

TITANIUM DIOXIDE TSM (TCM)

SPECIFICATIONS

TU 301-10-012-89

(Abstract)

The Specifications refer to nonpigmented rutile titanium dioxide TSM (TCM) which is powder poor-dispersed product of a yellowish or greyish tint produced from titanous chips and intended for manufacture of welding materials.

1 Technical Requirements

1.1 Titanium dioxide TSM (TCM) shall be produced pursuant to the requirements of this specifications and Manufacturer's process regulations approved in accordance with the established procedure.

1.2 Characteristics

1.2.1 The requirements to quality data of titanium dioxide TSM (TCM) are given in Table 1.

Table 1

Parameter	Value	Test method
Weight ratio of titanium dioxide, %, minimum	98	As per item 4.3
Weight ratio of phosphorus compounds in terms of P ₂ O ₅ , %, maximum	0.03	As per item 4.4
Weight ratio of sulphur compounds in terms of SO ₃ , %, maximum	0.08	As per item 4.5
Volatiles, weight ratio, %, maximum	0.3	As per item 4.6
Residue after wet sifting on mesh 0056, %, maximum	0.4	As per item 4.7

1.3 Packing

Prior to packing titanium dioxide, ensure that package, special containers and transport means are free of dust, garbage and remains of other materials.

Titanium dioxide shall be packed in six-layer paper or polyethylene bags with capacity of 25 kg, maximum. The bags shall be filled to 98 % of their volume maximum.

Paper bags shall be machine-stitched. Polyethylene bags shall be welded.

When transporting by motor, bags may be bound with cord or strength belts.

1.4 Marking

Each container unit with titanium dioxide shall be marked with the following data:

- manufacturer's name and trademark or its trademark only;
- product description and grade;
- color;
- gross weight and net weight;
- batch number;
- manufacture date;
- designation of the present specifications.

Paper bags with titanium dioxide shall be marked with the handling sign “Keep dry”.

2 Safety Requirements

2.1 The titanium dioxide is rated as IV hazard class substance in terms of action on human body.

2.2 Titanium dioxide dust gets into human body through respiratory organs and accumulating during long-term inhalation in case of high dust level, it may cause lungs disease.

2.3 Maximum allowable concentration of titanium dioxide in working air shall not exceed 10 mg/m^3 . The titanium dioxide in the air and waste water in the presence of other substances or factors does not form any toxic compounds.

The approximate safe level of titanium dioxide effect in the atmosphere air of populated areas is 0.5 mg/m^3 .

Maximum allowable concentration of titanium dioxide in utility basins water is 0.1 mg/dm^3 .

2.4 No non-recoverable wastes.

Waste water resulted from the manufacture of titanium dioxide flows into plant-wide sewerage for further treatment and settling.

For the purpose of atmosphere air protection against pollutants, maximum permissible emissions shall be controlled.

2.5 The titanium dioxide in the air and waste water in the presence of other substances or factors does not form any toxic compounds.

2.6 Clear titanium dioxide is fire and explosion-proof.

2.7 Conditions of titanium dioxide production and its use shall meet the requirements of "Sanitary Regulations of Processes Arrangement and Hygienic Requirements for Production Equipment" related to the production of dry and dusty materials.

For collective protection provide equipment sealing.

Production floor areas or laboratory premises shall be equipped with supply and exhaust ventilation to provide proper condition of the working air. Areas of dusting shall be equipped with the local aspiration system with dusty air purification in the bag filters FRKI (ФРКИ). The captured product returns to the process cycle.

2.8 All persons handling with titanium dioxide shall follow the personal hygiene rules, provided with protective clothing, shoes, hand protection, dust respirators ShB-1 (ШБ-1) "Lepestok-200".

2.9 Preliminary medical examinations upon entering employment and periodic medical examinations once per 24 months are required.

3 Acceptance Rules

3.1 Titanium dioxide shall be accepted in batches. A batch is amount of material, uniform in substance quality of the same grade and color, manufactured during one process cycle and accompanied with a quality document.

3.2 The quality document shall contain the following information:

- manufacturer's name and trademark or its trademark only;
- product description and grade;
- net weight;
- batch number;
- manufacture date;
- results of tests performed or confirmation of product compliance with the requirements of the present specifications;
- container types and number of packing units in the batch;
- designation of the present specifications.

4 Test Methods

4.1 Sampling

4.1.1 Sampling is performed as follows:

- snap samples are taken from packing units selected for testing;
- integrated sample is composed from snap samples;
- an average sample is taken from the integrated sample.

Snap samples are taken from any mass point submerging the sampling instrument along the vertical axis or diagonally.

All taken snap samples are joined together, mixed thoroughly, and the integrated sample of material is produced

An average sample mass shall be not less than 0.5 kg that is put into paper or polyethylene bag or cardboard case.

Paper bags and cardboard cases are not allowed in case of moisture test.

4.1.2 Vessels, bags and cardboard cases containing average sample are provided with a tag including the following information:

- product description and grades;
- manufacturer's name;
- batch number;
- date of sampling;
- sampler's name.

4.2 For analysis and preparation of solutions use reagents with classification of a.p. (ч.д.а.), ch.p. (х.ч.) or b.p. (ос. ч.).

When weighing, use laboratory balances of the second accuracy class with the maximum weighting limit of 200 g and graduation mark of 0.0002 g and the third class accuracy with the maximum weighting limit of 1,000 g and graduation mark of 0.01 g.

It is allowed to use other measuring instruments and other equipment whose metrological characteristics and specifications, respectively, are no worse than those specified in this document, as well as other reagents with quality no lower than specified herein.

When processing the definitive result shall be rounded to a numeric value of the same grade as the standardized value of grade composition.

4.3 Determination of titanium dioxide weight ratio

4.3.1 Equipment, materials and reagents applied

The unit for determination of titanium compounds (Figure 1).

Laboratory drying electric furnace providing heating up to the temperature of 105 °C and above, accuracy of automatic temperature control of ± 3 °C.

A hot plate with power selector switch.

Glass wool.

Watch glass with diameter of 40 to 70 mm.

A drop-meter without a cock, of the second accuracy class and with the capacity of 50 cm³, the graduation mark of 0.01 cm³.

A flask with two marks and the capacity of 100 cm³, the second accuracy class.

A measuring-glass with the capacity of 50 cm³ and the graduation mark of 5 cm³.

A round-bottomed flask with the capacity of 500 cm³ without an interchangeable cone, with the neck diameter of 34 mm, made of thermally resistant glass.

Technical carbonic acid.

Ammonium rhodanate, solution with weight ratio of 40 %.

Sulphuric acid, analytically pure, concentrated and solutions with volume concentration of 5 and 20 %.

Hydrochloric acid, analytically pure solution (1:1).

Ammonium iron alum, solution with concentration of 0.05 mol/dm³ in the sulphuric acid solution with mass concentration of 40 g/dm³.

Hydrogen peroxide (perhydrol), solution with weight ratio of 3 %.

Granulated zinc metal.

Cadmium metal, grade КД-0 with the following characteristics:

Weight ratio of cadmium is 99.96 % minimum;

weight ratio of impurities, %, maximum:

Zinc – 0.004;

Plumbum – 0.02;

Ferrum – 0.02;

Cuprum – 0.01;

Thallium – 0.003;

Nickel – 0.002;

Weight ratio of arsenic, antimony and slannum shall not exceed 0.003 % in total.

Titanium dioxide, especially pure.

Ammonium sulphate, analytically pure.

Distilled water.

4.3.2 Preparation for Analysis

4.3.2.1 Determination of weight ratio of the basic substance

In order to calculate molar (correction) factor of ammonium iron alum solution, weight ratio of the basic substance in the titanium dioxide shall be determined as per item 4.3.4, in this case weight quantity of 0.2000 g of titanium dioxide of basic portion 5–2, preliminary dried up to the constant weight at $(105 \pm 3) ^\circ\text{C}$.

4.3.2.2 Reduction gear preparation

The outlet of the reduction gear is filled with glass wool layer of 1 to 2 cm, then with distilled water to 1/2 of the height of the reduction gear and granular zinc (cadmium) (layer height is 28 to 35 cm), previously activated in an ingot-forming equipment with sulfuric acid solution of 20 % weight ratio, and washed with water.

After each 8 h of reduction gear operation the zinc (cadmium) is poured in the ingot-forming equipment and washed with water. The reduction gear is filled with 30 cm³ of sulfuric acid solution (1:1) that is left for 5–10 min, then the reduction gear is washed with water and filled with the washed zinc (cadmium) with adding of the activated zinc (cadmium). A zinc (cadmium) level in the reduction gear shall be always the same.

When decreasing zinc (cadmium) level, the reduction gear is added with a portion of activated zinc (cadmium).

The reduction gear in out-of-service condition shall be filled with water above the zinc (cadmium) level.

4.3.2.3 Determination of molar (correction) factor of ammonium iron alum dissolve 0.1000–0.1099 g of titanium dioxide, basic part, preliminary dried up to the constant weight at $(105 \pm 3) ^\circ\text{C}$ with the mixture of 5 cm³ of strong sulphuric acid and 2.5 g of ammonium sulphate, then having covered the beaker with the watch glass, heat actively it on the hot plate till its complete dissolution. The received solution shall be cooled, gradually adding 30 cm³ of water, mixed and tested as per item 4.3.5.

Molar factor (K) of the ammonium iron alum solution shall be calculated by the formula:

$$K = \frac{m \cdot x}{V \cdot 100 \cdot 0.003995} ,$$

where x – weight ratio of basic substance in the titanium dioxide, basic part 5-2, %;

V is the volume of ammonium iron alum solution with 0.05 mol/dm³ concentration that was spent for titration, cm³;

m is weight quantity, g;

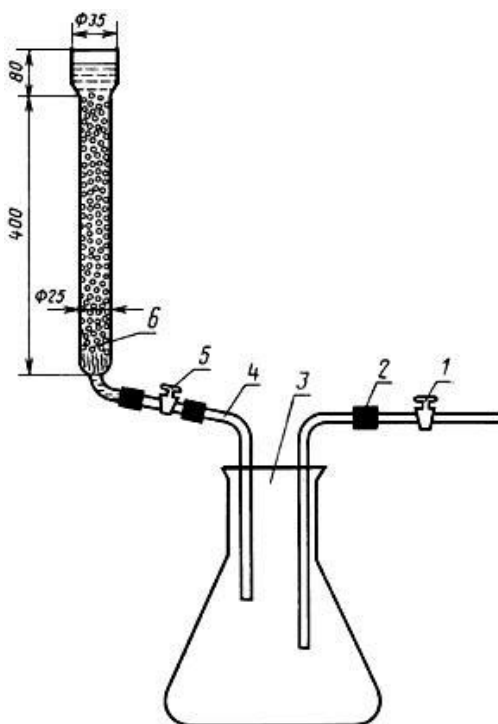
0.003995 is weight of titanium dioxide corresponding to 1 cm³ of ammonium iron alum solution of precisely 0.05 mol/dm³ concentration, g.

An arithmetic average value of three parallel measurements with the maximum permissible difference between them not exceeding 0.005 % shall be assumed as a test result, with confidence factor of 0.95.

4.3.3 Analysis Procedure

0.1000–0.1099 g of titanium dioxide preliminary dried up to the constant weight at $(105 \pm 3) ^\circ\text{C}$ shall be put in the beaker of 50 cm^3 , adding 2.5 g of ammonium sulphate and 5 cm^3 of strong sulphuric acid. The beaker is closed by the watch glass and heated till titanium dioxide dissolution. Then the solution is cooled, gradually adding 30 cm^3 of water, and mixed.

From reduction gear 6 of the unit (Figure 1) through cock 5 and tube 4 water is drained, the reduction gear is doubly washed with $20\text{--}25 \text{ cm}^3$ of sulphuric acid solution with weight ratio of 5 %, leaving the solution for 1–2 cm above the lower zinc (cadmium) level. This process shall be controlled with the cock.



- 1, 5 – cocks; 2 – tube for carbonic acid supply; 3 – flask for titration;
4 – tube for the tested solution drain; 6 – reduction gear for titanium recovery

Figure 1 – The unit for determination of titanium compounds

The solution under testing is quantitatively transferred into the reduction gear using sulfuric acid solution with volume ratio of 5 %, closed with the watch glass and recovered the titanium (IV) with the zinc for 10 min or cadmium for 30 min.

Flask for testing 3 is filled with 5 cm³ of ammonium rhodanate solution, then it is filled with carbonic acid and the recovered solution is drained from the reduction gear with continuous flow of carbonic acid. The watch glass shall be washed in 10 cm³ of sulphuric acid solution with volume ratio of 5 %, the solution is transferred to the reduction gear and jointed to the basic solution. Then the reduction gear is washed using sulphuric acid solution with volume ratio of 5 % and using water only once, filling the reduction gear by 1–2 cm above the zinc (cadmium) level. All washing water is drained in flask 3 (to check for washing accuracy through tube 4, 2–5 cm³ of washing water is taken and put in the beaker, then several drops of hydrogen peroxide are added inside; in this case color shall not be yellow) then it is titrated with ammonium iron alum solution until yellowish-orange color appears not vanishing during mixing.

4.3.4 Test Results Processing

Calculate titanium dioxide weight ratio X_1 , %, using the following formula:

$$X_1 = \frac{V \cdot K \cdot 0.003995 \cdot 100}{m},$$

where V is volume of ammonium iron alum solution spent for titration, cm³;

K – molar (correction) factor of the ammonium iron alum solution;

m – weight quantity, g;

0.003995 is weight of titanium dioxide corresponding to 1 cm³ of ammonium iron alum solution of precisely 0.05 mol/dm³ concentration, g.

An arithmetic average of two parallel measurements with absolute difference between them not above the maximum permissible difference equal to 1.0 % shall be assumed as a test result.

Allowable absolute combined error of test result is ± 1.0 % at confidence factor of 0.95.

4.4 Determination of weight ratio for phosphorus compositions in terms of phosphorus oxide P_2O_5 .

4.4.1 Equipment, materials and reagents applied

Photoelectric colorimeter.

Laboratory chamber electric resistance furnace providing heating up to the temperature of 1,000 °C and above; accuracy of automatic temperature control of ± 2 %.

Laboratory drying electric furnace providing heating up to the temperature of 105 °C and above; accuracy of automatic temperature control of ± 3 %.

A hot plate with power selector switch.

The high platinum crucible with the capacity of 18 cm³ (Figure 2)

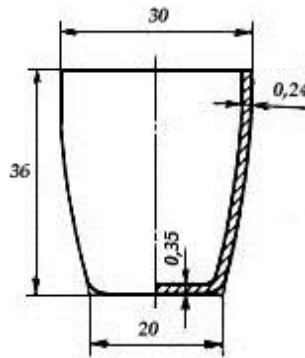


Figure 2 – High platinum crucible

The platinum lid (Figure 3)

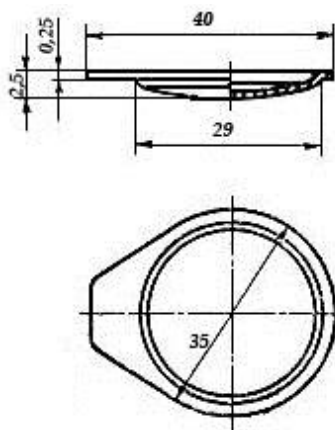


Figure 3 – Lid for the high platinum crucible

The glass mercury thermometer with the measurement limit from 0 to plus 150 °C.

The beaker with the rated capacity of 250 cm³, made of heat-resistant and chemically resistant glass.

The flask with one mark and the capacity of 50 cm³, the second accuracy class.

The flask with one mark and the capacity of 200 cm³, the second accuracy class.

The pipette with expansion in the shape of a barrel or a ball, accuracy class 2, capacity of 5 cm³.

The total delivery pipette, accuracy class 2, capacity of 5 cm³.

The total delivery pipette, accuracy class 2, capacity of 10 cm³.

Measuring tube with capacity of 100 cm³.

Glass funnel, diameter – 36 mm, height – 80 mm, made of chemically resistant glass.

Ash-free filter “Blue ribbon”.

The flask with the rated capacity of 250 cm³ without an interchangeable cone, with the neck diameter of 34 mm, made of thermally and chemically resistant glass.

Anhydrous sodium carbonate.

Potassium nitrate.

Ammonium molybdate, solution with weight ratio of 2.5 %.

Bismuth nitrate, solution with weight ratio of 1% in the sulphuric acid solution with concentration of 3.5 mol/dm^3 .

Potassium phosphate, monosubstituted.

Sulphuric acid, analytically pure solution with concentration 3.5 mol/dm^3 .

Ascorbic acid.

Ethyl alcohol, grade 1.

Phenolphthalein, alcoholic solution with weight ratio of 1 %.

Distilled water.

Colorimetric reagent; it is prepared with mixing of equal volumes of ammonium molybdate and bismuth nitrate solutions. Prior to using, add 1 g of ascorbic acid per each 100 cm^3 of the solution.

Phosphorus solutions A and B are prepared as follows.

Solution A.

0.4394 g of monosubstituted potassium phosphate, recrystallized twice and dried between sheets of filter paper shall be dissolved in water in the measuring flask of 1 dm^3 ; solution amount is brought to the mark with water and mixed thoroughly; 1 cm^3 of solution A contains 0.0001 g of phosphorus.

Solution B.

10 cm^3 of solution A is taken by a pipette and transferred to the measuring flask of 100 cm^3 , solution amount is brought to the mark with water and mixed thoroughly; 1 cm^3 of solution A contains 0.00001 g of phosphorus.

4.4.2 Calibration diagram plotting

$1.0; 2.0; 4.0; 5.0; 6.0 \text{ cm}^3$ of solution B are successively taken and put in the measuring flask of 50 cm^3 that corresponds to $1 \cdot 10^{-5}, 2 \cdot 10^{-5}; 4 \cdot 10^{-5}; 5 \cdot 10^{-5}; 6 \cdot 10^{-5}$ g of phosphorus, add 1 cm^3 of sulphuric acid solution, water up to the volume of 30 cm^3 and colorimetric reagent by 10 cm^3 . After adding each reagents,

solutions are mixed thoroughly. Solutions amount is brought with water up to the mark and mixed thoroughly.

10 minutes later measure extinction by the photoelectric colorimeter with red filter ($\lambda = 597 + 10$ nm) in a ditch with the light-absorbing layer thickness of 20 mm relative to the test solution containing all reagents except for the phosphorus.

Base on the obtained data the calibration diagram is constructed, plotting phosphorus weight in gram injected into the solution along the X-axis; and along the Y-axis – an arithmetic average of three measurements of optical density of each solution. The graduated solution is checked at the same time of test procedure but at least once a month.

4.4.3 Test Procedure

4.0 g of sodium carbonate, 0.1 g of potassium nitrate are added to titanium dioxide of 0.5000–0.5099 g dried up to the constant weight at temperature of (105 ± 5) °C, and then melt in the platinum crucible for 40–45 min. Melt is leached out in the beaker with hot water at temperature of plus 70 to plus 90 °C.

In case of green color provided by manganate ions, 3–5 drops of ethyl alcohol are added and heated until this color disappears. The solution is transferred to the measuring flask with the capacity of 200 cm³. After cooling to the room temperature the solution is added with water up to the mark, mixed thoroughly and filtered through the dry filter “blue ribbon” to the dry flask, removing the first 15–20 cm³ of filtrate. 5 cm³ of filtrate is put into the measuring flask of 50 cm³, 1 drop of phenolphthalein is added, then it is saturated by sulphuric acid solution until the solution is colorless. Then add 1 cm³ of sulphuric acid solution once more, water to the amount of 30 cm³, 10 cm³ of colorimetric reagent and bring to the mark by water. After adding each reagents, solutions are mixed thoroughly. After 10 min measure optical density by the photoelectric colorimeter as specified in item 4.4.2. The solution containing all reagents used in the course of the analysis is considered as the test one.

4.4.4 Processing of Results

Weight ratio of phosphorus compounds in terms of phosphorus oxide P_2O_5 X_2 , %, is calculated by the formula:

$$X_2 = \frac{m_1 \cdot 200 \cdot 2.29 \cdot 100}{m \cdot 5},$$

where m_1 – phosphorus weight determined by the calibration diagram, g;

m – weight quantity, g;

2.29 – scale factor in conversion from phosphorus to phosphorus dioxide P_2O_5 ;

5 – volume of the aliquot part, cm^3 ;

200 – total solution volume, cm^3 .

An arithmetic average of two parallel measurements with absolute difference between them not above the maximum permissible difference equal to 0.04 % shall be assumed as a test result.

Allowable absolute combined error of test result is ± 0.03 % at confidence factor of 0.95.

4.5 Determination of Weight Ratio of Sulfur Compounds in Terms of Sulfur Oxides SO_3

4.5.1 Equipment, materials and reagents applied

Laboratory tube-type electric resistance furnace providing heating of operating space up to the temperature $(1,250 \pm 25)$ °C.

Laboratory electric drying oven providing temperature of (105 ± 5) °C.

General-purpose laboratory balance of accuracy class 2 with the maximum weighing limit of 200 g and class 3 with the maximum weighing limit of 100 g.

Quartz or porcelain tube with the length of 600–700 mm with inside diameter of 18–20 mm. Prior to application, the tube shall be incinerated at the test temperature in the oxygen atmosphere (air).

Unglazed porcelain trays for incineration, length of 85 mm or 105 mm. Trays for work is prepared by incineration at the test temperature in the oxygen atmosphere (air) for 3–4 min. Completeness of sulphur burn-out from the tray is controlled by passing gaseous products from the furnace through starch solution containing 1–2 drops of iodine. The end of sulphur burn-out is determined when iodide starch solution colorless is finished.

Drop-meter of 2nd accuracy class, capacity of 25 cm³, division value is 0.05 cm³.

Flask with one mark or a ground plug, capacity of 1,000 cm³, accuracy class 2.

Standard titanium slag sample

Iodine, solution with concentration $C(1/2 I_2) = 0.01 \text{ mol/dm}^3$. To prepare the solution, take 1.27 g of metal iodine, dissolve it in 50 cm³ of potassium iodine solution with weight ratio of 5 % and dilute with water to 1 dm³. The solution is stored in dark glassware.

Potassium iodide, fresh solution with weight ratio of 5 %.

Potassium permanganate, solution with weight ratio of 0.3 % in the solution KOH with weight ratio of 10 %

Potassium hydroxide, solution with weight ratio of 10 %.

Starch, fresh solution with weight ratio of 1 %.

Technical gaseous oxygen or live oxygen in barrels

Sodium hydroxide, solution with weight ratio of 30 %

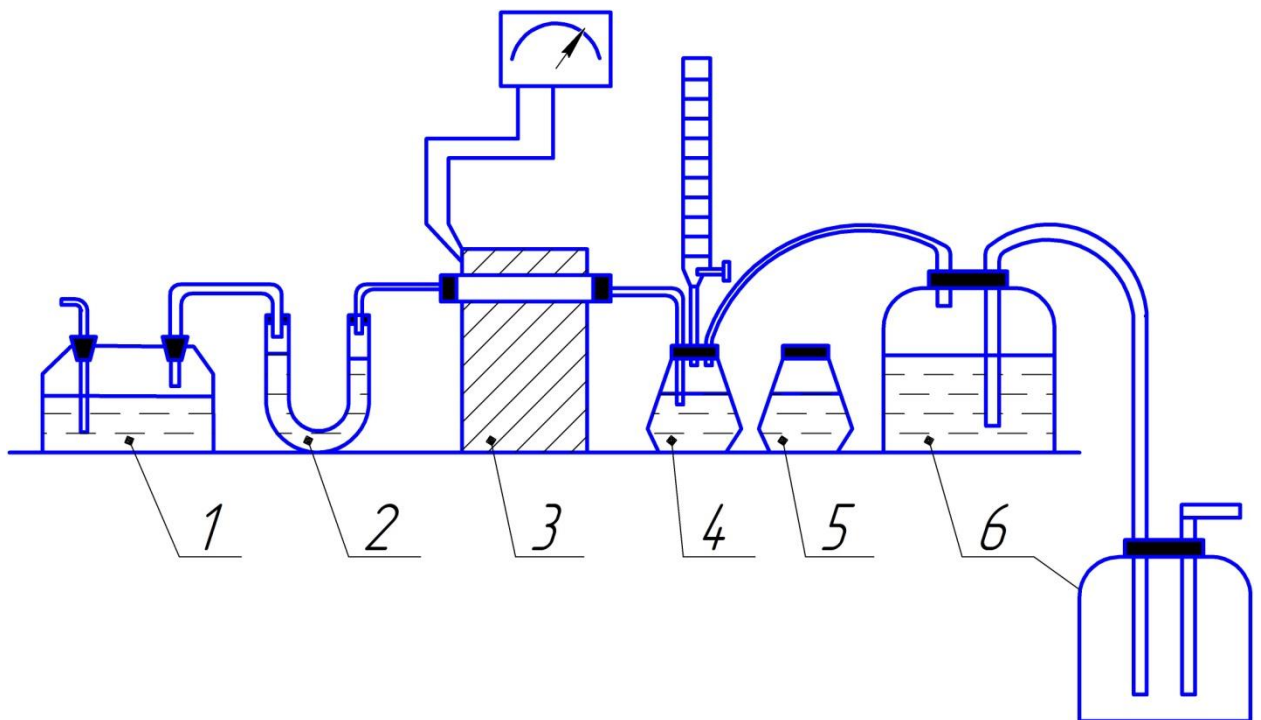
Strong sulphuric acid

Molten calcium chloride

Distilled water

The unit for determination of sulphur (Figure 4 or 5).

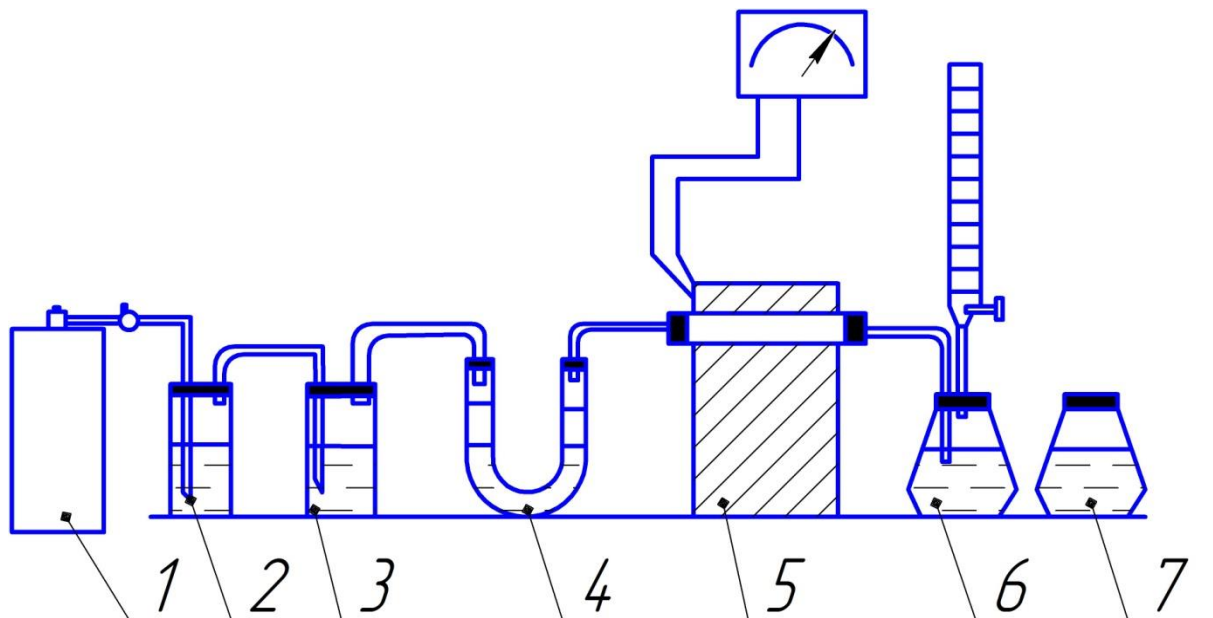
Prior to analysis, the unit (Figure 4) shall be checked for tightness at the test temperature and volatile chemical reducing agents. For this purpose the unit is connected to the oxygen cylinder, three-way cock downstream of U-shape tube with the calcium chloride is opened, in the air, and carefully opening the cylinder, pass the oxygen with the rate of 20–30 bubbles per minute. Then the cock is switched so that the oxygen flows in the furnace, clamp the connecting tube upstream of the absorption flask by the clamp lock. In 2–3 min bubbles generation in the cleaning flask stops. Wait for 5–7 min more and if there are no bubbles, the unit is considered as to be sealed.



1 – glassware with solution KMnO_4 ; 2 – tube with molten calcium chloride;
 3 – tube-type furnace; 4 – absorption flask; 5 – flask for comparison; 6 – aspiration unit;
 7 – drop-meter; 8 – millivoltmeter

Figure 4 – The unit for determination of sulphur by air aspiration

When testing the unit (Figure 5) for tightness, air is passed with the rate of 20–30 bubbles per minute. Then the clamp-lock is closed upstream of the cleaning flask with KMnO_4 and wait for 3-5 min. If there are no bubbles, the unit is considered as to be sealed. Then 150 cm^3 of water, 5 cm^3 of starch solution and several drops (0.5 cm^3) of iodine solution from the drop-meter are added into two absorption vessels. At the test temperature of $(1,250 \pm 25) \text{ }^\circ\text{C}$ oxygen (air) flow is passed through the furnace. If after 4–5 min solutions color in the absorber vessel disappears without stopping flow of oxygen (air), add several drops of iodine solution and continue to add iodine solution until the blue color remains constant and common intensity with the solution color in the test vessel.



1 – oxygen cylinder; 2 – flask with alkali liquor; 3 – flask with sulphuric acid;
 4 – tube with molten calcium chloride; 5 – tube-type furnace; 6 – absorption flask;
 7 – flask for comparison; 8 – drop-meter; 9 – millivoltmeter

Figure 5 – Unit for sulphur determination

Proper operation of all equipment can be checked as per the analysis of the standard specimen with known content of sulphur compounds by incinerating the specimen for several times.

4.5.2 Determination of iodine mass concentration

0.5000–0.5100 g of standard specimen of titanium slag or other standard specimen of this type is incinerated in the unit and titrated according to item 4.5.3.

Mass concentration of iodine solution C , g/cm^3 of sulphur trioxide is calculated by the formula:

where a – weight ratio of sulphur in the standard sulphur containing specimen, %;

2.5 – scale factor in conversion from sulphur to sulphur trioxide;

m – standard specimen weight, g;

V_1 – volume of iodine solution spent for titration, cm^3 ;

4.5.3 Analysis Procedure

1.0000 g of titanium dioxide in a uniform layer is put into the annealed tray.

Then the plug from the side of the oxygen cylinder from the porcelain tube of furnace prepared for operation; the tube is placed into the tray with sample of titanium dioxide, and using long wire hook it is moved to the furnace center; the tube is immediately plugged and the specimen is incinerated.

When gases flowing from the furnace to the absorption vessel starts bleaching of iodine solution, add this solution from the drop-meter with such rate that blue color of the solution does not disappear during incineration.

With decreasing rate of solution bleaching, slow down pouring of iodine solution and stop completely when the blue color in the absorption vessel remains the same as in the control vessel. Then oxygen is passed during one minute and if the color disappears, incineration is considered to be finished.

The test may be performed in the air flow by aspiration, in this case the oxygen cylinder is excluded from the unit. The analysis progress and result processing are the same as for incineration in the oxygen flow from the cylinder.

4.5.4 Processing of Results

Weight ratio of sulphur compounds X_{SO_3} in terms of sulphur trioxide, %, is calculated by the formula:

$$X_{SO_3} = \frac{V_2 \cdot c \cdot 100}{m},$$

where V_2 – volume of iodine solution spent for titration, cm^3 ;

c – mass concentration of iodine solution in g/cm^3 of sulphur trioxide;

m – weight of the sample, g.

An arithmetic average of two parallel measurements with absolute difference between them not above the maximum permissible difference equal to 0.006 % shall be assumed as a test result.

Allowable absolute combined error of test result is ± 0.003 % at confidence factor of $P = 0.95$.

4.6 Determination of Water and Volatile Matters Content

4.6.1 Method for the Determination of Water and Volatile Matters Content in the Exiccator

4.6.1.1 Equipment, materials and reagents applied

small beaker for weighing;

exiccator;

technical grade calcium chloride, incinerated.

4.6.1.2 Test Procedure

Weight quantity of product of 1 to 2 g is weighed with error of 0.0002 g maximum in the closed beaker for weighing, previously dried to the constant weight.

The open beaker with weight quantity distributed by even layer on the beaker bottom, and the lid are placed inside the exiccator with the incinerated calcium chloride, and dried up to the constant weight.

The first weighing shall be carried out in 24 h, other ones shall be carried out in each 3 hours until constant weight is achieved.

Weighing shall be performed with the error of not more than 0.0002 g. Prior to weighing, the beaker shall be closed.

4.6.1.3 Processing of Results

Water and volatile matters content X_3 , %, is calculated by the formula:

where m – tested product weight quantity, g;

m_1 – weight of the beaker with product being tested before drying, g;

m_2 – weight of the beaker with product being tested after drying, g;

An arithmetic average of two parallel measurements with the maximum permissible difference between them not exceeding the values given in Table 2 shall be assumed as a test result.

Table 2

Water and volatile matters content, %,	Permissible difference between the results of parallel measurements, %
Up to 0.5	0.004
Over 0.5 to 1.0	0.07
Over 1.0	0.15

4.6.2 Method for the determination of water and volatile matters content by drying in the thermostatic oven

4.6.2.1 Equipment, materials and reagents applied

Drying oven providing heating temperature of (105 ± 2) °C;

small beaker for weighing;

exiccator;

technical grade calcium chloride, incinerated.

4.6.2.2 Test Procedure

Weight quantity of 5 g of the product being tested, previously dried to the constant weight, is distributed with even layer on the beaker bottom for weighing.

The beaker with the substance is closed with the lid and weighed with accuracy of 0.0002 g maximum.

Place the open beaker with the weight quantity of product being tested, and the lid, into the drying oven for 1 hour at a temperature of (105 ± 2) °C. The first weighing is carried out after drying for 2 h. The beaker shall be closed with the lid, cooled down inside the exiccator with the incinerated calcium chloride, with error of 0.0002 g maximum.

Further drying is performed for 30 min till constant weight, that is, until a difference between further weighing exceeds 0.0005 g.

Prior to each weighing, the beaker is kept inside the exiccator for the same time.

4.6.2.3 Processing of Results

Water and volatile matters content is calculated according to item 4.6.1.3.

4.6.3 Method for the determination of water and volatile matters content by drying with an infrared lamp

4.6.3.1 Equipment, materials and reagents applied

The unit for determination of water and volatile matters content with the infrared lamp (Figure 6). The unit comprises the infrared lamp fixed on the support and enclosed by the shield of tin-plate. A height of lamp suspension above the table shall be 5 cm minimum.

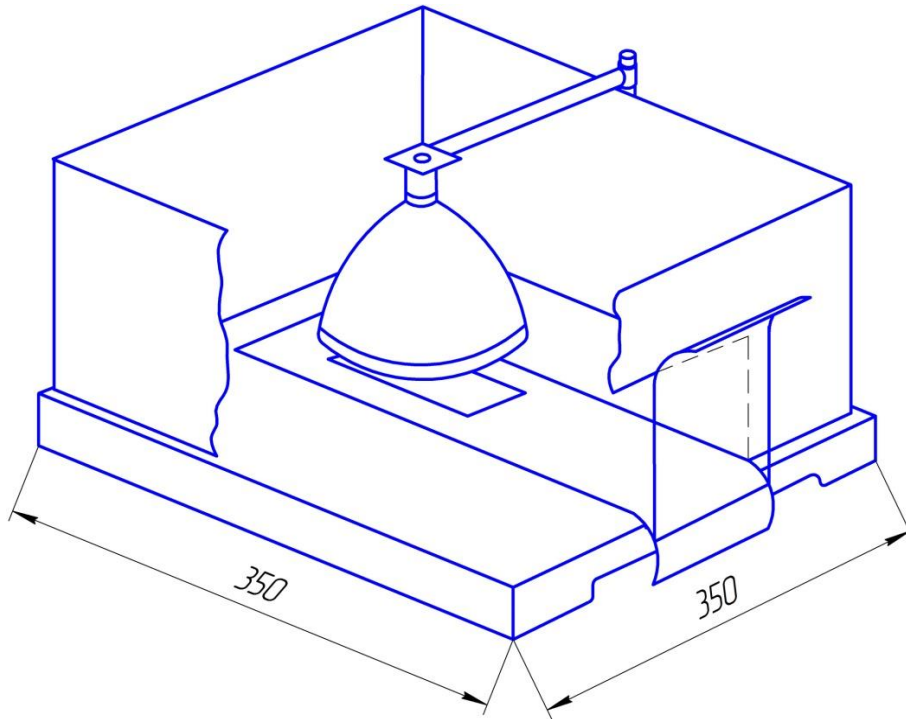


Figure 6 – Unit for the determination of water and volatile matters content

The front wall of the shield shall have an opening with a gate for installation of the tested specimens. A table of 350×350 mm is covered with asbestos, and its top – with tin-plate sheet. A shield height shall be such that to close the lamp. The unit is placed into the exhaust hood.

aluminum cup with thickness of 0.2 to 0.5 mm with the flat bottom of 70 to 90 mm in diameter, curb height of 5 to 10 mm;

watch glass;

small beaker for weighing;

exiccator;

technical grade calcium chloride, incinerated.

4.6.3.2 Test Procedure

Weight quantity of product of 1 to 2 g is weighed with accuracy of 0.0002 g maximum in the aluminum beaker closed with the watch glass or in the closed weighing bottle. The open beaker or cup with weight quantity and the lid are put inside the housing on the asbestos pad. Flow is supplied, the cup or the weighing

bottle with the tested product is placed in the circle center formed by the lamp light, and dried up the constant weight.

The first weighing is carried out after 15 min, further one – with the interval of 10 min.

Prior to each weighing, the cup or the beaker with the tested substance is closed by the lid, put into the exiccator with incinerated calcium chloride for cooling and kept for equal time intervals.

Water and volatile matters content is calculated according to item 4.6.1.4.

4.7 Determination of Sieve Residue after Wet Sifting

4.7.1 Equipment, materials and reagents applied

Drying oven providing heating temperature of $(105 \pm 2) ^\circ\text{C}$;

crystallized cylindrical cup;

watch glass;

beaker for weighing;

metal sieve, diameter of 200 mm, mesh of 0.056;

bristle brush.

4.7.2 Test Procedure

Weight quantity of titanium dioxide of 50 g is weighed with accuracy of 0.01 g maximum and put into the beaker of 1,000 cm³ capacity.

The titanium dioxide is dispersed with a brush in 500–600 cm³ of water and the produced dispersion is transferred on the sieve. The sieve residue is transferred into the beaker again, dispersion is repeated by the same water amount, and the dispersion is transferred on the sieve again. This operation is repeated for the third time, in this case the product remaining in the beaker is washed away by water and transferred on the sieve by the brush. The sieve with residues is put into the crys-

tallized cup half-filled with water and touch the sieve by the brush. The water in the cup is changed until after residue washing, it becomes clean, without traces of titanium dioxide. The test is considered to be finished if there is noticeable amount of titanium dioxide on the paper laid under the crystallized cup. Black paper is used during analysis. Titanium dioxide may be washed away under small amount of running water.

The sieve with residue is dried in the drying oven at (105 ± 2) °C. The dried residue is transferred on the previously weighted watch glass by the brush, and weight it with an error of 0.0002 g maximum.

4.7.3 Processing of Results

4.7.3.1 Sieve residue after wet sifting X_4 , %, is calculated by the formula

$$x_4 = m \cdot 100$$

where m is product weight, g;

m_1 is dry residue weight, g.

An arithmetic average of two parallel measurements with the maximum permissible difference between them not exceeding the values given in Table 3 shall be assumed as a test result.

Table 3

Sieve residue after sifting, %	Permissible difference between the results of parallel measurements, %	Sieve residue after sifting, %	Permissible difference between the results of parallel measurements, %
Up to 0.01	0.003	Over 0.1 to 0.5	0.04
Over 0.01 to 0.05	0.008	Over 0.5 to 1.0	0.07
Over 0.05 to 0.1	0.02	Over 1.0 to 1.5	0.15
		Over 1.5	0.25

5 Packing, Marking

5.1 Packing

Titanium dioxide is packaged in paper six-layer bags, polyethylene bags or packs with internal bag.

Containers shall be filled with power of titanium dioxide by 98 % maximum of full capacity of the container. When packing in paper six-layer bags, net weight shall not exceed 50 kg. Paper bags shall be machine-stitched. Polyethylene bags and packs shall be welded.

Titanium dioxide in the consumer container is packaged in the shipping container – cases made of wood or corrugated cardboard (Figure 7).

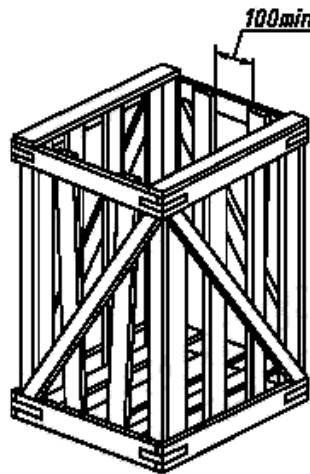


Figure 7 – Wooden case

Basic parameters of wooden cases shall meet the standard values given in Table 4.

Table 4 – Basic parameters of wood containers

Internal dimensions, mm			Limit material weight in the container, kg	Capacity, dm ³	Boards thickness, mm		Plates section, mm
length	width	height			end walls	bottom and lid side walls	
320	280	280	35	25.1	16	9	16 × 40
380	380	152	35	21.9	16	9	16 × 40
380	380	190	35	27.4	16	9	16 × 40
380	380	228	35	32.9	16	9	16 × 40
380	380	253	35	36.5	16	9	16 × 40
380	380	285	35	41.2	16	9	16 × 40
470	235	228	25	25.2	16	9	16 × 40
570	380	228	55	49.4	16	9	16 × 40
684	228	304	35	47.4	16	13	16 × 40
310	310	253	35	24.3	16	9	16 × 40
475	320	140	35	21.3	16	9	16 × 40
480	320	175	45	26.9	16	13	16 × 40
490	420	140	45	28.8	16	9	16 × 40

5.2 Marking

Each unit of consumer and shipping containers with titanium dioxide shall be marked with the following data:

manufacturer's name and trademark or its trademark only;

name, grade and class of material;

gross weight and net weight;

batch number;

manufacture date;

marking "Keep dry".

6 Transportation

The titanium dioxide shall be transported by any means of transport inside closed transport vehicles as per the shipping rules applicable for the given means of transport.

When transporting, multiple package and containers with titanium dioxide shall be combined into transportation blocks.

It is allowed shipping paint materials without combining them into transportation blocks in the following cases:

during transportation by motor transport;

during transportation by rail transport in carload lots in barrels with capacity above 100 dm³;

during transportation in the general-purpose containers, except for bulk ones.

When transporting materials inside metal shipping containers, wood block fillers shall be arranged between tiers of containers.

7 Storage

Packed titanium dioxide shall be stored in closed warehouses at ambient temperature of minus 40 °C to plus 40 °C.

It is allowed storing titanium dioxide at open areas when loading the cars for three days maximum.

During storage packages and special containers with titanium dioxide shall be laid in piles of 3 m high maximum on pads or wooden pallets.

Materials may be stored inside metal shipping containers laid in piles of 5.5 m high maximum. When laying, containers shall be arranged with the plugs and caps upwards.

8 Manufacturer's Warranty

The manufacturer guarantees the titanium dioxide compliance with the specifications provided that storage and use conditions are observed by the consumer.

The shelf life is 12 months after manufacture date.