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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

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

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 (H_2SO_4), 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 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 (HCI)**, solution 6 mol/l.

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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.