

TITANIUM DIOXIDE

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SYNONYMS

Titania; CI Pigment white 6; CI (1975) No. 77891; INS No. 171

DEFINITION

Titanium dioxide is produced by either the sulfate or the chloride process. Processing conditions determine the form (anatase or rutile structure) of the final product.

In the sulfate process, sulfuric acid is used to digest ilmenite (FeTiO_3) or ilmenite and titanium slag. After a series of purification steps, the isolated titanium dioxide is finally washed with water, calcined, and micronized.

In the chloride processes, (a) titanium-containing mineral is reacted with chlorine gas under reducing conditions to form anhydrous titanium tetrachloride, which is subsequently purified and converted to titanium dioxide either by direct thermal oxidation or by reaction with steam in the vapour phase; (b) titanium-containing mineral is reacted with concentrated hydrochloric acid to form a solution of titanium tetrachloride, which is further purified and hydrolysed to get titanium dioxide. The compound is filtered, washed and calcined.

Commercial titanium dioxide may be coated with small amounts of alumina and/or silica to improve the technological properties of the product.

C.A.S. number 13463-67-7

Chemical formula TiO_2

Formula weight 79.88

Assay Not less than 99.0% on the dried basis and on an aluminium oxide and silicon dioxide free basis.

DESCRIPTION

White to slightly coloured amorphous powder

FUNCTIONAL USES

Colour

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4) Insoluble in water, hydrochloric acid, dilute sulfuric acid, and organic solvents. Dissolves slowly in hydrofluoric acid and hot concentrated sulfuric acid.

Colour reaction Add 5 ml sulfuric acid to 0.5 g of the sample, heat gently until fumes of sulfuric acid appear, then cool. Cautiously dilute to about 100 ml with water and filter. To 5 ml of this clear filtrate, add a few drops of hydrogen peroxide; an orange-red colour appears immediately.

PURITY

Loss on drying (Vol. 4) Not more than 0.5% (105°, 3 h)

Loss on ignition (Vol. 4) Not more than 1.0% (800°) on the dried basis

Aluminium oxide and/or silicon dioxide Not more than 2%, either singly or combined
See descriptions under TESTS

Acid-soluble substances Not more than 0.5%; Not more than 1.5% for products containing alumina or silica.
Suspend 5 g of the sample in 100 ml 0.5 N hydrochloric acid and place on a steam bath for 30 min with occasional stirring. Filter through a Gooch crucible fitted with a glass fibre filter paper. Wash with three 10-ml portions of 0.5 N hydrochloric acid, evaporate the combined filtrate and washings to dryness, and ignite at a dull red heat to constant weight.

Water-soluble matter (Vol. 4) Not more than 0.5%
Proceed as directed under acid-soluble substances (above), using water in place of 0.5 N hydrochloric acid.

Impurities soluble in 0.5 N hydrochloric acid Antimony: Not more than 2 mg/kg
Arsenic: Not more than 1 mg/kg
Cadmium: Not more than 1 mg/kg
Lead: Not more than 10 mg/kg
See description under TESTS

Mercury (Vol. 4) Not more than 1 mg/kg
Determine using AAS (Cold vapour generation technique). The selection of sample size and method of sample preparation may be based on principles of methods described in Volume 4 (under "General Methods, Metallic Impurities").

TESTS

PURITY TESTS

Impurities soluble in 0.5 N hydrochloric acid

Antimony, arsenic, cadmium and lead (Vol.4)

Transfer 10.0 g of sample into a 250-ml beaker, add 50 ml of 0.5 N hydrochloric acid, cover with a watch glass, and heat to boiling on a hot plate. Boil gently for 15 min, pour the slurry into a 100- to 150-ml centrifuge bottle, and centrifuge for 10 to 15 min, or until undissolved material settles. Decant the supernatant through Whatman No. 4 filter paper, or equivalent, collecting the filtrate in a 100-ml volumetric flask and retaining as much as possible of the undissolved material in the centrifuge bottle. Add 10 ml of hot water to the original beaker, washing off the watch glass with the water, and pour the contents into the centrifuge bottle. Form a slurry, using a glass stirring rod, and centrifuge. Decant through the same filter paper, and collect the washings in the volumetric flask containing the initial extract. Repeat the entire washing process two more times. Finally, wash the filter paper with 10 to 15 ml of hot water. Cool the contents of the flask to room temperature, dilute to volume with water, and mix.

Determine cadmium, and lead using an AA-Electrothermal atomization technique, antimony by ICP-AES technique and arsenic using atomic absorption hydride technique.

Aluminium oxide and/or Silicon dioxide

Weigh about 0.5 g of the sample to the nearest 0.1 mg, in a platinum or nickel crucible, add 5 g potassium hydroxide and 2 g boric acid, mix and melt completely using a torch burner and allow to stand at room temperature. Place the reaction product along with crucible into 150 ml hot deionized water in a 250-ml PTFE beaker and dissolve residue by agitation. Wash the crucible with hot deionized water and remove it. Add 50 ml hydrochloric acid and transfer the contents into a 250-ml polypropylene volumetric flask. Wash the beaker three times with hot deionized water, transfer the washings to the volumetric flask and make up to volume (Solution A). Prepare the test solution by 5 times dilution of Solution A with 2% hydrochloric acid. Analyze aluminium and silica in the test solution by ICP-AES technique (Vol. 4). Set instrument parameters as specified by the instrument manufacturer. Use analytical lines for Al (396.152 nm) and Si (251.611 nm) and construct standard curve using standard solutions 0.2 – 5.0 µg/ml each. Read the concentration of Al and Si in sample solution (as µg/ml) and calculate the aluminium oxide and silicon dioxide content of the sample using the formula:

$$\%Al_2O_3 = \frac{1.889 \times C \times 250 \times 5}{W \times 10^6} \times 100$$

$$\%SiO_2 = \frac{2.139 \times C \times 250 \times 5}{W \times 10^6} \times 100$$

Where: C is concentration of Al or Si in the test solution, µg/ml
W is weight of sample, g

METHOD OF ASSAY

Prepare the test solution by 1000 times dilution of Solution A (prepared in the PURITY TEST for Aluminium oxide and Silicon dioxide) with 2% hydrochloric acid, taking care that dilution factor in each dilution step shall not be more than 20. Analyze Titanium in the test solution by ICP-AES technique (Vol. 4). Set instrument parameters as specified by the instrument manufacturer. Use the analytical line for Ti (334.941 nm) and construct standard curve using Ti standard solutions: 0.5 - 1.5 µg/ml. Read the concentration in the sample solution (as µg/ml) and calculate the titanium dioxide content of the sample using the formula:

%TiO₂ (on the dried basis)

$$= \frac{1.668 \times C \times 250 \times 1000}{W \times 10^6 \times (100 - \%LOD - \%Al_2O_3 - \%SiO_2) / 100} \times 100$$

Where: C is concentration of Ti in the test solution, µg/ml
W is weight of sample, g
%LOD is % loss on drying
%Al₂O₃ and %SiO₂ are content (%) of Aluminium oxide and silicon oxide