WI 210114.01

T 665

BALLOT NO. 03 SARG

DRAFT NO. 01

DATE October 26, 2023

WORKING GROUP CHAIR N/A

SUBJECT CATEGORY Coating & Graphic Arts

RELATED METHODS See "Additional Information"

CAUTION:
This Test Method may include safety precautions which are believed to be appropriate at the time of publication of the method. The intent of these is to alert the user of the method to safety issues related to such use. The user is responsible for determining that the safety precautions are complete and are appropriate to their use of the method, and for ensuring that suitable safety practices have not changed since publication of the method. This method may require the use, disposal, or both, of chemicals which may present serious health hazards to humans. Procedures for the handling of such substances are set forth on Safety Data Sheets which must be developed by all manufacturers and importers of potentially hazardous chemicals and maintained by all distributors of potentially hazardous chemicals. Prior to the use of this method, the user must determine whether any of the chemicals to be used or disposed of are potentially hazardous and, if so, must follow strictly the procedures specified by both the manufacturer, as well as local, state, and federal authorities for safe use and disposal of these chemicals.

Analysis of Talc
*(*Ten-year review of T 665 cm-12: Reconfirmation of T 655 Draft 1*)*

1. Scope

This method covers the identification and the chemical analysis of talc.

2. Summary

Procedures are given for the following: identification by X-ray diffraction, loss on ignition, silicon dioxide, R_2O_3 group, calcium oxide, and magnesium oxide.

3. Significance

3.1 Pure talc is a hydrous magnesium silicate, $Mg_3Si_4O_{10}(OH)_2$, with a theoretical chemical composition of 31.7% MgO, 63.5% SiO_2, and 4.8% H_2O. Unground it may be white, greenish, gray, or almost black, depending on the impurities present, but when ground, talc is normally white to grayish white.

3.2 Commercial talc may contain at least one of the following impurities: quartz, tremolite, kaolinite, serpentine, chlorite, muscovite, calcite, dolomite, and magnesite.

4. Apparatus, reagents, and materials

4.1 Any specialized apparatus, materials, and necessary reagents are included in the appropriate sections of the procedure.

4.2 All chemicals should be of reagent grade or better.

4.3 Water used in the procedure should be distilled or deionized.

4.4 A complete reagent blank run along with the sample is recommended, if the reagents are of suspect purity.

4.5 All weighings are to be performed on an analytical balance and recorded to four decimal places.

5. Sampling and test specimen

From each test unit of at least 400 g obtained in accordance with TAPPI T 400 “Sampling and Accepting a Single Lot of Paper, Paperboard, Fiberboard, or Related Product,” grind approximately a 10-g specimen to -325 mesh (<44 μm) for X-ray diffraction.

6. Procedures

6.1 *Identification by X-ray diffraction*. X-ray diffraction is the recommended technique for identification. Infrared spectroscopy, thermal analysis, and specialized microscopy techniques are also satisfactory. Conclusions based on chemical analysis alone should be made with caution.

6.1.1 *Apparatus:* standard X-ray diffraction system.

6.1.2 Prepare the specimen of the ground talc as described by Klug and Alexander (*1*).

6.1.3 Obtain the diffraction pattern (*1*) of the specimen and the software generated d-spacings.

6.1.4 For identification purposes, Table 1 contains a listing of ICDD, International Centre for Diffraction Data, card citations as the primary card files to access talc and the common mineral contaminants mentioned in 3.2. These citations will provide the complete list of d-spacings for comparison to the specimen’s d-spacings.

Table 1. X-ray diffraction data for identification (*2*)

Mineral	ICDD card citations		
Talc	13-558	19-770	29-1493
Chlorite	24-506	12-242	
Quartz	33-1161	46-1045	
Calcite	5-586	47-1743	
Dolomite	36-426		
Magnesite	8-479		

6.2 *Loss on ignition*

6.2.1 Accurately weigh approximately 1 g of the sample that has been dried for 1h at 105°C into a tared crucible. Ignite in a muffle furnace to constant weight starting at 400°C and gradually increasing to 1000°C. One hour at full heat is sufficient.

6.2.2 Allow the crucible and its contents to cool to ambient temperature, cool in a desiccator, and weigh.

6.2.3 Calculation:

$$\text{Loss on ignition, \%} = \frac{\text{Weight loss, g} \times 100\%}{\text{Sample weight, g}}$$

NOTE 1: EN ISO 12677:2011 is an alternate test method for % SiO_2, % CaO, MgO, and % R_2O_3.

6.3 *Silicon dioxide*

6.3.1 *Materials*:

6.3.1.1 *Balance*, 500-g capacity, accurate to 0.05 g.

6.3.1.2 Balance, analytical, accurate to 0.0005 g.

6.3.1.3 *Drying oven*, 105 ± 2°C.

6.3.1.4 *Platinum crucible with close fitting platinum lid*, capable of holding 1 g.

6.3.1.5 *Muffle furnace*, capable of maintaining 1000 ± 30°C.

6.3.1.6 *Desiccator*.

6.3.1.7 *Porcelain casserole*, capable of holding 600 mL.

6.3.1.8 *Watch glass,* sized to cover casserole.

6.3.1.9 *Hot-plate-type dryer*, sized to accommodate a 600-mL porcelain casserole.

6.3.1.10 *Platinum-tipped tongs.*

6.3.1.11 *Steam bath*.

6.3.1.12 *Filter paper*, low-ash, fine porosity, 150 mm in diameter.

6.3.2 *Reagents*

6.3.2.1 *Sodium carbonate* (Na_2CO_3), powder.

6.3.2.2 *Hydrochloric acid* (HCl), concentrated (sp. gr. 1.19).

6.3.2.3 *Hydrochloric acid*, 1 + 20 (one part of HCl with sp. gr. of 1.19 and 20 parts water).

6.3.2.4 *Sulfuric acid*, 1 + 1 (one part of concentrated H_2SO_4 and 1 part water).

6.3.2.5 *Hydrofluoric acid* (HF), 48%.

6.3.3 Accurately weigh approximately 0.5 g of the sample into a platinum crucible, mix 5 g of Na_2CO_3 into the same crucible, and cover with a close fitting platinum lid. Heat in a muffle furnace starting at 500°C for 10 min and gradually increase the temperature to 1100°C to obtain a clear, quiet fusion. Generally 60 min of strong heating is sufficient.

6.3.4 Allow the crucible and melt to cool in a desiccator to ambient temperature. Place the entire crucible and lid into a 600-mL porcelain casserole containing 200 mL of water. Boil until the melt disintegrates.

NOTE 2: Muffle furnace temperature may need to be reduced to approximately 550°C before removing crucible to prevent cracking- the plate in the desiccator.

6.3.5 Remove the crucible and lid with platinum-tipped tongs and carefully rinse out any adhering particles into the same casserole. Cover the casserole with a watch glass and carefully acidify the contents by slowly pouring concentrated HCl in the spout and down the sides of the casserole while maintaining a slow swirling motion. Take care to avoid losses due to spattering caused by the evolved CO_2.

6.3.6 Add 30 mL of concentrated HCl in excess and evaporate to dryness on a steam bath. When dry and no odor of HCl can be detected, wash down sides of casserole with water, add 150 mL of water, and bring to a slow boil. Filter while hot through low-ash, fine-porosity filter paper. Wash filter paper at least five times with hot 1 + 20 HCl followed by hot water washing until the filtrate is acid free.

6.3.7 Re-evaporate the filtrate; when dry, dilute to 150 mL as before and filter in the same paper. Wash well until acid free. Retain the filtrate for determination of R_2O_3 group.

6.3.8 Transfer the filter paper and its contents to a platinum crucible, char and burn the filter paper gently to avoid losses, and ignite the residue at 850°C for l h. Cool in a desiccator and quickly weigh (W_1).

6.3.9 Moisten the residue with 1 + 1 H_2SO_4, fill the crucible half way with 48% HF, and evaporate to dryness on a steam bath.

6.3.10 Repeat 6.3.9. When the residue is dry, ignite at 850°C for l h, cool in a desiccator, and quickly weigh (W_2).

NOTE 3: To be absolutely certain that all of the silica has been volatized, repeat 6.3.9 and 6.3.10 until a constant weight is attained.

6.3.11 Calculation:

$$SiO_2, \% = \frac{W_1 - W_2 \times 100\%}{\text{Sample weight, g}}$$

6.4 *Ammonium hydroxide grou*p (R_2O_3)

6.4.1 *Materials* (in addition to 6.3.1):

6.4.1.1 *Filter paper*, low-ash, medium porosity, 150 mm in diameter.

6.4.2 *Reagents*

6.4.2.1 *Potassium pyrosulfate*, $K_2S_2O_7$.

6.4.2.2 *Hydrogen peroxide*, H_2O_2, 2% solution.

6.4.2.3 *Methyl red indicator solution.*

6.4.2.4 *Ammonium hydroxide*, NH_4OH, concentrated (sp. gr. 0.90).

6.4.2.5 *Ammonium chloride*, NH_4Cl, 2% solution.

6.4.3 If any residue remains in the crucible from the silicon dioxide determination, fuse with a small amount of $K_2S_2O_7$, leach the fusion from the crucible with water, and combine with the filtrate from 6.3.7.

6.4.4 Add 5 mL of 2% H_2O_2 to the filtrate, bring to a boil and maintain boiling for 10 min. Remove from heat, neutralize while hot with concentrated NH_4OH to a methyl red end point, and add 2-3 drops in excess. Continue heating on a low plate until precipitate coagulates.

6.4.5 Filter the solution while hot through low-ash, medium-porosity filter paper and wash at least four times with hot 2% NH_4Cl. If increased accuracy is desired, the precipitate may be redissolved in hot 1 + 3 HCl and reprecipitated as described in 6.4.3.

6.4.6 Transfer the filter paper and its contents to a tared crucible, dry and char the paper, and ignite carefully to avoid reduction to a constant weight at 1000°C. Cool in a desiccator and weigh the residue as R_2O_3.

6.4.7 Calculation:

$$R_2O_3, \% = \frac{\text{Residue weight, g} \times 100}{\text{Sample weight, g}}$$

6.5 *Calcium oxide*

6.5.1 *Materials* (in addition to 6.3.1 and 6.4.1):

6.5.1.1 *Volumetric flasks*, 500 mL.

6.5.1.2 *Pipette*, 50 mL.

6.5.2 *Reagents*

6.5.2.1 *Potassium hydroxide*, KOH, 20% solution.

6.5.2.2 *Ascorbic acid, powder.*

6.5.2.3 *Hydroxynaphthol blue.*

6.5.2.4 *0.005M disodium ethylene dinitrilotetraacetic acid dihydrate* (EDTA), standard volumetric solution, standardize vs. primary standard grade calcium carbonate ($CaCO_3$).

6.5.3 Repeat 6.3.3 through 6.3.6, catching the filtrate in a 500-mL volumetric flask. Dilute to the mark with water and mix. Use for calcium oxide and magnesium oxide (6.6) determinations.

6.5.4 Remove a 50-mL aliquot, dilute to 300 mL, and adjust the pH to 12.0-12.5 with potassium hydroxide solution.

6.5.5 Add 50-100 mg of ascorbic acid, 25-50 mg of hydroxynaphthol blue indicator, and titrate with 0.005*M* EDTA to a deep blue end point.

6.5.6 Calculation:

$$\text{CaO, \%} = \frac{\text{Volume 0.05}M\text{ EDTA, mL} \times \text{CaO titer, g/mL} \times 500\text{ mL} \times 100\%}{\text{Sample weight, g} \times 50\text{ mL}}$$

6.6 *Magnesium oxide*

6.6.1 *Reagents*

6.6.1.1 *Ammonia buffer*, dissolve 66 g NH_4Cl in 300 mL water, add 560 mL NH_4OH (sp. gr. 0.90), and dilute to 1000 mL.

6.6.1.2 *Calmagite indicator.*

6.6.1.3 *0.05M EDTA*, standardize vs. primary standard grade calcium carbonate ($CaCO_3$).

6.6.2 Remove a 50-mL aliquot from the solution prepared in 6.5.2, dilute to 300 mL and adjust the pH to 10.0-10.5 with ammonia buffer.

6.6.3 Add exactly the amount of 0.005*M* EDTA equivalent to the calcium oxide as determined in 6.5.4.

6.6.4 Using calmagite indicator, titrate with 0.05*M* EDTA to a deep blue end point.

6.6.5 Calculation

$$\text{MgO, \%} = \frac{\text{Volume 0.05}M\text{ EDTA, mL} \times \text{MgO titer, g/mL} \times 500\text{ mL} \times 100\%}{\text{Sample weight, g} \times 50\text{ mL}}$$

7. Report

7.1 *Identification by X-ray diffraction:* report the presence of all minerals identified by name. If the presence of an unidentified phase(s) is suspected, this should be included in the report.

7.2 Report all chemical analysis results in percent, based on the “as received” weight of the talc, to a maximum of three significant figures.

8. Precision

Repeatability:

	Relative	Absolute
Loss on ignition	± 5%	0.3%
Silicon dioxide	± 5%	1.9%
R_2O_3 group	±15%	0.3%
Calcium oxide	± 5%	0.1%
Magnesium oxide	± 5%	1.7%

NOTE 4: Use whichever value is greater. Values were obtained using a talc sample of the following composition: loss on ignition = 5.4%; silicon dioxide = 60.4%; R_2O_3 group = 2.3%; calcium oxide = 0.5%; magnesium oxide = 31.4%.

9. Keywords

Talc, X-ray diffraction, Loss on ignition, Magnesium silicate, Silica, Ammonium hydroxide, Calcium oxide, Magnesium oxide

10. Additional information

10.1 Effective date of issue: To be assigned.

10.2 The 2002 edition included a statement of significance, identification by X-ray diffraction, and references. The procedures themselves were updated and a precision statement added. Tests for pH, brightness, free moisture, and screen residue were deleted as they are now separate TAPPI methods. The test for alum demand was deleted. In the 2012 edition, revisions included a better description of the igniting procedure, muffle furnace details, specific apparatus listing, and several editorial changes, along with updating to ICDD which has replaced JCPDS.

10.3 Related methods: TAPPI T 667 “pH of Fillers and Pigment Slurries,” TAPPI T 646 “Brightness of Clay and Other Mineral Pigments (0°-45° Directional),” TAPPI T 534 “Diffuse Blue Reflectance Factor of Clay and Mineral Pigment,” TAPPI T 657 “Sampling of Fillers and Pigments,” TAPPI T 671 “Free Moisture in Fillers and Pigments,” TAPPI T 681 “Screen Residue of Paper Coating Clays and Related Pigments (High-Speed Mixer Method),” and ASTM D-717 “Analysis of Magnesium Silicate Pigments.”

Literature cited

1. Klug and Alexander, “X-ray Diffraction Procedures for Polycrystalline and Amorphous Materials,” John Wiley and Sons, New York, 1974, Second Edition, pp. 372-373.
2. Fawcett, T., “Effective Use of the Powder Diffraction File.” circa 2000.

Your comments and suggestions on this procedure are earnestly requested and should be sent to the TAPPI Standards Department. ■