DETECTION OF ASBESTIFORM AMPHIBOLE IN TALC FOR COSMETIC USE

The method for the detection of asbestiform[1] amphibole in talc for cosmetic use consists of initial analysis for amphibole minerals above the 0.5% (w/w) level, followed by characterization of the amphibole mineral present, if any, as asbestiform or nonasbestiform by dispersion staining/optical microscopy.

X-Ray Diffractometry

The procedure consists of slow-scanning, under previously determined conditions, a compressed pellet of the sample talc in the 11.0-10.0°2θ (8.85-8.04Å) region for the presence of an amphibole peak.

1 - Standard Preparation

Optimal instrument conditions must first be determined with the use of tremolite standards - 1.0%, 0.75%, 0.5% tremolite by weight, prepared in a standard talc which is free of interfering peaks in the 11.0-10.0°2θ region. The tremolite standards are prepared in the following manner:

a. Weigh out appropriate amounts of standard talc and tremolite of at least 95% purity[2], both of which have been ground to pass a 325-mesh sieve. Transfer to a Waring Blendor® (or equivalent). Add 100 ml. of ethanol to the blender and blend at low speed for 5 minutes.

b. Carefully transfer, with repeated ethanol washings, the contents of the blender into a large beaker. Evaporate the ethanol on a steam bath.

c. The dried sample is then shaken in a plastic vial for 5 minutes on a Spex Mixer/Mill® (or equivalent) to remove clumps and caked sample resulting from the evaporation of ethanol.

d. The homogeneity of the prepared standard is determined by microscopy previous to the X-ray diffraction analysis.

1. According to the Mining Enforcement and Safety Administration and the National Institute of Occupational Safety and Health (Federal Register, Vol. 39, No. 127, p. 24319, July 1, 1974) the term "asbestos" is not applicable to the nonfibrous form of tremolite which has been found in certain talc deposits.

2. A commercial source of high purity tremolite is currently being sought, since much difficulty has been encountered in obtaining high grade tremolite.

e. The homogeneous standard is then pressed into a 1-1/4" pellet with a backing of boric acid. 2 gram of standard is transferred to the die-holder and evenly distributed on a polished, scratch-free die. 4 gram of boric acid is then distributed evenly on the talc layer. The mixture is pressed into a pellet under conditions suitable for obtaining a smooth planar surface (for example, a pressure of 15,000 - 24,000 lb.), calculated on a 3" ram, was found to produce suitable pellets). The resulting pellet must have a talc face which is free of flaws; if not, the pellet must be discarded. (This requirement is critical since excessive surface scatter will cause abnormally high background counts.) Two acceptable pellets are prepared from each standard.

2 - Sample Preparation

Two pellets are prepared from each sample in the manner described for the standard pellets (1., Step e.). A qualitative scan from 4-50°2θ is made on one of these pellets to ascertain the presence of amphibole above the 2% level or the presence of mineral impurities having interfering peaks in the 11.0-10.0°2θ (8.85-8.04Å) region of the scan. The presence of such interference will eliminate use of the x-ray diffraction method for the sample, and one will have to proceed directly to the microscopical procedure.

3 - Instrumentation

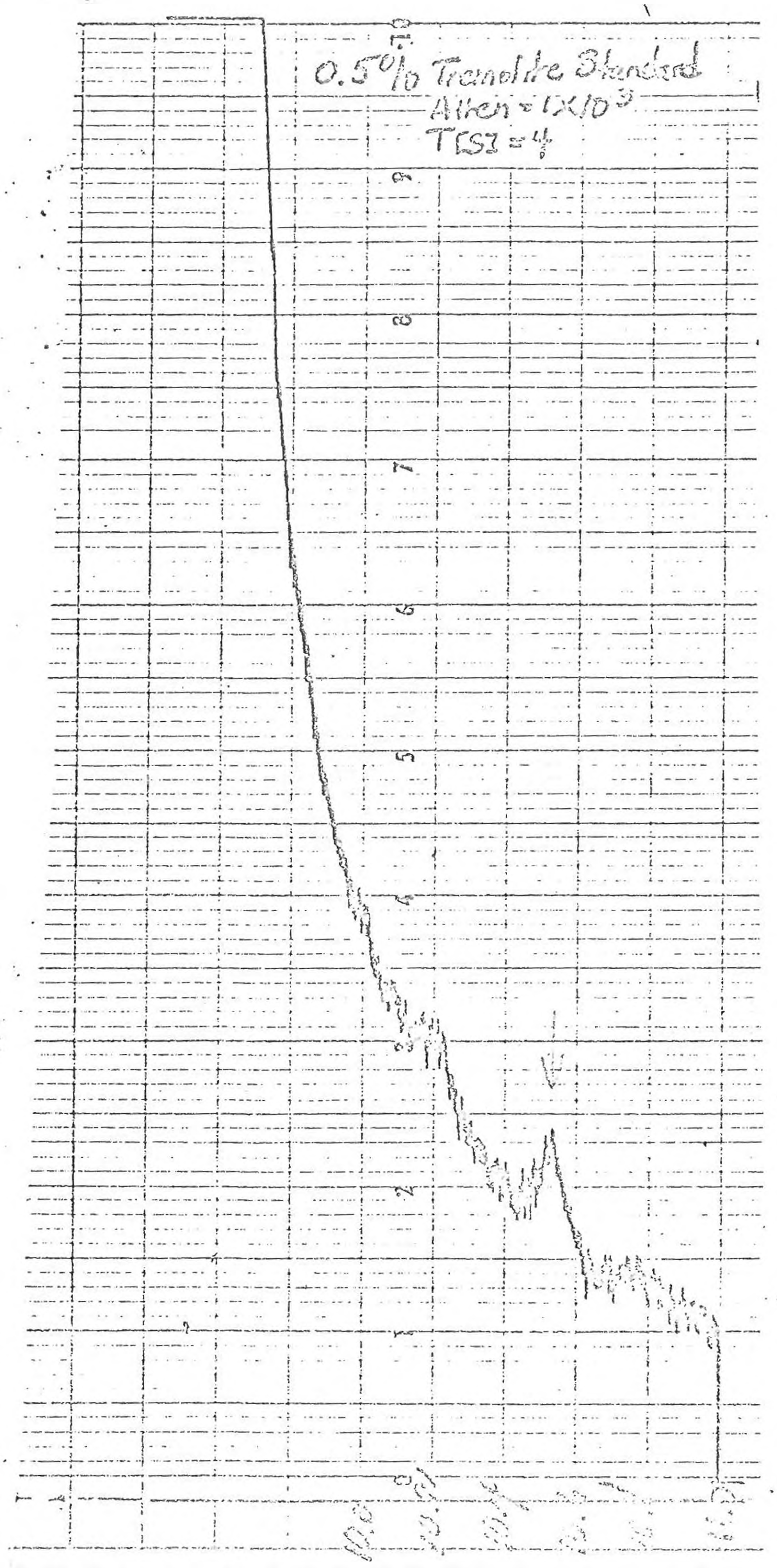
Instrumental variables are optimized on the 1% standard. Lower standards are then analyzed under the optimum conditions to determine the lower level of detection. Of major importance in obtaining maximum instrument sensitivity are a slow diffractometer speed combined with a compatible recorder speed, and high attenuation combined with a statistically acceptable time constant on the ratemeter. Under appropriate instrumental conditions the peak obtained for the 0.5% standard should be easily detectable above background noise as per the scan on the following page.

Instrumental conditions employed for the Siemens Diffractometer (Model No. M386-X-A4), and Counter and Recorder Unit (Type T) are:

Radiation: Cu with K_β filter at 40 KV and 24 ma
Divergence slit: 1° Receiving slit: 0.2 mm
Goniometer speed: 1/10°2θ/minute
Recorder speed: 300 mm/hr.
Attentuation = 1×10^3 impulses/second
Time constant, T (ε) = 4

Statistical error of 1.1% under these conditions.

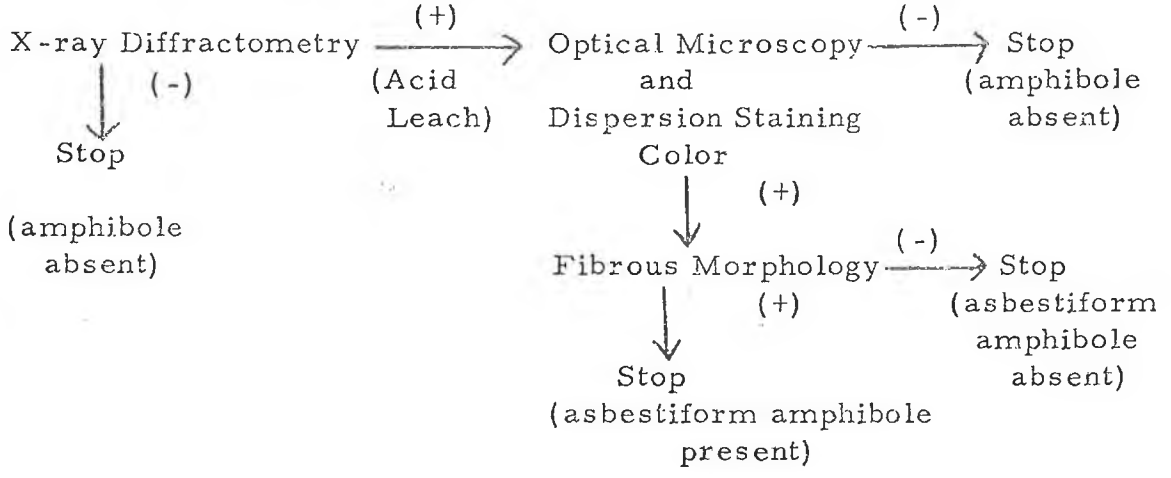
Rise Time = 0.18
Attenuator = 20


0.5% Tremolite Standard
Atten = 1X10^3
TCSI = 4

4 - X-Ray Diffraction Scans

The standard or sample pellet is placed in a suitable holder and slowly scanned between 11.0 and 10.0°2θ. The pellet is then rotated 90° with respect to its original position in the goniometer and rescanned between 11.0 and 10.0°2θ since pellet orientation may affect peak intensity. The presence of a reproducible peak (or peaks) is due to the presence of amphibole minerals; the absence of peaks in this region indicates the absence of amphibole in the sample, within the limit of detection of this technique.

If amphibole is detected in one or both pellets by x-ray diffractometry, then the sample must be put through the following confirming scheme:



Because of the interference caused by some carbonates (e.g. calcite) in the detection of tremolite asbestos in talc by optical microscopy/dispersion staining, it is necessary to first remove these carbonates by a simple acid leaching procedure:

1. Weigh out 2 g. of the talc into a 100 ml. beaker. Add 25 ml. of 10% v/v HCl slowly (to prevent excessive evolution of gas if carbonates are present) and heat, with occasional stirring on a steam bath for 30 minutes.

2. Filter on a vacuum filter, in either a porcelain cone with glass fiber filter mat or a porous glass bottom cup, and wash several times with hot water. Dry the talc.

Optical Microscopy and Dispersion Staining

1. Carefully disperse about 0.2 mg. of talc in one drop of Cargille HD liquid $n_{25}^{D} = 1.605$ and cover with a clean cover slip.

2. Examine the sample in the D. S. central stop mode. The substage diaphragm should be almost completely closed, the field diaphragm

may be partially closed to enhance color contrast, and the polarizer should be in position.

3. Tremolite, actinolite and presumably other amphibole minerals, under these conditions, will show the following D. S. colors: yellow changing to blue with rotation of the sample relative to the polarizer or yellow changing to orange with rotation. The variation of the color change is due to the fact that the tremolite may lie in one of two positions relative to its principal optical orientation.

4. In order for an amphibole mineral to be considered asbestiform or fibrous it must meet the following OSHA definition[3].

   A. Particles must appear to be fibrous rather than as crystals or slivers.

   B. The maximum diameter of a fiber to be counted is 3 microns.

   C. The maximum length of a fiber to be counted is 30 microns.

   D. The length to width ratio must be 5 or more to 1, that is, 5 times or more longer than wide.

   E. The separate or individual fibers must contain fibrils or the "bundle of sticks" effect, unless they are at a non-divisible stage. A fibril cannot be subdivided and would be counted, if it meets the other criteria. The electron microscope may be used to prove the fibrous nature of the particles. The length to width ratio of 5 or more to 1 is not meant to imply that other particles are not hazardous.

5. It is imperative that both D. S. color and fibrous morphology criteria be satisfied before identifying a particle as asbestiform amphibole since other substances (e.g. calcite) may show colors similar to those described.

[3] "Tremolite and Talc". U. S. Department of Labor, Occupational Safety and Health Administration, Field Information Memorandum #74-92, November 21, 1974.