueous suspension", "oil absorption", and "resistivity of aqueous extract", The requirement is to replace "similar to the agreed reference pigment" with "agreed upon".	User needs, easier implementation
Table 1 Table 2	Test Methods" column from Tables 1 and 2 of ISO 591-1:2000.	The details have been described in section 7.
7.1	Remove "7.1 General Rules" from ISO 591-1:2000. Remove "7.3 B Method: Chromium Chloride (II) Reduction" from ISO 591-1:2000.	Method B has been deleted; this explanation is redundant. This law faces difficulties in implementation domestically.
7.1.2.7	Remove the word "standard" from ISO 591-1:2000. Replace "3.16 g" with "3.1607 g".	This solution is used only as a general solution. For general solutions, accuracy to 0 is not required. . 0001g
7.1.2.8	Add "content (mass fraction) not less than 99.5%"	The regulations are more specific, making it easier to implement the standards.
7.1.3	Remove burettes, pipettes, and volumetric flasks.	These are all common laboratory instruments, so they will not be repeated.
7.1.4.3	The amount of aluminum sheet used is expressed as "2.5g" instead of "1g". The term "conduit" has been removed from ISO 591-1:2000. This standard uses only liquid seal tubes (i.e., absorbers), and the operating procedures have been slightly modified. Remove "or until it is obvious that the residue is composed of silica" from ISO 591-1:2000. The wording has been slightly modified to state that the substance is composed of (SiO_2) or silicon- containing materials .	Ensure the reduction reaction is complete and thorough. It is suitable for my country's national conditions and is easy to implement. The description is clearer, making operation easier.
7.1.5.2	Replace "precision" with "allowable difference".	Facilitate the implementation of standards
7.3–7.7, 7.9	Specific supplementary regulations were made regarding the test conditions.	Easy to operate
8-9	Add "8. Judgment of Inspection Results" and "9. Marking , Packaging, Transportation and Storage" live". Remove "8 Test Reports" from ISO 591-1: 2000 .	Suitable for my country's national conditions and user needs

References

[1] GB1706— Titanium dioxide pigment (neq) ISO 591:1977

[2] 1993 Specifications and test methods for water used in analytical laboratories (GB /T) 6682—1992, neq 1 SO

[3] GB/T6682 3696:1987)

HG/T 2457 General Rules for Inspection, Marking, Packaging, Transportation and Storage of Pigment Products (HG /T) 2457—1993)

GBH¹706-2006

Book Number: 155066·1-28769
Pricing: 10.00 Yuan

ICS 87.060.10
G 54

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

数码防伪

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"TiO_2 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　一色度法";

——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、淄博钴业股份有限公司、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 特性要求和容许范围

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〔$FeNH_4(SO_4)_2 \cdot 12H_2O$〕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±2)℃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：

$$T_1 = \frac{m_1 \times w(TiO_2)}{V_1 \times 100} \qquad \cdots\cdots(1)$$

式中：

m_1——所用二氧化钛标准参比物质的质量，单位为克(g)；

$w(TiO_2)$——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 $w(TiO_2)$ 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 $c\left(\frac{1}{5}KMnO_4\right)=0.1$ 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±2)℃。

7.1.3.4 干燥器：内盛合适干燥剂，如硅胶。

单位为毫米

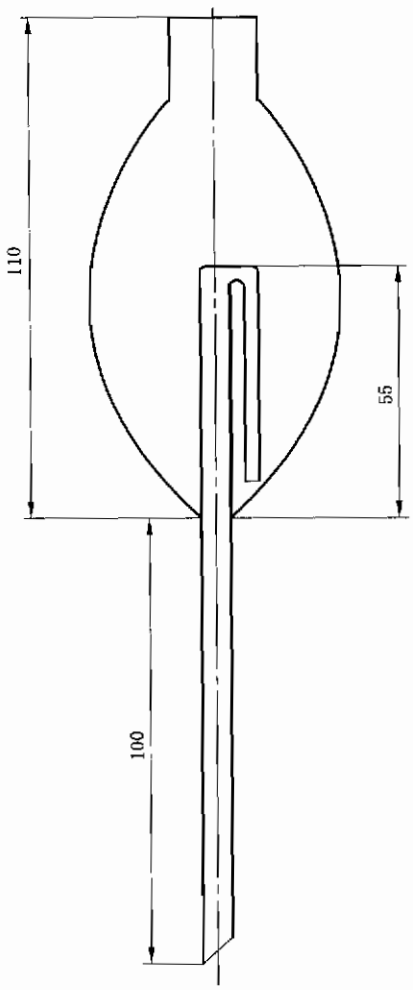


图 1　玻璃液封管

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±2)℃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℃,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:

$$\omega(TiO_2) = \frac{V_2 \times T_1 \times 100}{m_2} \qquad \cdots\cdots(2)$$

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随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 ϕ70 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

*

书号:155066·1-28769

定价: 10.00 元