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ANALYSIS AND CHARACTERIZATION OF HIGH-PURITY TALC FOR USE IN PROPELLANTS; DETERMINATION OF TALC IN PROPELLANTS

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Technical Support Directorate

U.S. ARMY ARMAMENT COMMAND
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Methods are described for the analysis and characterization of high-purity talc for use in nitrocellulose-base propellants. Procedures are described for silica, magnesium oxide, R_2O_3, ferric oxide, aluminum oxide, calcium oxide, zinc, sulfate, phosphorus pentoxide, carbon dioxide, moisture, loss on ignition, water-soluble matter, acid-soluble matter, sieve analysis, average particle size, apparent density, and pH. In the method

(continued)

20. ABSTRACT: (continued)

for silica and magnesium oxide, the sample is fused with sodium carbonate and the melt is dissolved in such a manner with 5 to 1 perchloric acid that the melt floats free of the crucible (in previous schemes for the analysis of talc there are frequently mechanical losses due to the difficulty of removing the magnesium silicate melt from the crucible). The solution is then evaporated to fumes of perchloric acid and the silica is filtered and ignited. The magnesium oxide is determined in the filtrate by precipitation as magnesium ammonium phosphate and a correction is made for the calcium oxide which is precipitated with the magnesium ammonium phosphate. R_2O_3 is determined after treatment with nitric and hydrofluoric acids and evaporation to fumes of perchloric acid. Iron oxide, aluminum oxide, calcium oxide, and zinc are determined by atomic absorption, after treatment with nitric and hydrofluoric acids and evaporation to fumes of perchloric acid. Sulfate and phosphorus pentoxide are determined by the precipitation as barium sulfate and ammonium phosphomolybdate, respectively, after treatment with nitric and hydrofluoric acids and evaporation to fumes of perchloric acid. Carbon dioxide is determined by combustion in an induction furnace with iron as a flux. The methods for moisture, loss in ignition, water-soluble matter, acid-soluble matter, sieve analysis, average particle size, apparent density, and pH are more or less conventional.

Three methods are described for the determination of talc in nitrocellulose-base propellants. The first method (which is the method recommended for the usual nitrocellulose-base propellants) is based on the silica content of the propellant, while the second method is based on the magnesium oxide content. In the third method, the talc is weighed as such, after dissolution of the propellant in morpholine, filtration, washing of the residue with water and dilute hydrochloric acid, and ignition to eliminate the graphite.

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INTRODUCTION

Powdered talc has many uses in explosives. It is used in nitrocellulose-base propellants to reduce gun erosion.[1,2*] Also, it is used in ammonium nitrate explosives for increasing detonation sensitivity and decreasing shock sensitivity[3*] and in ammonium nitrate prills to improve storage properties (by reducing break-up due to the phase changes of the ammonium nitrate).[4*] It is used in the manufacture of detonators to lower friction while filling.[5*] The US Army uses technical grade talc to simulate battle conditions in the training of troops.[6*] The biggest use of high quality talc is in toiletries.

Talc is a naturally occurring hydrated magnesium silicate with the approximate formula $3MgO \cdot 4SiO_2 \cdot H_2O$ (or $Mg_3Si_4O_{10}(OH)_2$) (equivalent to 63.4% SiO_2, 31.9% MgO, and 4.7% H_2O).[7,8*] It is not to be confused with artificially produced magnesium silicate which has the approximate formula $3MgSiO_3 \cdot 5H_2O$.[7*] Talc is very difficult to identify microscopically and positive identification is best made by a complete chemical analysis.[8*] There is no government specification for talc of the high purity that is used in propellants, although there is a specification for technical grade talc.[6*] This arsenal, therefore, undertook an investigation to establish quality assurance requirements for high-purity talc for use in propellants.

Several schemes have been proposed for the analysis of talc. ASTM[9*] recommends the classical scheme for determining silica, R_2O_3, calcium oxide, and magnesium oxide, whereby the sample is fused with sodium carbonate, the melt dissolved in water, and the silica dehydrated by evaporation to dryness with hydrochloric acid. The silica is ignited and treated with hydrofluoric acid in the usual manner, the residue is fused with potassium bisulfate, and the melt is combined with the silica filtrate. An R_2O_3 precipitation is then made and the calcium oxide is determined by the gravimetric oxalate method and the magnesium oxide by the phosphate method. ASTM[9*] also describes methods for the determination of moisture, loss in ignition, water-soluble matter, pH, and sieve analysis. The American Ceramic Society[10*] determines silica, R_2O_3, calcium oxide, and magnesium oxide somewhat similarly to ASTM but the precipitated calcium oxalate is titrated with permanganate; also, the R_2O_3 precipitate is treated with hydrofluoric acid to eliminate the silica and the result for "carried silica" is added to the total silica. The American Ceramic Society[10*] also describes methods for alkali metals, moisture, loss on ignition, acid-soluble matter, pH, and sieve analysis. The government specification for technical grade talc[6*] describes methods for silica and magnesium oxide similar to the ASTM methods

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* See References, page 18.

(the only chemical or physical requirements in this specification are for silica content, magnesium oxide content, and sieve analysis). TAPPI[8] determines silica, R_2O_3, magnesium oxide, and calcium oxide by fusion with sodium carbonate, dissolution of the melt in hydrochloric acid, addition of gelatin to coagulate the silica (without prior evaporation to dryness), precipitation of the R_2O_3, and determination of calcium oxide and magnesium oxide in the R_2O_3 filtrate by EDTA titrations. TAPPI[8] also describes methods for moisture, loss in ignition, pH, and sieve analysis. Kysela[11] determined silica, R_2O_3, magnesium oxide, and calcium oxide by fusion with a mixture of sodium carbonate, potassium carbonate and sodium borate, dissolution of the melt in hydrochloric acid, evaporation to dryness, addition of glycerol to coagulate the silica, precipitation of the R_2O_3 in the silica filtrate, and determination of the calcium oxide and magnesium oxide by EDTA titrations. Yurist and Korotkova[12] determined silica, magnesium oxide, calcium oxide, iron oxide, and aluminum oxide in talc by fusing with potassium bisulfate, dissolution of the melt in dilute sulfuric acid, filtration of the silica, and determination of the magnesium calcium, iron, and aluminum in the filtrate by a rather intricate system of EDTA titrations. Ohtsuki, Nakagawa, and Maeda[13] determined magnesium oxide, calcium oxide, and iron oxide in talc by atomic absorption, after treatment with hydrofluoric and sulfuric acids and evaporation to dryness.

A disadvantage of methods that involve an initial fusion is that considerable difficulty is encountered in bringing the melt into solution after the fusion. This trouble stems from the fact that the magnesium silicate makes the melt difficultly soluble in water, hydrochloric acid or sulfuric acid. Frequently, the melt can be removed from the crucible only by prolonged boiling with water or dilute acid, but when this technique is used the crucible becomes coated with a slimy mass of salts that adheres tenaciously and causes significant mechanical losses. Another disadvantage of the common methods is that complete recovery of silica cannot be obtained by one evaporation with hydrochloric acid.

In the present report an improved scheme is proposed for the analysis of talc. The silica and magnesium oxide are determined by fusion with sodium carbonate, dissolution of the melt in such a manner with 5 to 1 perchloric acid that the melt floats out of the crucible. The solution is then evaporated to fumes of perchloric acid and the silica is filtered and ignited. The magnesium oxide is determined in the filtrate by precipitation as magnesium ammonium phosphate and a correction is

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[8] Technical Assoc. of Paper Products Institute (TAPPI), Method T665 su-65.

[11] A. Kysela, Rev. Chim. (Bucharest) 17, (2), 109 (1966).

[12] I. M. Yurist and O. I. Korotkova, Zavodskaya Lab., 27, 274 (1961).

[13] M. Ohtsuki, M. Nakagawa, and T. Maeda, Bunseki Kagaku, 19, 1191 (1970).

made for calcium. R_2O_3 is determined after treatment with nitric and hydrofluoric acids and evaporation to fumes of perchloric acid. Ferric oxide, aluminum oxide, calcium oxide, and zinc are determined by atomic absorption after treatment with nitric and hydrofluoric acids, and evaporation to fumes of perchloric acid (the determination of zinc is important because zinc stearate in small amounts is added to some talcs that are used in propellants). Sulfate and phosphorus pentoxide are determined by precipitation as barium sulfate and ammonium phosphomolybdate, respectively, after treatment with nitric and hydrofluoric acids and evaporation to fumes of perchloric acid. Carbon dioxide is determined by combustion in an induction furnace with iron as a flux. The methods for moisture, loss in ignition, water-soluble matter, acid-soluble matter, sieve analysis, average particle size, and pH are more or less conventional.

Also considered in the present report are methods for the determination of talc in nitrocellulose-base propellants. Three methods are proposed. The first method is based on the silicon dioxide content of the propellant, while the second method is based on the magnesium oxide content. In the third method, the talc is weighed as such, after dissolution of the propellant in morpholine, filtration, washing of the residue with water and dilute hydrochloric acid, and ignition (to eliminate graphite).

EXPERIMENTAL

Methods for the Characterization of Talc

Silicon Dioxide and Magnesium Oxide. Place 2 g of sodium carbonate on the bottom of a platinum crucible and weigh. Add about 0.5 g of the talc and weigh to obtain the exact weight of the sample. Mix the sample with the sodium carbonate by means of a small spatula and brush off the spatula. Place 2 g of sodium carbonate on top of the mixture, cover with a platinum lid, heat with a Meker burner at moderate heat for a few minutes, and then heat at highest heat for 30 minutes. Remove the lid and place it in a covered 1-liter beaker. While the crucible is still in the flame, grasp its outside with tongs (do not insert the tongs into the crucible) and tilt in such a manner that the melt reaches to within 1/4 inch of the top of the crucible. While tilting the crucible, remove it from the flame and (while still tilted) dip it intermittently every few seconds into a tray of tap water. Do not allow water to enter the crucible. When the melt has completely solidified, turn the crucible upright and dip it into the water a few more times. Allow the melt to cool thoroughly to room temperature (this is important). Place the crucible into the 1-liter beaker upright.

Pour 75 ml of perchloric acid (5 volumes of reagent 70% perchloric acid mixed with 1 volume of water) onto the crucible and immediately cover the beaker with the watch glass. Wait about 1 minute, tip over the crucible, and swirl the beaker. The melt will float free from the crucible. Wash down and remove the lid and the crucible. Add 20 ml of dilute hydrochloric acid (1 to 2) to the crucible and police the crucible with water, collecting the washings in the 1-liter beaker. Warm the 1-liter beaker on the hot plate for a few minutes and break up the cake with a stirring rod. Evaporate to fumes of perchloric acid and fume at the highest heat of the hot plate for 20 minutes. Allow to cool, add about 200 ml of water, and stir to dissolve the salts. Filter through a Whatman No. 41 filter paper into a 400-ml beaker. Save the filtrate for the determination of magnesium oxide. Transfer the precipitate to the filter paper with water (using a policeman) and wash several times with water. Place the filter paper and precipitate into a platinum crucible, char, and burn off the filter paper while gradually raising the temperature, and then ignite at about 1100°C for 30 minutes. Cool in a desiccator and weigh. Add 5 ml of hydrofluoric acid (48%) and 2 drops of sulfuric acid and evaporate to dryness. Ignite at 900°C for 10 minutes, cool in a desiccator, and weigh. Calculate the percent silica as follows:

$$\% \ SiO_2 = \frac{\text{g loss in weight}}{\text{g of sample}} \times 100$$

Add 5 ml hydrochloric acid to the crucible from the silica determination, heat on the hot plate a few minutes, and wash the solution into the filtrate from the silica determination. Heat the solution at about 90°C, add ammonium hydroxide (1 to 1) until alkaline to methyl red, and add 2 or 3 drops excess ammonium hydroxide (1 to 1). Boil for a minute, adding more ammonium hydroxide to maintain alkalinity if necessary. Filter through a Whatman No. 40 filter paper into a 600-ml beaker and wash with hot ammonium chloride solution (2%). If the residue is very large, redissolve it in hot dilute hydrochloric acid (1 to 3) and reprecipitate the R_2O_3. Add hydrochloric acid until the solution is just acid to methyl red and add 5 drops excess hydrochloric acid. Adjust the solution to about 300 ml. To each solution individually add 50 ml of diammonium hydrogen phosphate solution (25%) and immediately add ammonium hydroxide drop by drop while stirring constantly until precipitation appears complete; then add 35 ml of ammonium hydroxide and continue stirring intermittently for several minutes. Allow to stand 3 or more hours. Filter through a Whatman No. 40 filter paper and transfer and wash with dilute ammonium hydroxide (5 to 95). Dissolve the precipitate into the original beaker with warm dilute hydrochloric acid (1 to 3) and wash with water. Dilute to about 300 ml. To each solution individually add 1 ml of diammonium hydrogen phosphate solution (25%) and immediately add ammonium hydroxide drop by drop while stirring constantly until precipitation appears complete; then add 10 ml ammonium hydroxide and continue stirring intermittently for several minutes. Allow to stand 3 hours or more. Filter through a Whatman No. 40 filter paper and transfer and wash with dilute ammonium hydroxide (5 to 95). Place the filter paper and precipitate into a tared platinum crucible. Char and burn off the filter paper slowly while gradually raising the temperature and then ignite at 1100°C for 30 minutes. Cool in a desiccator and weigh. Calculate the percent magnesium oxide as follows:

$$\%\ MgO = \frac{0.3621\ \{\text{g of precipitate} - (0.01844 \times \%\ CaO \text{ in sample} \times \text{g of sample})\}}{\text{g of sample}} \times 100$$

<u>R_2O_3</u>. Weigh a 2-g sample into a platinum dish and add 20 ml of nitric acid and 5 ml of hydrofluoric acid. Warm on the hot plate until the sample is dissolved and then evaporate to a volume of about 15 ml. Add 10 ml of perchloric acid and evaporate to strong fumes of perchloric acid. Add 50 ml of water and warm to dissolve the salts. Filter through a Whatman No. 42 filter paper into a 400-ml beaker and wash with water. Discard the filter paper. Dilute to about 200 ml and add 5 ml of hydrochloric acid. Heat to about 90°C, add ammonium hydroxide (1 to 1) until the solution is alkaline to methyl red, and add 3 drops excess ammonium hydroxide (1 to 1). Heat to boiling and boil for about a minute, adding more ammonium hydroxide to maintain alkalinity if necessary. Filter through a Whatman No. 41 filter paper and wash with water. Dissolve the precipitate into the original beaker with hot hydrochloric acid (1 to 3) and wash with water. Dilute to about 200 ml and reprecipitate and filter as before. Transfer the filter paper and precipitate to a tared platinum crucible. Char and burn off the filter paper slowly while gradually raising the temperature and then ignite at 1000°C for 30 minutes. Cool in a desiccator and weigh. Calculate the percent R_2O_3 as follows:

$$\%\ R_2O_3 = \frac{\text{g of precipitate}}{\text{g of sample}} \times 100$$

<u>Iron Oxide, Aluminum Oxide, Calcium Oxide, and Zinc</u>. Weigh a 2-g sample into a platinum dish and add 20 ml of nitric acid and 5 ml of hydrofluoric acid. Warm on the hot plate until the sample is dissolved and then evaporate to a volume of about 15 ml. Add 8 ml of perchloric acid and evaporate to a volume of about 1 or 2 ml. Add 50 ml of water, warm to dissolve the salts, cool, and dilute to 100 ml in a volumetric flask. Dilute appropriate aliquots of standard solutions of iron, calcium, and zinc to 100 ml in volumetric flasks and determine the absorbance of the samples and standards by atomic absorption in accordance with the recommendations of the instrument manufacturer. It may be necessary to dilute the samples further prior to the atomic absorption measurements. Calculate the percent iron oxide, calcium oxide, and zinc on the basis of the standard solutions and then calculate the percent aluminum oxide as follows:

$$\%\ Al_2O_3 = \%\ R_2O_3 - \%\ Fe_2O_3$$

Sulfate. Weigh a 2-g sample into a platinum dish and add 20 ml of nitric acid and 5 ml of hydrofluoric acid. Warm on the hot plate until the sample is dissolved and then evaporate to a volume of almost 15 ml. Add 8 ml of perchloric acid and evaporate to fumes of perchloric acid. Dilute to about 100 ml with water and warm to dissolve the salts. Filter through a Whatman No. 42 filter paper and wash with water. Discard the filter paper. Dilute to about 200 ml with water and add 2 ml of hydrochloric acid. Heat to boiling, add 10 ml of barium chloride solution (10%), and boil for 2 minutes. Allow to stand overnight. Filter through a Whatman No. 42 filter paper and transfer and wash with water. Place the filter paper and precipitate into a tared platinum crucible. Char and burn off the filter paper while slowly increasing the temperature and then ignite at 900°C for 30 minutes. Cool in a desiccator and weigh. Calculate the percent sulfate as follows:

$$\% \ SO_4 = \frac{0.4116 \times \text{g of precipitate}}{\text{g of sample}} \times 100$$

Phosphorus Pentoxide. Weigh a 2-g sample into a platinum dish and add 20 ml of nitric acid and 5 ml of hydrofluoric acid. Warm on the hot plate until the sample is dissolved and then evaporate to a volume of about 15 ml. Add 8 ml of perchloric acid and evaporate to fumes of perchloric acid. Add 25 ml of water and warm to dissolve the salts. Filter through a Whatman No. 42 filter paper into a 300-ml Erlenmeyer flask and wash with water. Discard the filter paper. Dilute to about 50 ml with water and add 5 ml of nitric acid. Add 50 ml of ammonium molybdate reagent[14] at one stroke and swirl immediately. Allow to stand overnight. Filter through a tared sintered glass crucible of fine porosity and transfer and wash with nitric acid solution (1 to 99). Dry at 120°C for 1 hour, cool in a desiccator, and weigh. Calculate the percent phosphorus pentoxide as follows:

$$\% \ P_2O_5 = \frac{0.0380 \times \text{g of precipitate}}{\text{g of sample}} \times 100$$

Carbon Dioxide. Mix 1 g of the sample with 1 g of high purity iron (for example, National Bureau of Standards Sample 55e) and determine the carbon by combustion in an apparatus with an induction furnace. Calculate the percent CO_2 as follows:

$$\% \ CO_2 = 3.66 \times \% \ C$$

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[14] American Soc. for Testing and Materials, Designation E50-73, Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals, Section 54, Reagent No. 102, Philadelphia, PA, 1975.

Moisture. Weigh a 5-g sample into a tared weighing dish (60 mm in diameter and 10 mm deep). Dry at 110°C for 2 hours, cool in a desiccator, and weigh. Calculate the percent moisture as follows:

$$\% \text{ moisture} = \frac{\text{g loss in weight}}{\text{g of sample}} \times 100$$

Loss on Ignition. Transfer 1 g of the dried sample from the moisture determination to a tared platinum crucible, ignite at 900° - 1000°C for 20 minutes, cool in a desiccator, and weigh. Calculate the percent loss on ignition as follows:

$$\% \text{ loss on ignition} = \frac{\text{g loss in weight}}{\text{g of sample}} \times 100$$

Water-Soluble Matter. Weigh a 5-g sample into a 400-ml beaker, add 5 ml of isopropyl alcohol or ethyl alcohol, and stir well. Dilute to about 200 ml with water, heat to boiling, and boil 1 or 2 minutes. Filter through a double Whatman No. 42 filter paper into a tared 250-ml beaker and wash with water. Evaporate nearly to dryness on the hot plate and dry in an oven at 110°C for 1 hour. Cool in a desiccator and weigh. Calculate the percent water-soluble material as follows:

$$\% \text{ water-soluble material} = \frac{\text{g of residue}}{\text{g of sample}} \times 100$$

Acid-Soluble Matter. Weigh a 5-g sample into a 400-ml beaker, add 5 ml of isopropyl alcohol or ethyl alcohol, and stir well. Dilute to about 200 ml with hot dilute hydrochloric acid (5 to 95). Digest at about 95°C for 30 minutes. Filter through a double Whatman No. 42 filter paper into a tared 250-ml beaker and wash with water. Evaporate nearly to dryness on the hot plate and dry in an oven at 110°C for 1 hour. Cool in a desiccator and weigh. Calculate the percent acid-soluble matter as follows:

$$\% \text{ acid-soluble matter} = \frac{\text{g of residue}}{\text{g of sample}} \times 100$$

Sieve Analysis. Transfer a 25-g sample to a 325 mesh sieve and wet with isopropyl alcohol or ethyl alcohol from a wash bottle. Wash the talc through the sieve with tap water. Rinse the sieve with distilled water and dry it at 110°C. Brush the residue retained on the sieve into a small tared weighing dish and weigh. Calculate the percent material passing through the 325 mesh sieve as follows:

$$\% \text{ passing through No. 325 sieve} = \frac{\text{g of sample} - \text{g of residue}}{\text{g of sample}} \times 100$$

Particle Size. Determine the average particle size by use of the Fisher Sub-Sieve Sizer.[15,16]

Apparent Density. Weigh a 50-ml graduated cylinder (ground-glass top) fitted with a rubber stopper. Transfer 30 g of sample to the cylinder by use of a powder funnel. With the stopper in place, drop the cylinder vertically 30 times from a height of 2.0 inches onto a hard leather pad (or book), regulating the height of the fall by inserting the cylinder through a support ring of suitable size. Avoid warming the cylinder by contact with the palms of the hands. Tap the sides of the cylinder gently once with a straight edge to level the height, read the volume occupied, and weigh. Calculate the apparent density as follows:

$$\text{Apparent density} = \frac{\text{g of sample}}{\text{ml occupied}}$$

pH. Weigh a 5-g sample into a 150-ml beaker and add 50 ml of water which had previously been boiled and cooled to about 70°C. Stir well. Cool to 25°C and measure the pH with a pH meter.

Determination of Talc in Propellants

By Determination of the Silica Content of the Propellant. Weigh a 5-g sample of the propellant into a platinum dish, add 10 ml of nitric acid, and warm gently to dissolve. Evaporate to dryness and then ignite at about 600°C until the residue is white. Allow to cool and brush the residue into a platinum crucible. Fuse with 3 g of sodium carbonate over a Meker burner. Allow to cool, place the crucible into a 400-ml beaker containing about 150 ml of water, and boil until the melt is dissolved. Wash down and remove the crucible. Add 35 ml of perchloric acid, evaporate to fumes of perchloric acid, and fume strongly for 20 minutes. (If the sample contains tin, add 20 ml of hydrobromic acid (48%), again evaporate to fumes of perchloric acid, and fume strongly for 10 minutes). Allow to cool, add about 150 ml of water, filter through a Whatman No. 41 filter paper, and transfer and wash with water.

Place the filter paper and precipitate into a platinum crucible, char and burn off the paper while gradually raising the temperature, and then ignite at about 1100°C for 30 minutes. Cool in a desiccator and weigh. Add 3 ml of hydrofluoric acid and 2 ml of sulfuric acid,

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[15] Military Standard MIL-STD-1233, Procedures for Determining Particle Size, Particle Size Distribution, and Packed Density of Powdered Materials, Method 100, March 21, 1962.

[16] Fisher Scientific Co., Directions for Determination of Average Particle Diameters of Powders with Fisher Sub-Sieve Sizer, Pittsburgh, PA.

evaporate to dryness on the hot plate, and ignite at 900°C for 10 minutes. Cool in a desiccator and weigh. Calculate the percent talc in the propellant as follows:

$$\% \text{ talc} = \frac{\text{g loss in weight}}{\text{g of sample} \times 0.55} \times 100$$

By the Determination of the Magnesium Oxide Content of the Propellant (Not Applicable to Samples Containing Calcium). Proceed as in the first paragraph of the above method but save the filtrate and discard the precipitate. Add 5 ml of hydrochloric acid to the solution and heat to about 90°C. Add ammonium hydroxide (1 to 1) until the solution is alkaline to methyl red and add 2 or 3 drops excess ammonium hydroxide (1 to 1). Heat to boiling and boil for 1 or 2 minutes, adding more ammonium hydroxide (if necessary) to maintain the alkalinity. Filter through a Whatman No. 40 filter paper and wash with water. Discard the precipitate. Make the solution just acid with hydrochloric acid. Add 10 ml of diammonium phosphate solution (25%) while stirring vigorously, then add 20 ml of ammonium hydroxide slowly while stirring vigorously and continue stirring intermittently for several minutes. Allow to stand overnight and filter through a Whatman No. 42 filter paper. Transfer and wash with dilute ammonium hydroxide (5 to 95). Place the filter paper and precipitate into a tared platinum crucible. Char and burn off the filter paper slowly while gradually raising the temperature, and then ignite at 1100°C for 30 minutes. Cool in a desiccator and weigh. Calculate the percent talc in the propellant as follows:

$$\% \text{ talc} = \frac{0.362 \times \text{g of precipitate}}{\text{g of sample} \times 0.30} \times 100$$

By Actual Weighing of the Talc (Not Applicable to Samples Containing Tin Dioxide). Weigh a 3-g sample of the propellant into a 250-ml beaker. Add 40 ml of morpholine, cover with a watch glass, and warm on an electric hot plate under a hood until the propellant is dissolved. Heat to boiling, insert a stirring rod, stir, and immediately filter through a tared Gooch crucible (previously ignited at red heat). Wash the beaker and crucible with portions of morpholine that are first placed into the covered beaker and heated nearly to boiling. Transfer and wash with water and then wash with hot hydrochloric acid (1 to 3) and water. Heat at 110°C for 30 minutes and ignite at 700°C for 1 hour. Cool in a desiccator and weigh. Calculate the percent talc in the propellant as follows:

$$\% \text{ talc} = \frac{\text{g of residue}}{\text{g of sample}} \times 100$$

DISCUSSION AND RESULTS

The 5 to 1 perchloric acid was found by innumerable experiments to dissolve the melt away from the sides of the crucible so that the cake floats free when the technique described in this report is used. Hydrochloric acid (no matter what the concentration) was not satisfactory.

The perchloric acid method for silica gave results that were about 0.3% higher than those obtained by the hydrochloric acid method.

The small amount of salts left after the hydrofluoric acid treatment (mostly sodium salts according to spectrographic analysis) dissolves readily in the hydrochloric acid and could be conveniently added to the solution prior to the magnesium oxide determination.

Experiments were conducted to establish the extent of the errors involved if only a single precipitation was made of the magnesium ammonium phosphate. For this study, a standard solution of magnesium was prepared by dissolving 1.9298 g of high purity magnesium metal in hydrochloric acid (1 to 9) and diluting to 1 liter (50 ml = 0.1600 g MgO). Various amounts of hydrochloric acid, perchloric acid, and sodium chloride were added and the magnesium was precipitated, using a single and double precipitation. The results (Table I) showed that surprisingly good results were obtained using one precipitation in the presence of the large amounts of perchloric acid (ammonium perchlorate) in the absence of sodium chloride. However, sodium chloride (5 g) caused rather high results whether the perchloric acid was present or not. Obviously, the reprecipitation of the magnesium ammonium phosphate is necessary.

As indicated by Hillebrand, Lundell, Hoffman, and Bright,[17] any calcium oxide present when magnesium is precipitated as magnesium ammonium phosphate is converted on ignition essentially to $Ca_3(PO_4)_2$. This was checked by adding various amounts of standard calcium oxide solution (prepared by dissolution of calcium carbonate in dilute hydrochloric acid) to portions of the standard magnesium solution, precipitating the magnesium as described in the procedure, and making a correction for $Ca_3(PO_4)_2$, using the theoretical factor of 1.844 for converting CaO to $Ca_3(PO_4)_2$. The results obtained in this study (Table II) showed that the correction is valid although slightly high results are obtained in the presence of more than about 0.02 g CaO. It is probable that more accurate results can be obtained for the determination of magnesium oxide in the presence of calcium oxide by use of the correction technique than can be obtained by use of

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[17] W. F. Hillebrand, G. E. F. Lundell, H. A. Bright, and J. I. Hoffman, "Applied Inorganic Analysis," p. 640, Wiley, New York, 1953.

Table I.
Effect of Perchloric Acid (Ammonium Perchlorate) and Sodium Chloride on the Precipitation of Magnesium Ammonium Phosphate

Added	No. of Precipitations	MgO Found (g)
0.1600 g MgO + 1 ml HCl	1	0.1597
0.1600 g MgO + 5 ml $HClO_4$	1	0.1602
0.1600 g MgO + 10 ml $HClO_4$	1	0.1605
0.1600 g MgO + 20 ml $HClO_4$	1	0.1609
0.1600 g MgO + 30 ml $HClO_4$	1	0.1591
0.1600 g MgO + 5 g NaCl + 1 ml HCl	1	0.1680
0.1600 g MgO + 5 g NaCl + 10 ml $HClO_4$	1	0.1670
0.1600 g MgO + 5 g NaCl + 20 ml $HClO_4$	1	0.1683
0.1600 g MgO + 5 g NaCl + 30 ml $HClO_4$	1	0.1654
0.1600 g MgO + 5 g NaCl + 10 ml $HClO_4$	2	0.1596
0.1600 g MgO + 5 g NaCl + 20 ml $HClO_4$	2	0.1597
0.1600 g MgO + 5 g NaCl + 30 ml $HClO_4$	2	0.1600
0.1600 g MgO + 5 g NaCl + 30 ml $HClO_4$	2	0.1598

Table II.
Results for Magnesium Oxide after a Correction for Calcium Oxide (Deduction for $Ca_3(PO_4)_2$ in the Ignited Precipitate)

MgO Present (g)	CaO Present (g)	MgO Found after Correction (g)
0.1600	0.0000	0.1601
0.1600	0.0000	0.1596
0.1600	0.0010	0.1598
0.1600	0.0050	0.1600
0.1600	0.0100	0.1604
0.1600	0.0200	0.1608
0.1600	0.0250	0.1606
0.1600	0.0500	0.1610
0.1600	0.1000	0.1611
0.0000	0.0100	0.0004
0.0000	0.0250	0.0003
0.0000	0.0500	-0.0001
0.0000	0.1000	0.0006

the conventional method involving separation of calcium by precipitation as calcium oxalate, since the precipitation of calcium oxalate is never quite complete and calcium oxalate tends to occlude magnesium.

It would be expected that the atomic absorption method for calcium would be more accurate than the calcium oxalate precipitation method insofar as the relatively small amounts of calcium oxide found in high-purity talc are concerned. This follows since the atomic absorption method eliminates the errors caused by incomplete precipitation of the calcium oxalate and the occlusion of salts by calcium oxalate. In addition, the atomic absorption method eliminates the hygroscopicity error of the CaO ignition procedure.

The loss on ignition of the talc represents loss of CO_2 and some of the combined water (OH). Apparently, all of the combined water is not eliminated. On heating at about 900° - 1000°C, talc is converted into a mixture of amorphous SiO_2 and $MgSiO_3$.[18,19]

The results obtained for silica, magnesium oxide, R_2O_3, ferric oxide aluminum oxide, calcium oxide, zinc, sulfate, phosphorus pentoxide, moisture, loss on ignition, water-soluble matter, acid-soluble matter, sieve analysis, average particle size, apparent density, and pH in four samples of high-purity talc are shown in Table III. Also shown are results for spectrographic analysis. The results obtained for the determination of talc in propellants by the three methods (SiO_2, MgO, and morpholine methods) are shown in Table IV. The methods check each other. For general use the SiO_2 method is recommended.

On the basis of the results (Table III) and the theoretical composition of talc, it is recommended that a proposed specification for talc for use in propellants should include the suggested requirements shown in Table V. It was not deemed necessary to have a requirement (or test) for acid-soluble matter, phosphorus pentoxide, sulfate, and apparent density, insofar as a specification is concerned.

The requirement for zinc (zinc stearate) was left in abeyance for the following two reasons: (1) it will be necessary for the propellant group to establish whether the addition of zinc stearate to talc is advantageous, (2) the analytical work on the determination of zinc stearate in talc is inconclusive.

Concerning the analytical work, Mistron ZSC, which is coated with zinc stearate according to the manufacturer,[20] contained 0.008% zinc

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[18] M. Koltermann, Neues Jahrb. Mineral, Monatsh., (4), 97 (1964).

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[20] United Sierra Division, Cyprus Mines Corp, Typical Data Sheet, Mistron ZSC, Box 1201, Trenton, NJ.

Table III.
Results for the Analysis of Four Samples of High-Quality Talc

Determination	Results (%)			
	Supreme	Mistron Vapor	Mistron ZSC	Fisher (USP)
Color	white w/slight tint of grey	same	same	same
SiO_2	60.8; 61.0; 61.0	61.0; 61.3; 61.4	61.6; 61.4	61.6; 61.5
MgO	31.6; 31.3; 31.2	31.6; 31.4; 31.4	31.1; 31.4	31.4; 31.6
R_2O_3	1.16	1.41	1.47	1.16
Fe_2O_3	0.38	0.11	0.16	0.22
Al_2O_3	0.78	1.30	1.31	0.94
CaO	0.02	0.48	0.40	0.43
Zn	0.0006; 0.0005	0.001; 0.002	0.008; 0.008	0.005; 0.004
SO_4	<0.01	<0.01	<0.01	<0.01
P_2O_5	0.23	<0.001	<0.001	<0.001
CO_2	0.15	0.84	1.03	0.29
Moisture	0.22	0.27	0.25	0.17
Loss on ignition	1.52	4.41	3.61	2.88
Water-soluble matter	0.07	0.10	0.11	0.11
Acid-soluble matter (5 to 95 HCl)	2.20	3.13	2.69	0.90
Through 325 sieve	100	100	100	100
Avg. particle size (microns)	1.95	0.58	0.60	1.45
Apparent density, g/ml	0.51	0.18	0.18	0.45
pH (in pH units)	7.8	8.4	8.5	7.2
Spectrographic analysis [a]				
Na	<0.1	<0.1	<0.1	<0.1
K	<0.1	not detected	not detected	not detected
Ti	not detected	<0.02	<0.02	<0.02
Mn	<0.002	<0.03	<0.03	not detected
Sn	<0.1	not detected	not detected	not detected
B	<0.02	not detected	not detected	not detected
Ni	<0.01	<0.005	<0.005	<0.005
Cu	<0.001	<0.005	<0.005	<0.005

[a] The following were not detected: Li, Sr, Ba, Bi, Cd, Hg, V, Mo, Pb, As, Co, Ag, Zr, W, Sb, Be, and Cr.

Table IV.
Results for Talc in Nitrocellulose-Base Propellants

Sample	By Determination of SiO_2	By Determination of MgO	By Morpholine Method
8426 [a]	0.70; 0.69	0.70; 0.73	0.70; 0.71
8471 [b]	0.48; 0.44	0.48; 0.47	0.48; 0.44

[a] Contains (%): 0.32 K_2SO_4, 1.67 dibutylphthalate, 0.72 diphenylamine, 3.95 methyl centralite, 0.54 graphite.

[b] Contains (%): 0.04 K_2SO_4, 1.61 dibutylphthalate, 0.85 diphenylamine, 6.20 methyl centralite, 0.43 graphite, 0.66 metallic tin.

Table V.
Recommended Requirements for a Specification for Talc for Use in Propellants

Test	Requirement
Color	White or greyish white
Silica (SiO_2)	60.5 - 63.5%
Magnesium oxide (MgO)	31.0 - 32.0%
R_2O_3	1.6% max
Iron oxide (Fe_2O_3)	0.5% max
Aluminum oxide (Al_2O_3)	1.5% max
Calcium oxide (CaO)	0.6% max
Carbon dioxide (CO_2)	1.2% max
Moisture	0.3% max
Loss on ignition	2.0 - 5.0%
Water-soluble matter	0.15% max
Through No. 325 sieve	100%
Average particle size	0.5 - 2.0 microns
pH	7.0 - 8.5

(equivalent to 0.078% zinc stearate). Mistron Vapor, which is very close in composition to Mistron ZSC, contained 0.001% zinc. However, Fisher USP, which apparently was not coated with zinc stearate, contained 0.005% zinc. Obviously it would seem desirable to develop a method for analyzing for zinc stearate in talc by determining the stearate radical. It is believed that this determination could be performed by gas chromatography.

It is suggested that work proceed on writing the specification for talc for use in propellants without waiting for the clarification of the zinc stearate requirement. When the latter problem is finally resolved, an addendum to the specification could be issued establishing a grade of talc containing zinc stearate.

SUMMARY

Methods are described for the analysis and characterization of high-purity talc for use in nitrocellulose-base propellants. Procedures are described for silica, magnesium oxide, R_2O_3, ferric oxide, aluminum oxide, calcium oxide, zinc, sulfate, phosphorus pentoxide, carbon dioxide, moisture, loss on ignition, water-soluble matter, acid-soluble matter, sieve analysis, average particle size, apparent density, and pH. In the method for silica and magnesium oxide, the sample is fused with sodium carbonate and the melt is dissolved in such a manner with 5 to 1 perchloric acid that the cake floats free of the crucible (in previous schemes for the analysis of talc there are frequently mechanical losses due to the difficulty of removing the magnesium silicate melt from the crucible). The solution is then evaporated to fumes of perchloric acid and the silica is filtered and ignited. The magnesium oxide is determined in the filtrate by precipitation as magnesium ammonium phosphate and a correction is made for the calcium which is precipitated with the magnesium ammonium phosphate. R_2O_3 is determined after treatment with nitric and hydrofluoric acids and evaporation to fumes of perchloric acid. Iron oxide, aluminum oxide, calcium oxide, and zinc are determined by atomic absorption after treatment with nitric and hydrofluoric acids and evaporation to fumes of perchloric acid. Sulfate and phosphorus pentoxide are determined by the precipitation as barium sulfate and ammonium phosphomolybdate, respectively, after treatment with nitric and hydrofluoric acids and evaporation to fumes of perchloric acid. Carbon dioxide is determined by combustion in an induction furnace with iron as a flux. The methods for moisture, loss on ignition, water-soluble matter, acid-soluble matter, sieve analysis, average particle size, and pH are more or less conventional.

Three methods are described for the determination of talc in nitrocellulose-base propellants. The first method (which is the method recommended for the usual nitrocellulose-base propellant) is based on the silica content of the propellant, while the second method is based on the magnesium oxide content. In the third method, the talc is weighed as such after dissolution of the propellant in morpholine, filtration, washing of the residue with water, and dilute hydrochloric acid, and ignition (to eliminate the graphite).

RECOMMENDATIONS

It is recommended that the work in this report be used as a basis for a specification for talc for use in propellants (and possibly other explosives). Also, it is recommended that the method for talc, depending on the silica content, be incorporated into MIL-STD-286B (Military Standard, Propellants, Solid: Sampling, Examination, and Testing).

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