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Determination of titanium dioxide

1. Scope

1.1 This method describes a procedure for the volumetric quantitative determination of titanium dioxide (TiO_2) in paper and raw materials such as pigments, fillers, and minerals, as well as in materials in various stages of process such as white water, modified or unmodified coating colors, inks, etc. It is suitable for all levels of TiO_2 content.

1.2 TAPPI T 421 “Qualitative (Including Optical Microscopic) Analysis of Mineral Filler and Mineral Coating of Paper” describes a qualitative procedure for determining the presence or absence of titanium in paper.

2. Summary

The titanium, in a dilute sulfuric acid solution, is reduced with aluminum (1) and determined volumetrically by titrating with standardized ferric ammonium sulfate solution.

3. Significance

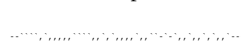
3.1 The accurate determination of titanium dioxide is essential to economical paper mill operation, especially when it is used as a filler material.

3.2 Results obtained by this method will include chromium, antimony, and any other substance which is reduced by aluminum and subsequently oxidized by ferric ions. However, appreciable quantities of interfering materials are usually not likely to be encountered.

4. Apparatus

4.1 *Reduction apparatus*, shown in Fig. 1. It comprises a stoppered 500 mL Erlenmeyer flask with a 5-mm delivery tube leading to a 250 mL beaker and a glass rod extending downward from the stopper to hold a piece of aluminum foil.

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- 4.2 *Ashing equipment:* a silica, or preferably a platinum, dish and a muffle furnace at $925 \pm 25^\circ\text{C}$.
 4.3 *Vycor beaker,* 250 mL.
 4.4 *Zirconium crucible,* 30 mL.

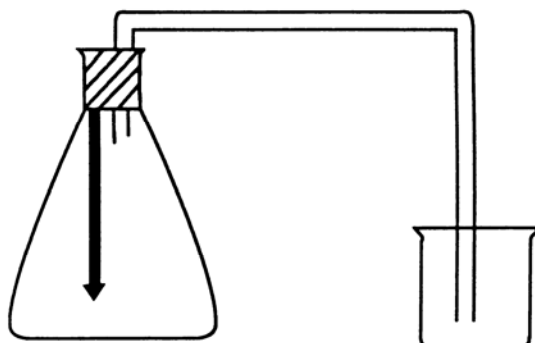


Fig. 1. Apparatus for aluminum reduction.

5. Reagents

- 5.1 *Dissolving solution*
 5.1.1 *Ammonium sulfate,* $(\text{NH}_4)_2\text{SO}_4$.
 5.1.2 *Sulfuric acid,* concentrated H_2SO_4 .
 5.1.3 Heat together 100 g of ammonium sulfate and 180 mL of concentrated sulfuric acid until the salt is dissolved and light fumes appear. Cool and store in a glass stoppered bottle.
 5.2 *Hydrochloric acid,* concentrated HCl .
 5.3 *Aluminum metal foil,* electrolytic grade, preferably about 0.2 mm (0.008 in.) thick.
 5.4 *Standard titanium dioxide,* obtainable from the National Institute of Standards and Technology (NIST), with analysis.
 5.5 *Hydrogen peroxide,* reagent grade.
 5.6 *Hydrogen peroxide,* 3% H_2O_2 . Dilute 10 mL of 30% H_2O_2 to 100 mL with distilled water. This solution is sufficiently stable for 5 days.
 5.7 *Standardized ferric ammonium sulfate solution.* Dissolve 30.16 g of fresh $\text{FeNH}_4(\text{SO}_4)_2 \cdot 12 \text{H}_2\text{O}$ in 800 mL of distilled water containing 15 mL of concentrated H_2SO_4 . Add 10 mL 3% H_2O_2 and boil until O_2 is no longer evolved; dilute to exactly 1000 mL and mix well. Filter if cloudy. Standardize against the standard TiO_2 solution using 0.1900 to 0.2100 g of TiO_2 and following the same procedure as for paper ash or pigments. Calculate the solution factor as follows:

$$\text{Factor} = \frac{\text{g NIST TiO}_2 \times \% \text{TiO}_2 \text{ in NBS NIST TiO}_2}{\text{mL of titrant} \times 100}$$

This factor should be about 0.005 g of TiO_2 per mL of the solution.

- 5.8 *Ammonium thiocyanate indicator.* Dissolve 24.5 g of NH_4SCN in 80 mL of hot distilled water, filter, bring to room temperature, and make up to 100 mL in a graduated cylinder with distilled water. Keep in a well-stoppered, dark-colored bottle.
 5.9 *Sodium bicarbonate solution.* Prepare a saturated solution with distilled water at the time of analysis. About 17 g of NaHCO_3 added to 150 mL of water will be required (may be replaced with water if the carbon dioxide cylinder is used).
 5.10 *Potassium pyrosulfate,* $\text{K}_2\text{S}_2\text{O}_7$, reagent grade (for fusing mineral specimens).
 5.11 *Carbon dioxide (optional),* cylinder of compressed CO_2 .