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An American National Standard

# Standard Test Methods of Qualitative Examination of Mineral Filler and Mineral Coating of Paper[1]

This standard is issued under the fixed designation D686; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 These test methods cover two procedures for the qualitative determination and identification of the mineral constituents of filled and coated papers.

1.2 Due to the similarity in chemical composition and physical size and shape of some of the various possible constituents contained in a given paper specimen, more precise, quantitative methods may at times be required for positive identification.

1.3 It is recommended that one become thoroughly familiar with these test methods by analyzing paper samples of known mineral component content.

1.4 The test methods appear as follows:

| | Sections |
|---|---|
| Method A—Qualitative Chemical Analysis | 4 to 11 |
| Method B—Microscopical Identification | 12 to 19 |

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

NOTE 1—These test methods are technically equivalent to TAPPI T 421.

## 2. Referenced Documents

2.1 *ASTM Standards:*[2]

D585 Practice for Sampling and Accepting a Single Lot of Paper, Paperboard, Fiberboard, and Related Product

D586 Test Method for Ash in Pulp, Paper, and Paper Products[3] [3]

D921 Discontinued 1980; Method of Test for Titanium Dioxide in Paper[3]

D1030 Test Method for Fiber Analysis of Paper and Paperboard

2.2 *TAPPI Standards:*[4]

T 401 Fiber analysis of paper and paperboard

T 438 Zinc and cadmium in paper and pigments

T 421 Qualitative (Including Optical Microscopic) Analysis of Mineral Filler and Mineral Coating of Paper

## 3. Significance and Use

3.1 Qualitative chemical analyses of the mineral component of a paper specimen, Test Method A, serve to identify the ions of any such minerals. The results may then be interpreted in terms of the minerals themselves. Direct identification of some of these minerals or their ions is frequently possible using optical microscopical examination, Test Method B. For additional information, see Annex A1.

3.2 The analysis can be considerably simplified if it is desired only to establish the presence or absence of a particular filler.

3.3 A microscopical examination of the ash usually proves to be a useful adjunct to chemical analysis, and if possible should be attempted (see Sections 12-18).

## Test Method A—Qualitative Chemical Analysis

## 4. Apparatus

4.1 *Crucible*, platinum, with lid, for use in 9.7.1 and in ashing the sample that is being examined. Porcelain or silica crucibles may be used if their weight does not change under the ignition conditions.

4.2 *Muffle Furnace*, electric, controlled to maintain a temperature of 525 ± 25°C.

4.3 *Laboratory Oven*, electric, controlled to maintain a temperature of 150 ± 3°C.

4.4 *Blowpipe*.

4.5 *Wire Loop*, platinum.

[1] These test methods are under the jurisdiction of ASTM Committee D06 on Paper and Paper Products and are the direct responsibility of Subcommittee D06.92 on Standard Documents Relating to Paper and Paper Products.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Withdrawn.

[4] Available from Technical Association of the Pulp and Paper Industry (TAPPI), 15 Technology Parkway South, Norcross, GA 30092, http://www.tappi.org.

4.6 *Spot Plate*, black, glazed.

4.7 *Other Apparatus*—Beakers, 250-mL; watch glass; volumetric flasks, 100-mL; filter funnels and fairly rapid, low-ash filter paper,[5] and Bunsen burner.

## 5. Reagents

5.1 *Acetic Acid, (Glacial, 99.7 % $CH_3$-COOH, sp gr 1.05)*, approximately 1 *N* solution. Add approximately 11.5 mL glacial acetic acid to 50 mL water in a volumetric flask and dilute to 100-mL mark.

5.2 *Ammonium Chloride Solution* (*$NH_4OH$, 10 %*).

5.3 *Ammonium Hydroxide (sp gr 0.90)*—Concentrated ammonium hydroxide ($NH_4OH$).

5.4 *Ammonium Oxalate Solution* (*$(NH_4)_2$-$C_2O_4H_2O$, 3.5 %*).

5.5 *Ammonium Sulfate*, $(NH_4)_2SO_4$.

5.6 *Barium Chloride Solution* (*$BaCl_2$, 10 %*).

5.7 *Charcoal Black*.

5.8 *Cobalt Nitrate Solution*—Dissolve 8 g of cobalt nitrate ($Co(NO_3)_2 6H_2O$) in 100 mL of water.

5.9 *Diphenylthiocarbazone (Dithizone) Solution*—Dissolve 10 mg dithizone in 100 mL carbon tetrachloride, ($CCl_4$).

5.10 *Hydrochloric Acid (2 N, sp gr 1.19)*—Concentrated hydrochloric acid (HCl). Add 15 mL of concentrated HCl to approximately 75 mL water in a 100-mL volumetric flask, cool, and dilute to 100-mL mark.

5.11 *Hydrogen Peroxide (30 % $H_2O_2$)*, or a solution of 3 % $H_2O_2$ used in proportionately greater quantities. Extreme caution should be used when handling 30 % $H_2O_2$ solution as it is very active when in contact with skin. Eye protection should be worn.

5.12 *Iodine Solution* (*0.1 N*).

5.13 *Lead Acetate Paper*—Immerse strips of filter paper in a saturated solution of lead acetate ($Pb(C_2H_3O_2)_2\ 3H_2O$); withdraw from solution and allow to air dry.

5.14 *Lime Water*, saturated solution. Dissolve about 0.2 g of calcium hydroxide ($Ca(OH)_2$) in 100 mL of water and filter.

5.15 *Magnesium Reagent*—Dissolve 0.5 g of para-nitrobenzeneazoresorcinol in 100 mL of sodium hydroxide (NaOH) solution (1 %).

5.16 *Microcosmic Salt Solution*—Dissolve 5 g of sodium ammonium phosphate ($NaNH_4HPO_4\ 4H_2O$) in water and dilute to 100 mL.

5.17 *Morin (3,5,7,2′,4′-pentahydroxyflavanone)*—Saturated solution of morin in methyl alcohol.

5.18 *Potassium Dichromate Solution* (*$K_2Cr_2O_7$, 4 %*).

5.19 *Potassium Ferrocyanide Solution*—Dissolve 15 g of ($K_4Fe(CN)_6\ 3H_2O$) in 1000 mL of water.

5.20 *Potassium Hydroxide Solution (2 N)*—Dissolve 11.2 g of potassium hydroxide (KOH) in 75 mL water; cool and dilute to 100 mL.

5.21 *Sodium Carbonate*—Powdered sodium carbonate ($Na_2CO_3$).

5.22 *Sodium Hydroxide Solution (2 N)*. Dissolve 8 g (NaOH) in 75 mL water; cool and dilute to 100 mL.

5.23 *Sulfuric Acid (5 %, sp gr 1.84)*—Concentrated sulfuric acid ($H_2SO_4$). Add 3 mL of concentrated $H_2SO_4$ to 75 mL of water, cool, dilute to 100 mL.

## 6. Purity of Reagents

6.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[6] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

## 7. Sampling

7.1 Obtain a sample of the paper to be tested in accordance with Practice D585.

## 8. Test Specimens

8.1 From each test unit, for each complete determination, cut test specimens of sufficient size to yield at least 0.15 g of ash.

8.2 An additional specimen of each test unit should be available for testing without previous ashing.

## 9. Procedure

9.1 An outline scheme of the qualitative procedure is given in Fig. 1.

9.2 *Sulfite, Sulfide, and Carbonate (Unignited Coating or Paper Sample)*:

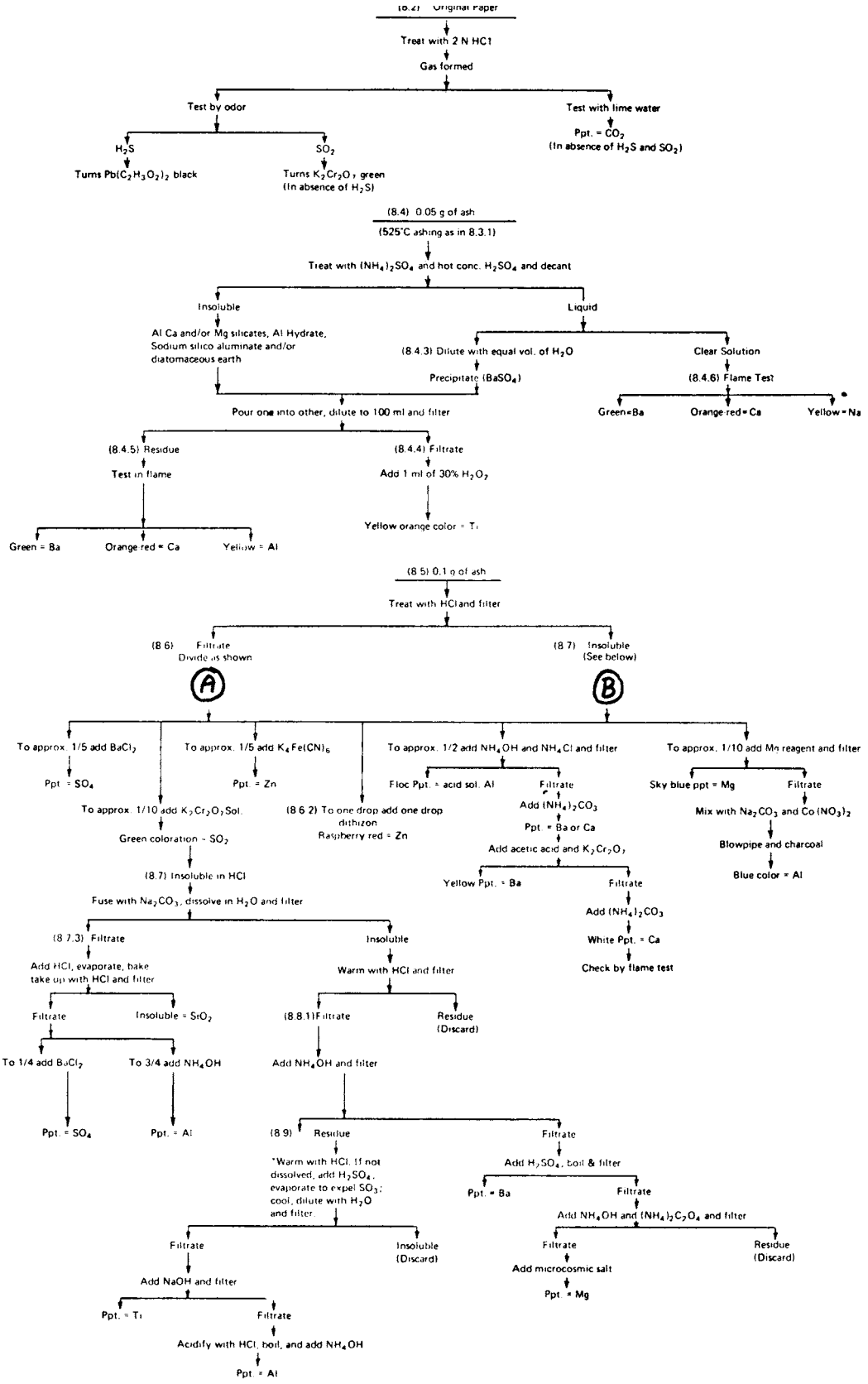
9.2.1 Treat a portion of the unignited coating or paper sample in a small beaker or test tube with 2 *N* HCl. Note whether effervescence takes place and the odor of any escaping gas. Liberation of $SO_2$ and $H_2S$ indicates the presence of sulfites and sulfides, respectively. Warm the contents of the beaker and test the vapor with moistened lead acetate paper. The development of a metallic gray or black color confirms the presence of sulfide. In the absence of sulfides, add either a small crystal of potassium dichromate or a few drops of a 4 % dichromate solution to a small portion of the HCl solution of the sample. A green coloration indicates the presence of a reducing agent, in this case probably a sulfite.

NOTE 2—Mixtures of sulfites and sulfides are not known to be used in loading or coating paper.

9.2.2 If sulfites and sulfides are absent, effervescence alone is a good indication of the presence of a carbonate, which may be confirmed by holding a glass rod with a drop of saturated lime water just above the solution. Cloudiness (milky) appearance of the supported drop indicates the presence of $CO_2$. This precipitate may later dissolve. A confirmatory test of $CO_2$ in the presence of sulfites is to oxidize the sulfites to sulfates by

[5] Whatman No. 40, available from A. H. Thomas Co., P. O. Box 779, Philadelphia, PA 19105, or its equivalent, has been found satisfactory for these test methods.

[6] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals,* BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary,* U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.




*This procedure is necessary only if both Ti and HCl insoluble Al compounds are present and it is necessary to estimate relative amounts of Ti and Al.

**FIG. 1 Qualitative Analysis of Mineral Filler and Mineral Coating of Paper**