1. SUBJECT OF THE STANDARD

A subject-matter of the Standard is titanium white - an inorganic pigment whose main component is titanium dioxide.

2. APPLICATIONS

This Standard concerns titanium white applied as a pigment in the industries of paints and coatings, plastics, paper, rubber, cement, ceramics, etc.

3. RELATED STANDARDS

PN-EN ISO 15528:2013-10 Paints, varnishes and raw materials for paints and varnishes - Sampling

PN-EN ISO 591-1:2002 Titanium dioxide pigments for paints – Part 1: Specifications and methods of test

PN-EN ISO 787-2:2000 General methods of test for pigments and extenders - Part 2: Determination of matter volatile at 105 degrees C

PN-EN ISO 787-3:2002 General methods of test for pigments and extenders – Part 3: Determination of matter soluble in water – Hot extraction method

PN-EN ISO 787-5:1999 General methods of test for pigments and extenders – Part 5: Determination of oil absorption value

PN-EN ISO 787-9:2000 General methods of test for pigments and extenders – Part 9: Determination of pH value of aqueous suspension

PN-EN ISO 787-14:2003 General methods of test for pigments and extenders – Part 14: Determination of resistivity of aqueous extract

PN-EN ISO 787-18:1999 General methods of test for pigments and extenders – Part 18: Determination of residue on sieve - Mechanical flushing procedure

PN-EN ISO 787-24:1999 General methods of test for pigments and extenders – Part 24: Determination of relative tinting strength of coloured pigments and relative scattering power of white pigments – Photometric methods

PN-EN 27965-1:1994 Packaging -- Sacks -- Drop test -- Part 1: Paper sacks

PN-EN ISO 780:2016-03 Packaging -- Distribution packaging -- Graphical symbols for handling and storage of packages

PN-EN 13698-1:2005 Pallet production specification. Part 1: Construction specification for 800 mm x 1200 mm flat wooden pallets

PN-EN 13698-2:2009 Pallet production specification. Part 2: Construction specification for 1000 mm x 1200 mm flat wooden pallets

4. CLASSIFICATION AND NOTATION

4.1. Classification

4.1.1. Types

Depending on the crystallographic structure, two types of titanium dioxide are distinguished in the titanium white:

A: anatase **R:** rutile

4.1.2. Grades

Depending on the type and the content of titanium dioxide, titanium pigments are classified into the following grades:

	A (anatase)		R (rutile)		
Grade	A1	A2	R1	R2	R3

4.2. Classification of TYTANPOL® titanium white

TYTANPOL®	A-11	R-001	R-002	R-003	R-210	R-211	R-220	R-213	R-310	RS
Type	A	R	R	R	R	R	R	R	R	R
Grade	A1	R2	R2	R2	R2	R2	R2	R3	R3	R1

4.3. Exemplary notation of TYTANPOL® titanium white, grade R2

TYTANIUM WHITE (R2) TYTANPOL® R-001

5. REQUIRED PROPERTIES

5.1. Appearance

The product shall be in the form of fine, dry, white powder; easy break up by pressing with spatula without grinding effect.

5.2. Chemical and physical requirements

The essential requirements are given in the Table 1, whereas conditional requirements are given in the Table 2. The conditional requirements have to be defined by interested parties by agreement.

Requirements concerning matter volatile at 105°C after preconditioning given in a Table 2 shall be relevant only, if this property is mentioned by interested parties or listed in the contract.

Agreed reference pigment, revealed in the Table 2 shall fulfil requirements according to the Table 1.

5.2.1. Essential requirements

Table 1

Property	Requirements depending on type and grade					Test method
	Type A		Type R			
	A1	A2	R1	R2	R3	
Titanium dioxide content, % (m/m), min.	98	92	97	90	80	PN-EN ISO 591-1:2002
Matter volatile at t=105ºC at dispatch time, % (m/m), max.	0.5	0.8	0.5	to be agreed between the interested parties		Pkt.7.6.2. of the Standard
Matter soluble in water, % (m/m), max.	0.6	0.5	0.6	0.5	0.7	PN-EN ISO 787-3:2002*
Residue on sieve mesh side (45μm), % (m/m), max.	0.1	0.1	0.1	0.1	0.1	PN-EN ISO 787-18:1999
* *If necessary coagulant can be applied.*						

5.2.2. Conditional requirements

Table 2

Property	Requirements depending on type and grade					Test method
	Type A		Type R			
	A1	A2	R1	R2	R3	
Colour 1. Comparison of lightness and full shade (colour tinge) of white system 2. Comparison of lightening power and undertone (colour tinge) of grey system	Similar to that of the agreed reference pigment					See Section 7.6.1 of the Standard
Scattering power	To be agreed between the interested parties					PN-EN ISO 787-24:1999
Matter volatile at 105ºC after 24 h preconditioning at 23±2ºC and relative humidity of 50 ± 5%, %(m/m), max.	0.5	0.8	0.5	1,5	2,5	PN-EN ISO 787-2:2000
pH value of aqueous suspension	Similar to that of the agreed reference pigment					PN-EN ISO 787-9:2000
Oil absorption value						PN-EN ISO 787-5:1999
Resistivity of aqueous extract	-	Similar to that of the agreed reference pigment		-	Similar to that of the agreed reference pigment	PN-EN ISO 787-14:2003

6. PACKAGING, STORAGE AND TRANSPORT

6.1. Packaging

Titanium white should be packed in the quantities of 25 +/- 0.125 kg to vented paper bags according to PN-EN 27965-1:1994 or in the quantities of 500 ± 2.5 kg or 1000 ± 5 kg to the respective flexible containers (i.e. big- bag). On every packaging an inscription shall be placed giving at least the following information:

a) name and/or brand of the manufacturer;
b) notation acc. to 4.3;
c) net weight;
d) batch number;
e) date of manufacture (if it is not included in the batch number).

Place where the inscriptions shall be located according to PN-EN ISO 780:2016-03

If a palletization is applied, unit loads with max. weight of 1 000 kg shall be formed on pallets 800x1200 mm according to PN-EN 13698-1:2005, on pallets 1000x1200 mm according to PN-EN 13698-2:2009 or on other pallets agreed upon with customer.

Number of layers of bags placed flatwise on a pallet, different depending on the size of a pallet, shall not exceed eleven. Load on a pallet should be so wrapped by means of the foil that it makes a compact, stable unit load together with the pallet.

Another type of packing and marking is allowable after the written agreement between the customer and the manufacturer, if it ensures preservation of the quality of the product, at least as effectively.

6.2. Storage

Titanium white shall be stored in original, undamaged packages acc. to 6.1, in roofed and dry rooms. Number of stored layers of bags placed flatwise in a stockpile shall not exceed twenty four. In case of a product stored in unit loads formed on pallets, it is allowed to pile maximum 3 pallets.

It is allowed to store a pigment in bulk after the written agreement between the customer and the manufacturer, if the storage conditions ensure preservation of the product's quality at least so effectively as for the package acc. to 6.1.

6.3. Transportation

Titanium white, packed and marked acc. to 6.1, shall be conveyed by roofed means of transport in accordance with the regulations being in force. Number of loaded layers of bags placed flatwise in a stockpile shall not exceed twenty four. In the case of transporting the product in transport units formed on pallets, stacking is allowed up to maximum 2 layers.

Bulk handling by means of transport destined for conveyance of dusty goods (e.g. vehicle for cement handling) is allowed after the written agreement between the customer and the manufacturer, if the transport conditions ensure preservation of the product's quality at least so effectively as for the package acc. to 6.1.

6.4. Functional quality

Titanium white TYTANPOL® is stable and chemically durable product.

In case of proper storage and transport according to the present standard the functional quality (useful life) of the product remains unchanged.

7. TESTING

7.1. Programme of testing

If it is not stated in a different way in the contract between producer and customer the product is subject to the incomplete or complete tests for conformity with requirements given in the section 5. The Programme of these tests is presented in the Table 3.

Table 3

Type of testing	Incomplete tests	Complete tests
Visual inspection	+	+
Determination of titanium dioxide content	+	+
Determination of matter volatile at the temperature of 105ºC	+	+
Determination of matter soluble in water	-	+
Determination of residue on sieve (45 µm)	+	+
Colour: 1. Comparison of lightness and full shade (colour tinge) of white system 2. Comparison of lightening power and undertone (colour tinge) of grey system	 + +	 + +
Comparison of scattering power	-	+
Determination of matter volatile at the temperature of 105ºC after 24h preconditioning at 23±2ºC and relative humidity of 50±5 %	-	+
Determination of pH for aqueous suspension	+	+
Determination of oil absorption value	+	+
Determination of resistivity for aqueous extract	+	+
Complete tests are carried out only on request of the customer. The incomplete tests are performed for every batch of the product defined acc. to7.2.		

7.2. Batch size

Batch of product consists of amount of the pigment of one type and grade of pigment originating from one stored batch.

7.3. Sampling

Sampling from individual batches shall be carried in accordance with standard PN-EN ISO 15528:2013-10

7.4. Preparation of sample for testing

Samples shall be prepared in accordance with the requirements of individual tests, if such requirements have been defined.

7.5. Purity of reagents

If the standard specifications don't provide otherwise, the used reagents shall be of analytical grade. For preparation of solutions shall be applied distilled water or one with equivalent purity.

7.6. Description of tests

Except determination of colour and moisture all tests are carried out according to methods given in the mentioned standards (see Tables 1 and 2).

7.6.1. Colour

Principle of the method is based on the spectrophotometric or colorimetric measurement of the tristimulus values X,Y,Z for white paste draw-downs and the determination on their lightness and shade and analogous measurement of the tristimulus values X,Y,Z for grey paste draw-downs and the determination on their lightning power and undertone.

7.6.1.1. Determination of lightness and shade (colour tinge) in white paste

7.6.1.1.1. Apparatus and materials

a) Spectrophotometer for the wavelength range of 400 ÷ 700 nm with the measuring geometric condition d/0 or a tristimulus colorimeter with the measuring geometric condition 45/0.
b) Laboratory triple-roll mill.
c) Automatic muller.
d) General laboratory balance with the accuracy of 0.01 g.
e) Grindometer with the measuring range of 0 ÷ 100 µm.
f) Film applicator enabling coating with a layer having the thickness of 150 µm.
g) Spatula or palette knife made of stainless steel or plastic.
h) Medical syringe with the volume of 5 cm^3.
i) White cardboard sheets of the "Morest" type.

j) Linseed and tung oil (20 % of tung oil), e.g. of the type 04/6, with the flow time (agreed upon viscosity) of 550 ÷ 650 s measured by means of a Ford viscosity cup having the orifice diameter of 4 mm.
k) Colloidal silica Aerosil 130.
l) Reference pigment of the rutile or anatase type, with the known value of lightness and shade, according to the type of test sample.

7.6.1.1.2. Preparation of original paste

Weigh 950 g of linseed and tung oil and 50 g of silica Aerosil 130, mix by means of a spatula and pass it through a triple-roll mill to obtain the fineness of grind below 10 µm, measured by means of a grindometer. Place the prepared paste in a tight container.

7.6.1.1.3. Preparation of white paste

Weigh 3 g of a reference rutile pigment or 2.8 g of a reference anatase pigment, to the nearest 0.01 g. Measure by means of a syringe 2.8 ml of the original paste, place it on the lower plate of an automatic muller and mix by means of spatula with weighed amount of the reference pigment. Lower the upper plate of the automatic muller and grind the mixture under the load of 0.02 MPa in two stages, each of them consisting of 25 turns. After each stage, the paste shall be collected with the spatula, spreaded on a circular area with the diameter of ca. 50 mm over the lower plate and the spatula cleaned by wiping it on the upper plate.

White paste for the test pigment shall be prepared in the same way.

7.6.1.1.4. Execution of measurements

The milled white paste for the reference pigment shall be put by means of a film applicator on a white "Morest" cardboard sheet and, immediately after the application, its tristimulus values X, Y, Z shall be measured with a spectrophotometer or tristimulus colorimeter. Perform the measurement once more after the repeated preparation of white paste. In case of differences between the measurements exceeding 0.2 for the respective tristimulus values X, Y, Z, the process of white paste preparation and the execution of measurement shall be repeated.

Measurement for the test pigment shall be performed in the same way.

Calculation of results

7.6.1.1.4.1. Calculation of lightness

Colour lightness L_s of the test pigment shall be calculated using the equation:

$$L_s = L_R + (Y_S - Y_R) \qquad (1)$$

where:

L_R - is the known value of lightness for the reference pigment,

Y_S - is the average value of tristimulus value Y for the test pigment,

Y_R - is the average value of tristimulus value Y for the reference pigment.

The result shall be rounded to the nearest 0.1.

7.6.1.1.4.2. Calculation of full shade of white paste

Colour (full) shade S_s in the white paste of the test pigment shall be calculated using the equation:

$$S_S = S_R + (Z_s - X_s) - (Z_R - X_R) \qquad (2)$$

where:

S_R - is the known value of shade for the reference pigment,

X_S, Z_S - is the average value of tristimulus values X, Z for the test pigment,

X_R, Z_R - is the average value of tristimulus values X, Z for the reference pigment.

The result shall be rounded to the nearest 0.1.

7.6.1.2. Determination of lightening power and undertone of grey paste

7.6.1.2.1. Apparatus and materials

a) Spectrophotometer for the wavelength range of 400 ÷ 700 nm with the measuring geometric condition d/0 or a tristimulus colorimeter with the measuring geometric condition 45/0.
b) Laboratory triple-roll mill.
c) Automatic muller.
d) General laboratory balance with the accuracy of 0.01 g.
e) Grindometer with the measuring range of 0 ÷ 100 µm.
f) Film applicator enabling coating with a layer having the thickness of 40 µm.
g) Spatula or palette knife made of stainless steel or plastic.
h) White cardboard sheets of the "Morest" type.
i) Linseed and tung oil (20 % of tung oil), e.g. of the type 04/6, with the flow time (agreed upon viscosity) of 550 ÷ 650 s measured by means of a Ford viscosity cup having the orifice diameter of 4 mm.
j) Colloidal silica Aerosil 130.
k) Carbon black.
l) Reference pigment of the rutile or anatase type, with the known value of lightening power and undertone in the grey paste, according to the type of test sample.

7.6.1.2.2. Preparation of original paste

Weigh 414 g of linseed and tung oil and 586 g of carbon black, mix by means of a spatula and pass it through a triple-roll mill to obtain the fineness of grind below 10 µm, measured by grindometer. Place the prepared paste in a tight container.

7.6.1.2.3. Preparation of black paste

Weigh 58 g of the original paste obtained acc. to 7.6.1.2.2 and 892 g of linseed and tung oil, as well as 50 g of colloidal silica, mix by spatula and pass it through a triple-roll mill to obtain the fineness of grind below 10 µm, measured by grindometer. Place the prepared paste in a tight container.

7.6.1.2.4. Preparation of grey paste

Weigh 3 g of a reference rutile pigment or 2.8 g of a reference anatase pigment, to the nearest 0.01 g. Weigh 3 g of the black paste obtained acc. to 7.6.1.2.3, place it on the lower plate of an automatic muller and mix by means of a spatula with the weighed amount of the reference pigment. Lower the upper plate of the automatic muller and grand the mixture under the load of 0.02 MPa in two stages, each of them consisting of 25 turns. After every stage, the paste shall be collected with a spatula; spreaded on a circular area with the diameter of ca. 50 mm over the lower plate and the spatula shall be cleaned on the upper plate. Grey paste for the test pigment shall be prepared in the same way.

7.6.1.2.5. Execution of measurements

The ground grey paste for the reference pigment shall be put by means of a film applicator on a white "Morest" cardboard sheet and, immediately after the application, its tristimulus values X, Y, Z shall be measured with a spectrophotometer or tristimulus colorimeter. Perform the measurement once more after the repeated preparation of grey paste. In case of differences between the measurements exceeding 0.2 for the respective tristimulus values X, Y, Z, the process of grey paste preparation and the performance of measurements shall be repeated.

Measurement for the test pigment shall be performed in the same way.

7.6.1.2.6. Calculation of results

7.6.1.2.6.1. Calculation of lightening power

Lightening power P_S of the test pigment shall be calculated using the equation:

$$P_S = P_R + (Y_S - Y_R) \times 100 \qquad (3)$$

where:

P_R - is the known value of lightening power for the reference pigment,

Y_S - is the average value of tristimulus value Y for the test pigment,

Y_R - is the average value of tristimulus value Y for the reference pigment.

The result shall be rounded to the nearest 10.

7.6.1.2.6.2. Calculation of undertone of grey paste

Undertone U_S in the grey paste of the test pigment shall be calculated using the equation:

$$U_S = U_R + (Z_S - X_S) - (Z_R - X_R) \qquad (4)$$

where:

U_R - is the known value of undertone for the reference pigment,

X_S, Z_S - is the average value of tristimulus values X, Z for the test pigment,

X_R, Z_R - is the average value of tristimulus values X, Z for the reference pigment.

The result shall be rounded to the nearest 0.1.

7.6.2. Determination of matter volatile at temp. 105°C

The method for determining the volatile content at temp. 105°C described below allows for precise and very quick measurement, which is especially important when handling bulk shipments of pigment (in tank trucks).

For arbitration purposes to determine the volatile content the PN-EN ISO 787-2: 2000 method should be applied.

7.6.2.1. Apparatus and materials

a) Moisture analyzer (weighing dryer) with an accuracy of 0.001 g with a halogen heating module (power max. 450 W)
b) stainless steel tray, round
c) sampling spatula

7.6.2.2. Execution of measurements

The determination of the volatile content by a moisture analyser is fully automatic. Put a sample of the substance in the amount of 5 g ± 0.7 g on the moisture analyser tray in a thin even layer. Set the drying temperature at 105°C and drying time at 20 minutes. Close the moisture analyser lid with the handle. The measurement starts immediately after closing. After 20 minutes, the measurement is automatically stopped. Read the result.

7.6.2.3. Results

The arithmetic mean of at least two parallel determinations should be adopted as the result, if they differ by no more than 10% of the higher value. The result should be given with an accuracy of 0.1%.

7.7. Assessment of test results

Batch of the product is ought to be accepted as complying with requirements of the standard if the results of production tests for a sample collected acc. to 7.3 comply with the requirements given in section 5.

In the case of a separate contract between the manufacturer and the customer, the assessment of measurement results should be made in compliance with the requirements of the contract.

7.8. Manufacturer's certificate on test results

The manufacturer is obliged to present for every batch of product a certificate confirming its compliance to the requirements of the standard or contract.

THE END