

INTERNATIONAL STANDARD

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Paper and board — Determination of titanium dioxide content

Papier et carton — Détermination de la teneur en dioxyde de titane



Reference number
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Foreword

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International Standard ISO 5647 was prepared by Technical Committee ISO/TC 6, *Paper, board and pulps*.

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Paper and board — Determination of titanium dioxide content

WARNING — The method specified in this International Standard involves the use of some hazardous chemicals and of gases that can form explosive mixtures with air. Care shall be taken to ensure that the relevant safety precautions are observed.

1 Scope

This International Standard specifies a method for determining titanium dioxide content in all kinds of paper and board, in particular coated or filled products. It comprises two procedures for the final determination of titanium, one of them relying on spectrophotometry and the other on flame atomic absorption spectrophotometry. The limits of the determination depend on the amount of sample taken (8.1).

NOTE 1 The method is designed for the determination of titanium dioxide. Titanium present in other forms, for example as a constituent of clay, will not interfere in the determination.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 186:1985, *Paper and board — Sampling to determine average quality*.

ISO 287:1985, *Paper and board — Determination of moisture content — Oven-drying method*.

ISO 2144:1987, *Paper and board — Determination of ash*.

3 Definition

For the purposes of this International Standard, the following definition applies.

titanium dioxide content: The titanium dioxide content of paper or board (expressed on an oven-dry basis), determined after ashing the sample and dissolving the ash by the method specified in this International Standard.

4 Principle

Titanium dioxide is determined by ashing the sample and dissolution of the ash in sulfuric acid and ammonium sulfate. Determination of the titanium by spectrophotometry, applying hydrogen peroxide, or by flame atomic absorption spectrophotometry, adding potassium chloride.

5 Reagents

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity (5.1).

5.1 Distilled water or deionized water, conductivity less than 1 mS/m.

5.2 Sulfuric acid, (H_2SO_4), ρ density 1,84 g/ml.

5.3 Ammonium sulfate, $[(\text{NH}_4)_2\text{SO}_4]$.

5.4 Diluted sulfuric acid.

In a beaker with about 500 ml of water (5.1) add carefully 100 ml of sulfuric acid (5.2) and 40 g of ammonium sulfate (5.3). Dilute to 1 litre with water (5.1).

5.5 Standard titanium dioxide solution, 500 mg of TiO_2 per litre.

Weigh 0,500 g of titanium dioxide (TiO_2) in a 500 ml beaker, add 40,0 g of ammonium sulfate (5.3) and 100 ml of sulfuric acid (5.2). In a fume hood, gradually heat the mixture to boiling and then boil for 5 min to 10 min. Cover the beaker and allow the solution to cool to room temperature and, while stirring, pour it carefully into a larger beaker containing about 300 ml of water (5.1). When the solution has reached room temperature decant it quantitatively into a 1000 ml volumetric flask. Rinse both beakers with water and add the rinsings to the volumetric flask. Make up to the mark with water (5.1).

5.6 Hydrogen peroxide, solution containing 30 g of H_2O_2 per litre.

(This solution is only required for the spectrophotometric procedure.)

5.7 Potassium chloride, solution containing 20 g of KCl per litre.

(This solution is only required for the flame atomic absorption spectrophotometric procedure.)

5.8 Hydrochloric acid (HCl), solution 6 mol/l.

6 Apparatus

In addition to the usual laboratory apparatus the following are required.

6.1 Dishes, of platinum or quartz

Wash the platinum dishes thoroughly and remove any traces of impurities by cleaning with fine sand. Soak and boil the dishes in the 6 mol/l hydrochloric acid solution (5.8). Avoid contact with metals other than platinum.

Wash the quartz dishes thoroughly and soak and boil them in the 6 mol/l hydrochloric acid solution (5.8).

6.2 Spectrophotometer or filter photometer, for determining absorbance at 410 nm. It shall have 10 mm cells provided with lids. (This apparatus is only required for the spectrophotometric procedure.)

6.3 Atomic absorption spectrophotometer, fitted with an acetylene and dinitrogen monoxide burner and a titanium hollow-cathode lamp. (This apparatus is only required for the flame atomic absorption spectrophotometric procedure.)

7 Selection of the sample

Select a representative sample in accordance with ISO 186. Tear the air-dry sample into pieces of suitable size. Use protective cotton gloves. Do not use cut or punched edges or other parts where metallic contamination may have occurred.

8 Preparation of ash solution

8.1 Carry out the determination in duplicate. Weigh, to the nearest 0,01 g, two test portions of about 10 g of paper or board and place each test portion in a clean dish (6.1). Determine the moisture content of a separate test sample as prescribed in ISO 287.

NOTE 2 Select the amount of sample to be ashed, taking into account the anticipated titanium dioxide content. If a 10 g portion is ashed, the optimum range of the titanium dioxide content is from 1,5 g/kg to 8 g/kg (spectrophotometric procedure) or from 0,8 g/kg to 3,2 g/kg (flame atomic absorption spectrophotometric procedure).

8.2 Ash the test portion as described in ISO 2144. Allow the dish to cool in a desiccator. If the ash content of the sample is of interest, weigh the dish with its contents and calculate the amount of ash obtained.

8.3 Transfer the ash to a 250 ml glass beaker. Add 4 g of ammonium sulfate (5.3) and 10 ml of the sulfuric acid (5.2). Mix well and cover the beaker with a watch glass. Heat in a fume hood until fumes of sulfur trioxide are evolved, and continue heating for 30 min. Allow the solution to cool to room temperature and then carefully add it, drop by drop, to about 50 ml of water contained in another beaker. Allow the solution to cool and decant it into a 100 ml volumetric flask. Rinse both beakers with water and add the rinsings to the flask. Finally, make up to the mark with water, and mix well.

9 Reagent blank solution

Prepare a reagent blank solution, using 4 g of ammonium sulfate (5.3) and 10 ml of sulfuric acid (5.2) in a glass beaker, as described in 8.3.

10 Spectrophotometric procedure

10.1 Preparation of the calibration graph

Into one of a series of six 50 ml volumetric flasks, place 25,0 ml of the reagent blank solution (clause 9). Into the remaining five 50 ml volumetric flasks, transfer the quantities of the standard titanium dioxide solution (5.5), shown in table 1. To each flask, add 7,5 ml of the hydrogen peroxide sol-

ution (5.6), and make up to the mark with the diluted sulfuric acid (5.4), and mix well.

Table 1 — Set of calibrating solutions

Standard titanium dioxide solution (5.5) ml	Corresponding mass of TiO_2 mg/l
Reagent blank (clause 9)	0
5,0	50,0
10,0	100,0
15,0	150,0
20,0	200,0
25,0	250,0

For each solution, measure within 1 h of the preparation, the absorbance at 410 nm using 10 mm cells. Use the diluted sulfuric acid (5.4) in the reference cell.

Construct a calibration graph by plotting the absorbance readings against the corresponding titanium dioxide concentrations.

10.2 Preparation of the test solutions

If the ash solution (8.3) contains undissolved matter, filter it through a hardened ashless filter. Do not wash the filter.

With a pipette, transfer 25,0 ml of the filtrate to a 50 ml volumetric flask. Add 15 ml of diluted sulfuric acid (5.4) and 7,5 ml of the hydrogen peroxide solution (5.6). Make up to the mark with the diluted sulfuric acid (5.4) and mix well.

10.3 Spectrophotometric determination

If the test solution (10.2) is turbid or coloured, prepare a reference solution by transferring 25 ml of the filtrate to a 50 ml volumetric flask and making it up to the mark with diluted sulfuric acid (5.4).

If the test solution is clear and colourless, use the diluted sulfuric acid (5.4) in the reference cell.

Within 1 h, measure the absorbance at 410 nm in 10 mm cells. Subtract the value obtained for the reagent blank (clause 9) and read the titanium dioxide concentration from the calibration graph.

Proceed to clause 12.

11 Flame atomic absorption spectrophotometric procedure

11.1 Preparation of the calibration graph

11.1.1 Preparation of the calibration solution

Into one of a series of five 50 ml volumetric flasks, place 25,0 ml of the reagent blank solution (clause 9) and add 2,5 ml of the potassium chloride solution (5.7). Into the remaining four 50 ml volumetric flasks, transfer 2,5 ml of the potassium chloride solution (5.7) and add the quantities of the standard titanium dioxide solution (5.5) shown in table 2. For each flask, make up to the mark with the diluted sulfuric acid (5.4), and mix well.

Table 2 — Set of calibrating solutions

Standard titanium dioxide solution (5.5) ml	Corresponding concentration of TiO_2 mg/l
Reagent blank (clause 9)	0
5,0	50,0
10,0	100,0
15,0	150,0
20,0	200,0

11.1.2 Adjustment of apparatus

Prepare the spectrophotometer for titanium measurement according to the manufacturer's instructions using a wavelength of 364,3 nm.

CAUTION — To avoid explosion, it is necessary to light the burner with a mixture of air-acetylene before switching to acetylene-dinitrogen monoxide.

11.1.3 Reagent blank determination

Carry out a reagent blank determination (table 2, term 0) following the same mode of operation and utilizing the same quantities of reagent specified in 11.1.1, but omitting the standard titanium solution (5.5). Aspirate this solution into the flame, and adjust the instrument to zero absorbance against the reagent blank test solution.

11.1.4 Spectrophotometric measurement

Aspirate the series of titanium calibration solutions (11.1.1) in succession into the flame and measure the absorbance for each. Take care to keep the aspiration rate constant throughout the preparation of the calibration graph. Spray water through the burner after each measurement.

11.1.5 Plotting the calibration graph

Plot a graph taking as abscissae the titanium dioxide concentrations, expressed in milligrams per litre, and, as ordinates, the corresponding values for the measured absorbance.

NOTE 3 If an instrument is used equipped with a system for automatic calculation of data, the instructions in the paragraph above may be disregarded.

11.2 Determination

11.2.1 Preparation of test solution

Prepare the test solution using the ash solution (8.3). If the solution contains suspended matter, allow this to settle. Transfer with a pipette 25,0 ml of the solution to a 50 ml volumetric flask. Add 2,5 ml of the potassium chloride solution (5.7), make up to the mark with diluted sulfuric acid (5.4) and mix well.

11.2.2 Spectrophotometric measurement

Carry out the spectrophotometric measurement on the test solution prepared in 11.2.1 as specified in 11.1.4 after having adjusted the instrument to zero absorbance against the reagent blank test solution (11.1.3). Read the amount of titanium dioxide from the calibration graph.

NOTE 4 If the readings obtained for the test solution exceed the range of the calibration solutions, an appropriate dilution of the test solution should be made using the diluted sulfuric acid (5.4). Add an appropriate volume of the potassium chloride solution (5.7) to keep the potassium concentration at the same level as in the calibration solutions.

12 Expression of results

Calculate the titanium dioxide $w(\text{TiO}_2)$, expressed in grams per kilogram of an oven-dry sample using the following equation

$$w(\text{TiO}_2) = \frac{\rho(\text{TiO}_2) \cdot V_1 \cdot V_3}{10^3 V_2 \cdot m}$$

where

$\rho(\text{TiO}_2)$ is the titanium dioxide concentration, in milligrams per litre, of the test solution (10.2 for the spectrophotometric procedure, or 11.2.1 for the flame absorption procedure), read from the calibration graph;

V_1 is the volume, in millilitres, of the test solution (10.2 for the spectrophotometric procedure, or 11.2.1 for the flame absorption procedure), prepared for spectrophotometric measurement (standard 50 ml);

V_2 is the volume, in millilitres, of the ash solution (8.3), taken for the preparation of the test solution (standard 25 ml);

V_3 is the total volume, in millilitres, of the ash solution (8.3) (standard 100 ml);

m is the mass, in grams, of the test portion, calculated on an oven-dry basis.

NOTE 5 If the standard volumes are used, the expression reduces to

$$w(\text{TiO}_2) = \frac{0,2\rho(\text{TiO}_2)}{m}$$

13 Test report

The test report shall include the following particulars:

- date and place of testing;
- reference to this International Standard;
- the method used (spectrophotometry or flame atomic absorption spectrophotometry);
- all information necessary for complete identification of the sample tested;
- the mean result of the two determinations, to the nearest 0,1 g/kg;
- any departures from the standard procedure and any other circumstances that may have affected the test results.