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SUBJECT: Talc Analysis

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The attached paper, Microscope Procedure for Talc Powders, is a practical procedure and guideline for the microscopic evaluation of cosmetic grade talc powders. It is intended for hands on use with the Zeiss Universal polarizing microscope.


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MICROSCOPIC PROCEDURE FOR TALC POWDERS

I. SETTING UP:

A. Sample Preparation:

1. Powder:
 - a. 100 mesh or below: Use as is.
 - b. Larger than 100 mesh: Screen through 100 mesh and grind the plus (on) 100 mesh in mortar and pestle or ball mill to reduce to minus 100 mesh.
2. Rocks: Pulverize to minus 100 mesh in mortar and pestle, and ball mill if necessary.

B. Sample Mounting:

1. Clean slide with IPA.
2. Place one small drop of appropriate index oil on slide before adding the powder.
3. Add small "pinhead" amount of powder to oil and mix in gently to disperse as evenly as possible.
4. Gently place an 18 mm square cover slip over specimen preparation.

C. Set Microscope for Critical Illumination:

Critical or Kohler illumination technique is detailed on page 8 of the Zeiss operating instructions manual G41-140-E.

1. Open the condenser iris diaphragm wide and using the 16X objective, focus the specimen.
2. Close the field (illuminator) diaphragm almost completely and bring it into the field of view using the condenser centering screws.
3. Close the field diaphragm completely, focus it sharply by racking the condenser up or down and then center the round spot of light on the crosshairs using the condenser centering screws.
4. Open the field diaphragm so the light just fills the field of view. The microscope is now set for critical illumination.
5. If the objective is changed to another powder (6.3X or 60X etc.) steps 1 through 4 should be repeated for maximum viewing results.

6. After the above steps have been taken the condenser iris diaphragm can be closed to obtain the desired brightness preferred for viewing.

II. VIEWING MODES:

A. Plain Polarized Light:

Light from the illuminator passes through the lower (condenser) polarizing filter and provides plain polarized light for normal "bright field" observation.

B. Crossed Polarized Light:

When the upper polarizing filter (analyzer) is slid into the light path, the polarizing elements cancel each other and a dark field results. Crystalline (birefringent) particles on the specimen stage will now appear as bright objects on a dark background. This is probably the best set up for viewing talc samples.

C. Nomarski Differential Interference Contrast:

(See pages 12 and 13 of Zeiss Operating Instructions for detailed instructions).

This method of viewing is ideal for bringing out surface detail on transparent objects. It also enhances contrast between two different materials such as an oil in water emulsion or irregularities on a glass or plastic surface. The surface of larger talc plates may be studied in this way.

D. Phase Contrast:

(See pages 10 and 11 of the Zeiss Operating Instructions)

Phase contrast is also used to increase the contrast between transparent objects. It has no special advantage in talc powder evaluation.

E. Dispersion Staining:

This technique may be used for all minerals, but is especially useful for the detection of quartz particles in talc grinds. Using the dispersion staining objective with central stop, quartz particles will appear blue in 1.550 RI oil. Talc plates on edge and talc shards also appear blue, but not in all positions of stage rotation. Other particles will be colorless or faintly orange, yellow or blue.

F. Conoscopic Observation:

This is a method to view the interference pattern, formed by a crystalline particle, in the back focal plane of the objective. These patterns are distinctive and different for the uniaxial and the biaxial minerals and may be used in identification. This method is especially valuable for recognizing doubtful carbonate mineral particles.

See page 64 of The Particle Atlas for explanation and use of interference figures.

III. TALC SAMPLE EVALUATION:

Mount the sample in 1.600 R.I. oil as described in part I B.

- A. Determine that the sample contains talc (see Mineral Identification sheets).
- B. Determine the shapes of the talc particles present, e.g. platy, scaly or granular and estimate the amounts of each type of talc present. In talc powders which have not been ground to the liberation point, the platy and scaly talc will be present as large agglomerated granular shaped bundles. These may be ground further to liberate the plates and scales as smaller individual particles for evaluation.

The preferred talc particle for Baby Powder is a thin platy or flaky particle about 15 to 75 microns across and with a smooth flat surface. A model for this type of talc is Windsor V-66 talc. This type of particle gives the talc excellent lubricity, spreadability and softness on the skin.

Very fine platy particles, scaly, and scaly agglomerates and granular talc detract from and decrease the natural lubricity of the larger platy talc particles. For acceptable and preferred skin response, a talc should usually contain at least 85% of platy talc particles.

- C. Identify and estimate the amounts of non-talc associate minerals in the talc powder (see Mineral Identification sheets). The amounts may be estimated by weight percent based on the following table:

	<u>% by weight</u>
Trace	0.5% or less
Minor	0.5% to 10%
Major	over 10%

D. From a product safety standpoint it is necessary that the talc powder be free of asbestiform minerals. These are chrysotile (fibrous serpentine), and the fibrous forms of the amphibole mineral group. If amphiboles are present they will usually be tremolite/actinolite and rarely anthophyllite. Trace amounts of nonfibrous tremolite/actinolite are sometimes observed, however, this is not grounds for rejecting the talc. If the tremolite/actinolite is present in minor amounts, this would be a high risk talc and should not be considered for use in JBP. Microscopy by itself is not sufficient to claim a talc is free of asbestiform minerals. SEM plus EDAX analysis and X-ray diffraction (CTFA J4-1) tests should also be run to determine freedom from asbestos.

IV. MINERAL IDENTIFICATION SHEETS:

The following sheets describe applied methods for the practical identification of the common associate minerals found in talc powders. The methods provide that the sample is mounted in 1.600 R.I. oil and is viewed under crossed or slightly uncrossed polars.

Talc:

Platy talc and scaly talc appear pale white to transparent. Extinction is wavy and irregular. Turned up edges and plates on edge look like elongate rods or needles; these have sharp parallel extinction. Plates or shards on edge may often be flipped over by lightly pressing on the cover glass, then the true nature of the particle becomes evident. Small granular talc particles show sharp extinction. Larger platy or scaly aggregates show no extinction and the laminar structure of the ore is often quite apparent.

The Becke line shows the refractive index of talc to be below 1.600 in all positions. Elongate fragments and plates on end have a higher refractive index than flat exposures and are length slow (blue when parallel to the slow direction of the Gypsum Plate).

Tremolite/Actinolite (Amphiboles):

These particles are usually white to yellow white elongated prisms with parallel sides and sometimes with surface cleavage lines or furrows parallel to the length. The ends are broken in irregular patterns.

Extinction is oblique and the angle of extinction may be as high as 20%. On rare occasions the particles will be oriented to show parallel extinction and must be reoriented to show the oblique extinction.

Refractive indices $X = 1.599 - 1.612$, $Y = 1.613 - 1.626$, $Z = 1.625 - 1.637$. In all the possible orientations the Becke line will indicate a higher index than the 1.600 mounting oil. The Becke line and mineral fragment will have least contrast when viewed parallel to the X direction, and maximum contrast when viewed parallel to the Z (elongate) direction.

Elongate sections are length slow, e.g., the slow ray (highest index = Z) is in the elongate crystal direction. When the length of the particle is aligned in the same direction as the slow ray in the gypsum plate, the particle will appear blue.

Tremolite/Actinolite particles tend to remain large in talc grinds. Therefore, larger crystals can be more easily found (for identification) by wet screening the talc through a 400 mesh screen and looking at the plus 400 mesh fraction.

Carbonate Minerals (Calcite, Magnesite, Dolomite):

The three common carbonate minerals found in talc are listed above. These minerals are difficult to tell apart by optical mineralogy, and may simply be listed as carbonate mineral, since their properties are very similar.

The carbonates crystallize in the hexagonal system and occur as equidimensional rhomb or irregular granular particles. Fragments may also appear as slightly elongated prisms. Twinning striations bisecting the corners of the rhomb are often observed.

Birefringence is high (1.658 - 1.486=1.72) and colors are often opaque white. Thinner particles begin to show lower order interference colors and color fringes are often present in irregular flake shaped particles.

As may be seen from the R.I. values given above, the Becke line is readily apparent in 1.600 R.I. oil and indicates a much higher and much lower index in the 90° opposing extinction positions.

A distinctive feature of the carbonates in talc grinds is the interference figure obtained using conoscopic observation. The uniaxial cross pattern and birefringent color rings of the carbonate interference figure are quite different than the other minerals which might be present, e.g., talc, amphiboles (tremolite), quartz or chlorite. See page 64 of The Particle Atlas for explanation and use of interference figures.

Quartz:

Quartz is one of the commonest minerals, and is present to some degree in nearly all talc samples. The particles occur as irregular shaped angular fragments usually white to grey in color. Thicker chips may have a yellow color. The particles show no orientation of shape and extinction (like the amphiboles). Quartz crystallizes in the hexagonal system, so the conoscopic interference pattern shows a uniaxial cross in correctly oriented fragments. There are no color fringes since the birefringence is low.

The easiest identification for quartz in talc is to use the dispersion staining technique with 1.550 high dispersion R.I. oil. See Section II Viewing Modes, part E.

Chlorite:

Chlorite is a generic name for a group of micaceous clay like minerals comprising mg, al silicate with varying amounts of iron. Optically chlorite is difficult to distinguish from talc. In some cases a high iron content will give the scaly aggregate particles a yellow to dark brown color. It will be necessary to depend on X-ray Diffraction analysis to determine chlorite in talc powders.

Rutile:

This mineral is sometimes found occurring as small inclusions in the talc particles. Due to its very high refractive index (over 2.0) it is apparent as very high contrast particles when observed in plain polarized light. The Becke line is very bright. Rutile particles will be red to brown in color.

Opaque Minerals:

Completely opaque or nearly opaque particles are usually minerals containing high concentrations of iron or nickel. These can not be identified by optical transmission microscopy and should be listed simply as opaque minerals. However, one exception is the mineral magnetite (Fe_3O_4) which is magnetic and may be seen to move in the oil under the influence of a strong magnet applied close to the cover slip.

V. REFERENCE MATERIAL:

The following reference material will be of great value and are available in the Microscope room.

A. The Particle Atlas:

A photomicrographic reference for the microscopical identification of particulate substances.

B. The Industrial Use of the Polarizing Microscope:

A course manual specifically prepared by McCrone Institute for the use of the polarizing microscope in mineral identification.

C. Zeiss Operating Instructions - G41-140-E:

Instructions and illustrations for the operation of the Zeiss Universal microscope.

D. Microscopy From the Very Beginning:

F. K. Mollring
General information on microscopy for the beginner and experienced microscopist.

E. Textbooks on Mineralogy:

Elements of Optical Mineralogy
Volumes I, II, III Winchell & Winchell

Optical Mineralogy - Kerr

Microscopic Determination of the Ore Minerals -
Geol. Survey Bulletin 914

The Microscopic Determination of the Nonopaque Minerals -
Geol. Survey Bulletin 848

Microscopic Determination of Nonopaque Minerals
Albert S. Wilkerson - Rutgers University 1953

F. Prepared Slides and Powdered Samples:

These consist of pre-prepared slides and powdered samples of the actual minerals normally associated with talc powders.

Also a variety of talc samples gathered worldwide.