
**Paper and board — Determination of
titanium dioxide content**

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

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 6, *Paper, Board and Pulps*.

This second edition cancels and replaces the first edition (ISO 5647:1990), of which it constitutes a minor revision. The changes compared to the previous edition are as follows:

- **Clause 4:** The option of using Inductively Coupled Plasma/Atomic Emission Spectrometry (ICP/AES) has been added.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Paper and board — Determination of titanium dioxide content

WARNING — The method specified in this document 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 document 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 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 documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

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ISO 287, *Paper and board — Determination of moisture content of a lot — Oven-drying method*

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ISO 2144, *Paper, board, pulps and cellulose nanomaterials — Determination of residue (ash content) on ignition at 900 °C*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

3.1

titanium dioxide content

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 document

4 Principle

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

The determination of titanium, after ashing the sample and dissolution of the ash, can also be done by ICP/AES (Inductively Coupled Plasma/Atomic Emission Spectrometry) provided that the results have been validated.

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 Sulphuric acid (H₂SO₄), ρ density 1,84 g/ml.

5.3 Ammonium sulphate [(NH₄)₂SO₄].

5.4 Diluted sulphuric acid. In a beaker with approximately 500 ml of water (5.1) carefully add 100 ml of sulphuric acid (5.2) and 40 g of ammonium sulphate (5.3). Dilute to 1 l with water (5.1).

5.5 Standard titanium dioxide solution, 500 mg of TiO₂ per litre.

Weigh 0,500 g of titanium dioxide (TiO₂) in a 500 ml beaker, add 40,0 g of ammonium sulphate (5.3) and 100 ml of sulphuric 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 1 000 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 H₂O₂ 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 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 sulphate (5.3) and 10 ml of the sulphuric acid (5.2). Mix well and cover the beaker with a watch glass. Heat in a fume hood until fumes of sulphur 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.

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9 Reagent blank solution

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Prepare a reagent blank solution, using 4 g of ammonium sulphate (5.3) and 10 ml of sulphuric 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 solution (5.6), and make up to the mark with the diluted sulphuric 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 sulphuric acid (5.4) in the reference cell.

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

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

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 sulphuric acid (5.4) and 7,5 ml of the hydrogen peroxide solution (5.6). Make up to the mark with the diluted sulphuric 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 sulphuric acid (5.4).

If the test solution is clear and colourless, use the diluted sulphuric 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 sulphuric 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 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 iTeH STANDARD PREVIEW

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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 sulphuric 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 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 sulphuric 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 formula:

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