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To: Windsor Minerals Inc., Windsor, Vermont 05089

From: R.C. Reynolds Jr., Department of Earth Sciences, Dartmouth College, Hanover, New Hampshire 03755

Subject: Analysis of Talc Products and Ores for Asbestiform Amphiboles

INTRODUCTION:

The purpose of this study is to develop methods for measuring the concentration of asbestiform amphiboles in fine-grained talc products and talc ores. In principle, the problem might be simply solved by the microscopic counting of amphibole grains in samples that are suspended in oils of suitable refractive index, so that amphiboles are optically emphasized with respect to talc. In practice, however, grain counting is valid only if the dimensions of each grain are measured. This requirement arises from the large particle size range present, and from the wide variation of aspect ratios among the amphibole grains (see plate 16). In any event, grain-counting methods are inapplicable to whole samples because an inordinate number of grains must be considered. This makes the analysis time prohibitively long. For example, if a concentration of 100 ppm amphibole is assumed, and if 100 amphibole grains is the acceptable minimum that provides good statistical data, then one million grains must be considered. This requirement would be

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easily met if the amphiboles were easily distinguished from other minerals. But even in suitable refractive index oils, many grains are ambiguous and require manipulations in order to verify their identity. Such ambiguous grains are (1) fibrous talc and carbonate grains in certain orientations (plate 21), (2) inclusion-filled grains (plate 21), (3) broken amphibole grains that have equant shapes (plate 9), and (4) grain aggregates (plate 20). If only 1% of the grains are ambiguous, and experience indicates that this is a conservative figure, then the example cited above would demand that attention be paid to 10,000 grains in a single sample.

For the reasons described above, a concentration technique is mandatory because it brings the amphiboles into a reasonable concentration range for optical or other methods of analysis. Such a method has been developed, and it is described in this report.

EXPERIMENTAL METHODS:

Advantage can be taken of difference in density between talc and amphiboles (see Table 1). The sample can be suspended in bromoform and centrifuged to float talc and settle denser minerals. However, the fine-grained nature of the ground ores and products brings colloidal forces into play which cause flocculation of the sample, and this renders a clean separation impossible.

Dispersion of talc in bromoform requires that the particles

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be plated with organic molecules whose film thicknesses separate the grains sufficiently so that van der Waals forces cannot cause agglomeration. In addition, the plated grains must make up an oleophilic colloid, that is, they must be wetable by non-polar organic solvents (bromoform). Experiments were performed with various concentrations of butylamine hydrochloride, cetyltrimethylammonium bromide and benzethonium chloride monohydrate. The latter reagent was far superior in promoting dispersion in bromoform. The effectiveness of dispersion was judged by the time required for visual evidence of flocculation of the talc product.

The addition of benzethonium chloride monohydrate (hereafter, abbreviated BCM) lowered the density of the bromoform. Methylene iodide was added to bring the density back to desired levels. The proportions of sample and reagents used for the separations described in this report, are:

1 g talc,

2.0 g BCM,

20 ml Bromoform (d=2.8) and

8.4 ml Methylene iodide (d=3.3).

This mixture provides a density of 2.88 g/cm^3, at 20°C, as measured by pycnometer. Attempts to raise the density by further additions of methylene iodide promoted flocculation. Consequently, future separations of amphiboles should be made at reagent concentrations similar to these.

Separations of amphiboles were made from ground talc product and from talc ore, provided by V. Zeitz of Windsor Minerals.

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Talc was weighed into centrifuge tubes, and the reagents added in the proportions cited above. The tubes were shaken vigorously for approximately one minute, placed in a size 2 International Centrifuge, and centrifuged for 5 minutes at 500 RPM, followed by 5 minutes at 1800-2000 RPM. The centrifuge was allowed to slow with no braking (to minimize counter-rotating currents in the liquid) and the tubes were withdrawn and placed in racks for isolation of the heavy mineral fractions.

A glass rod was fitted with a rubber stopper, and this was carefully inserted into the tube and lowered to the bottom soas to isolate the heavy fraction (See Figure 1). The tube was decanted* and flushed with acetone from a polyethylene wash bottle. The plunger was removed, washed with acetone into the tube, and the tube was filled with acetone and shaken and centrifuged. The heavy fraction was washed twice more with acetone by means of the centrifuge, decanted, and dried overnight at 80°C. The tube with sample was cooled in a dessicator and weighed on an analytical balance. The concentrate at this stage consisted mostly of carbonate (magnesite plus some dolomite, see Plate 5). The carbonates were removed by acid dissolution as described below.

The sample tube was filled with 4NHCl and heated in a water bath for 2 hours at 80-90°C. The sample was centrifuged

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* The reagent mixture cannot be saved. Slow decomposition occurs which liberates iodine and reduces the density. After 24 hours this process is sufficiently severe to lower the density below that of talc.

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twice more to wash soluble salts out of the insoluble heavy mineral fraction. It was dried overnight at 80°C. The sample was scraped out of the tube, transferred to a vial, and the centrifuge tube was washed, dried, and weighed. From the three weights obtained, values were calculated for (1) percent heavy minerals, and (2) percent insoluble heavy minerals.

Talc ore and talc product, provided by V. Zeitz of Windsor Minerals, were run through this procedure. In addition, talc ore was spiked with known amounts of actinolite (ground and sized 2-2μ by settling in water), and separated to test the efficiency of the method. The concentration of actinolite in the product concentrate was estimated by optical examination of the insoluble heavy mineral fraction. A simple estimate was made, and this was normalized to the total sample weight by means of the figure for the percentage of the total represented by the separate. Values for actinolite in the ore and in the spiked samples of ore were obtained by an X-ray fluorescence method that utilizes known amounts of potassium added as an internal standard. X-ray fluorescence methods were used because of the failure of various X-ray diffraction methods which were attempted. All of these had unacceptable precision (±100%), probably due in most part to the small (milligram) amounts of material available for analysis. Consequently, the X-ray fluorescence method provides the best means of measuring actinolite, although it would be useless for the determination of other fiberform amphiboles.

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RESULTS:

Two-gram samples of ore and ore spiked with actinolite were separated and analyzed as described above. Results for these samples are summarized by Table 2. Table 3 shows concentrations of heavy fractions (mostly carbonate), and of actinolite in talc ore and talc product. Actinolite concentration in the product was measured for a 12 gram sample.

DISCUSSION:

The data of Table 2 show that (1) the total amount of heavy minerals separable from talc ore is reproduceable, and (2) the amounts of actinolite recovered from spiked samples agree quite closely with the actual amounts added. The agreement between actinolite found and actinolite nominal is about as good as can be expected, given the existence of errors of one or more milligrams that can arise as a result of the weighing procedures described above.

The samples studied contain actinolite as the dominant fiberform amphibole phase. This conclusion is based upon (1) the common occurrence of extinction angles of 15°, (2) the α refractive index of 1.616, and (3) the high calcium content of the residues which contained no other calcium-bearing minerals. However, some anthophyllite might be present in very small amounts (See Plates 7 and 8). It was identified on the basis of (1) parallel extinction, (2) extreme aspect ratio, and (3) lack of the length-parallel striations that characterize

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actinolite.

The data of Table 1 show that cummingtonite and anthophyllite can have densities as low as 2.85, and these would float with talc in the liquids used (density = 2.88). But these low densities are for amphiboles that are pure magnesium end-members, and as such, are mineralogical curiosities that are very rare in nature. It is likely that the usual compositions of anthophyllite and cummingtonite would be closer in density to actinolite. Because all amphiboles have similar surface chemistry, the separation techniques described here would almost certainly work for their concentration from talc and talc ore matrices. In fact some progress has been made to that end, though the results are preliminary and can only be described as promising.

CONCLUSIONS:

1. Mixtures of bromoform, methylene iodide, and benzethonium chloride monohydrate provide a suitable heavy liquid for the centrifugal separation of fiberform amphiboles from talc in samples composed of clay to silt-sized grains.
2. The ore sample contains 2300 ppm actinolite, and the talc product contains ~170 ppm actinolite.
3. Actinolite is the dominant fiberform amphibole

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in the ore and talc product provided by Windsor Minerals. Small amounts of anthophyllite may be present.

4. Calcium analyses on acid-treated heavy mineral residues serve as an effective means of determining actinolite.

5. The determination of crocidolite, cummingtonite, or anthophyllite in concentrates is probably best accomplished by a microscope method.

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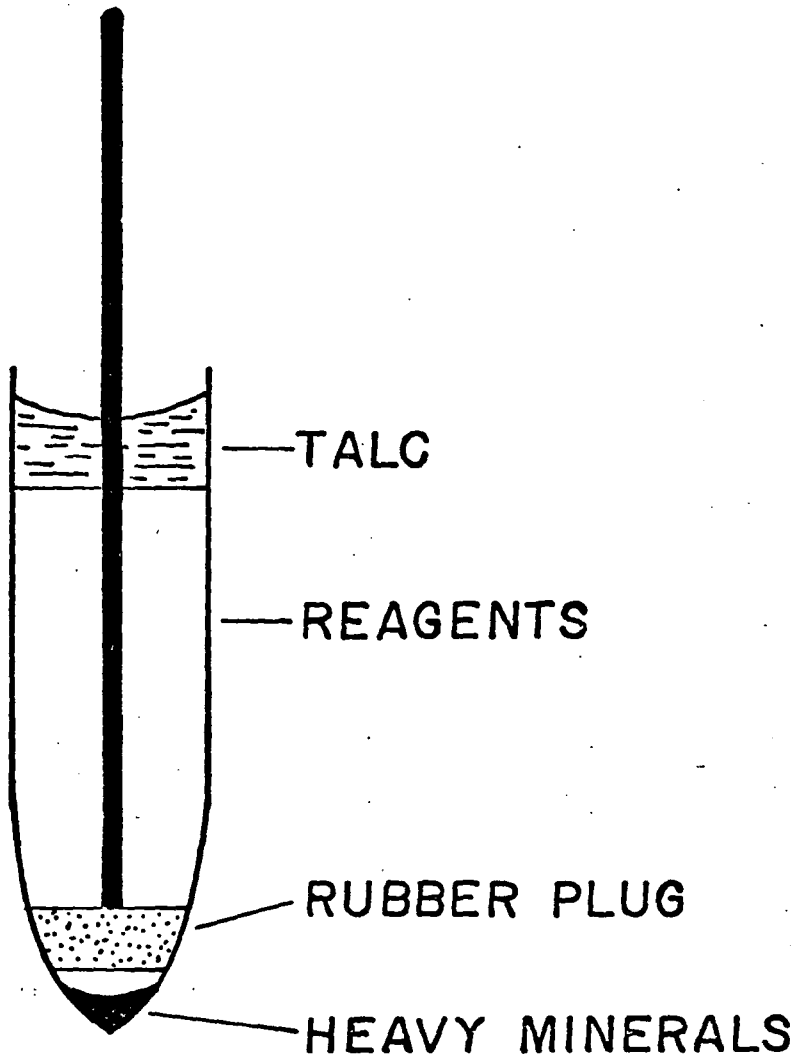


Figure 1. Apparatus for isolation of heavy minerals from centrifuge tube.

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TABLE 1 - Densities of Common Fiberform Amphiboles and Talc (Hurlbut, C.S., 1971, Dana's Manual of Mineralogy, 18th Ed., Wiley).

Mineral	Density
Talc	2.7-2.8
Tremolite-Actinolite	3.0-3.3
Cummingtonite	2.85-3.2
Anthophyllite	2.85-3.2
Crocidolite	3.2-3.3

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TABLE 2 - Residue Weights and Actinolite in Talc Ore and Actinolite-Spiked Talc Ore.

Sample	Heavy Minerals; mg/2g Sample	Acid Insoluble Heavy Minerals; mg/2g Sample	mg Actinolite found*	mg Actinolite nominal
Ore	478	14.3	4.6	---
Ore + 4.3 mg Actinolite	445	19.8	7.8	8.9
Ore + 8.6 mg Actinolite	458	20.4	9.8	13.2
Ore + 12.9 mg Actinolite	459	30.1	17.3	17.5

*Based on a nominal value of 9.7% Ca in actinolite,

$$Ca_2(Mg,Fe)_5Si_8O_{22}(OH)_2$$

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TABLE 3 - Heavy Fraction and Actinolite in Talc.

Sample	% Heavy Minerals (∿Carbonate)	PPM Actinolite
Talc Ore	23.9	2300
Talc Product	--	170

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APPENDIX

Photomicrographs have been taken of the minerals in talc and talc ores. Attempts have been made to catalog the dominant mineral species, and to depict the range in form characteristic of each mineral. For both talc product and talc ore, photos have been taken of (1) the bulk materials, (2) the heavy mineral fractions, and (3) the acid insoluble heavy mineral fractions. The photos below are accompanied by brief descriptions of each, and some mineral grains are appropriately labelled according to the following key.

Talc	T
Carbonate	C
Chromite	Cr
Actinolite	Ac
Anthophyllite	An
Epidote	Ep

Although the forms of the minerals shown on the photos are characteristic, many of the photos show non-representative mineralogical compositions. For example, a good deal of searching was required to find actinolite and carbonate grains in bulk talc products (Plates 14 and 11). Similarly, anthophyllite is extremely uncommon and considerable amounts of sample must be examined in order to find one grain (Plates 7 and 8).

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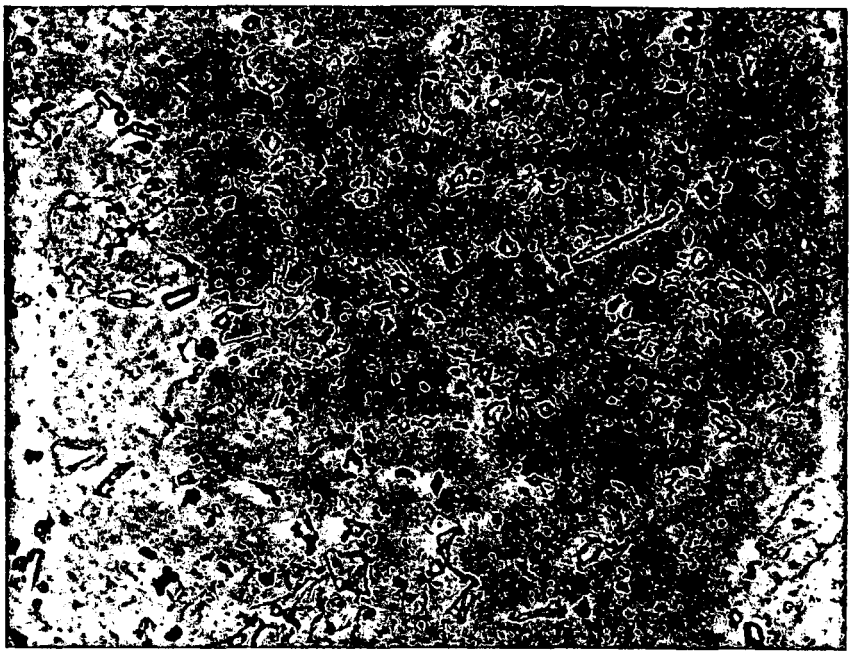


Plate 1

Talc Ore, Bulk; x 100; n=1.503
Low relief background is talc, high relief equant grains are carbonate, and the long grain in the upper right corner is actinolite.

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Plate 2

Talc Ore, Bulk; x 400; n = 1.503
Platy talc, actinolite, and carbonate.
Note the length-striated character of actinolite;
this is characteristic.

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Plate 3

Talc Ore, Bulk; x 400; n=1.503
Platy talc and carbonate. The talc is inclusion-free and shows a well-developed platy morphology.

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Plate 4

Talc Ore, Heavy Fraction; x 400; n = 1.503
The field shows epidote, carbonate, talc, chromite, and actinolite.

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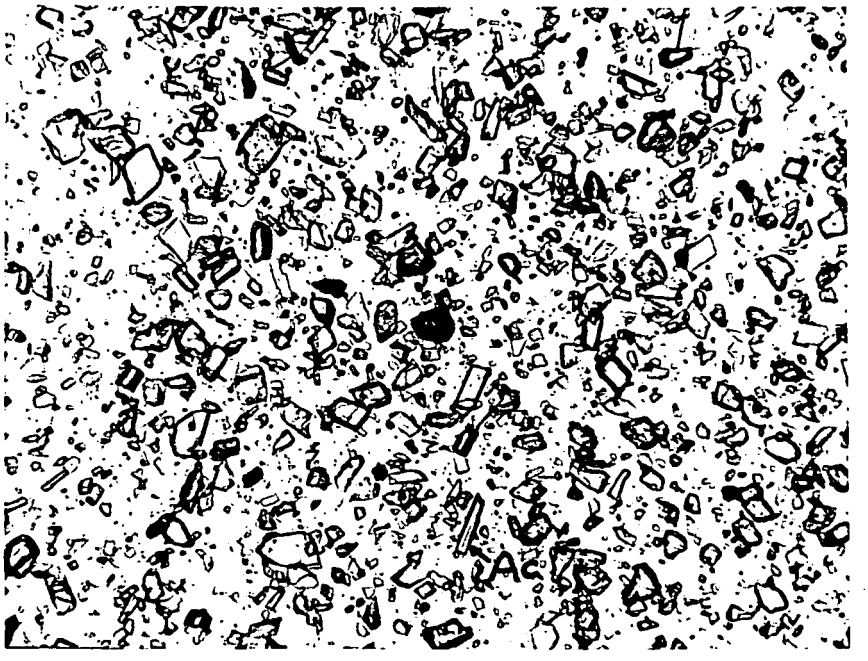


Plate 5

Talc Ore, Heavy Fraction; x 100; n = 1.503
This photo shows the general character of the typical heavy mineral fraction. Small amounts of talc are invisible in the background. Carbonate is the dominant mineral. Opaque grains are chromite, and actinolite is labelled Ac.

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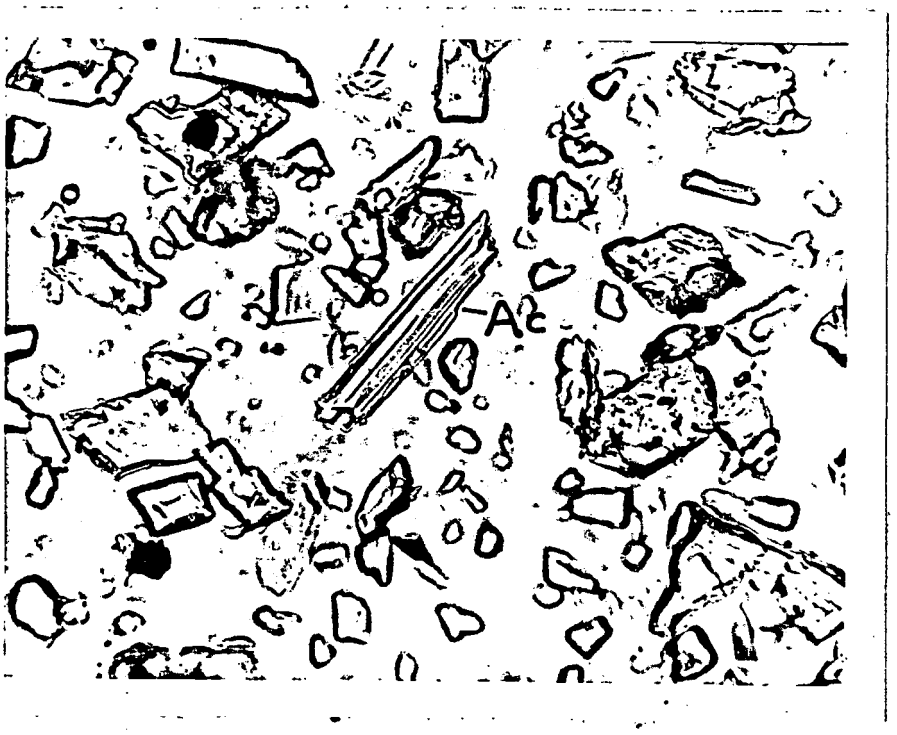


Plate 6

Talc Ore, Heavy Fraction; x 400; n = 1.503
This view shows mostly carbonate, some chromite (opaque) and an actinolite grain of typical morphology at the center.

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Plate 7

Talc Ore, Acid Insoluble Heavy Fraction; x 100; n = 1.503
Actinolite, talc, chromite, and a large anthophyllite fiber.
Note that much of the talc is of poor morphology and/or
is inclusion-filled.

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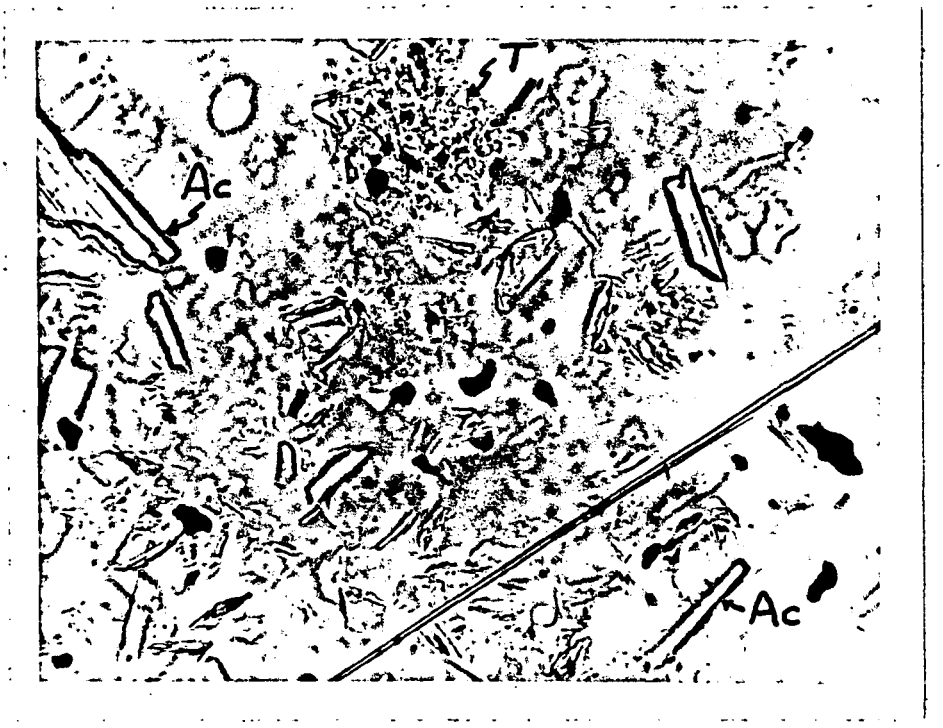


Plate 8

Talc Ore, Acid Insoluble Heavy Fraction; x 400; n = 1.503
Inclusion-filled talc, actinolite and anthophyllite.

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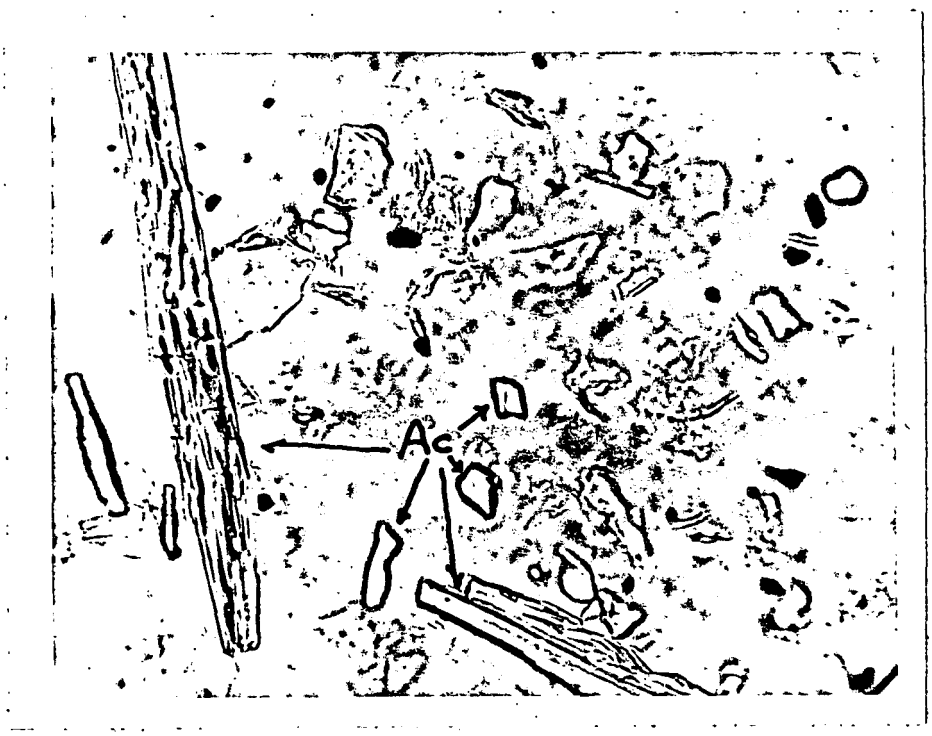


Plate 9

Talc Ore, Acid Insoluble Heavy Fraction; x 400; n = 1.503
Platy talc and actinolite. Note that the small equant grains of actinolite could be easily mistaken for carbonate.

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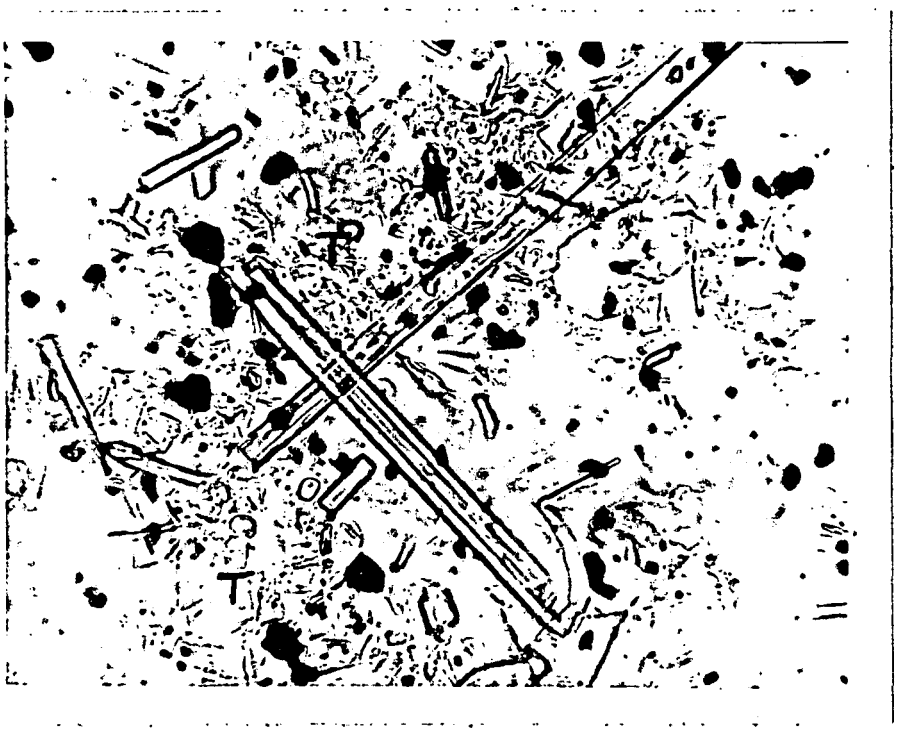


Plate 10

Talc Ore, Acid Insoluble Heavy Fraction; x 400; n = 1.503
Inclusion-filled talc with partial fibrous morphology, platy talc, and characteristic grains of actinolite.

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Plate 11

Talc Product, Bulk; x 100; n = 1.503
Platy talc with a few carbonate grains
(high relief).

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Plate 12

Talc Product, Bulk; x 400; n = 1.503
Typical platy talc of good morphology.

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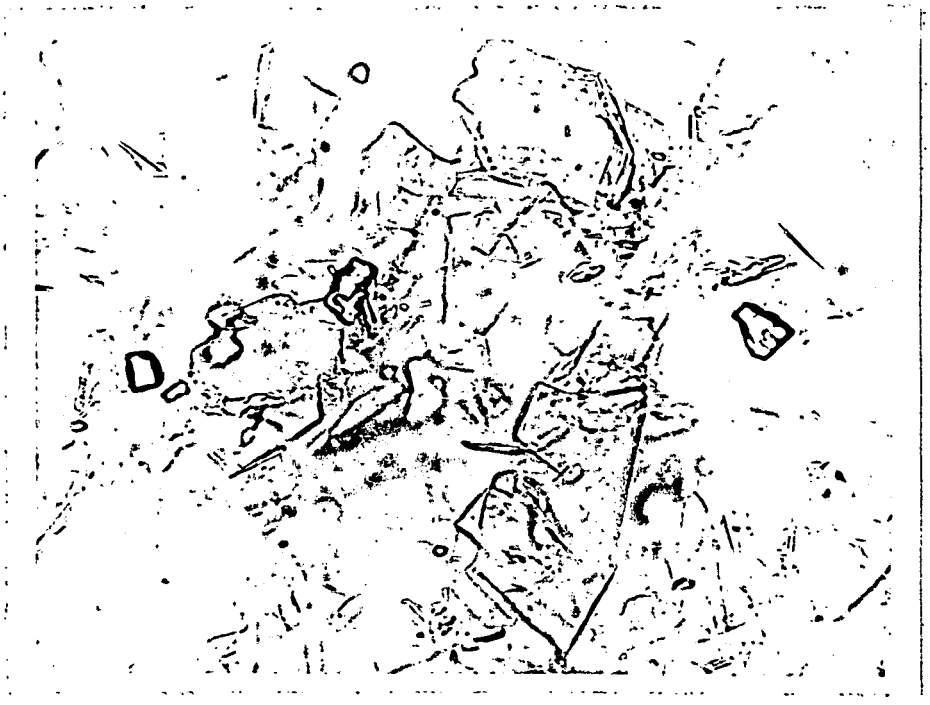


Plate 13

Talc Product, Bulk; x 400; n = 1.503
Platy talc and carbonate.

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Plate 14

Talc Product, Bulk; x 400; n = 1.503
Platy talc and one actinolite grain.

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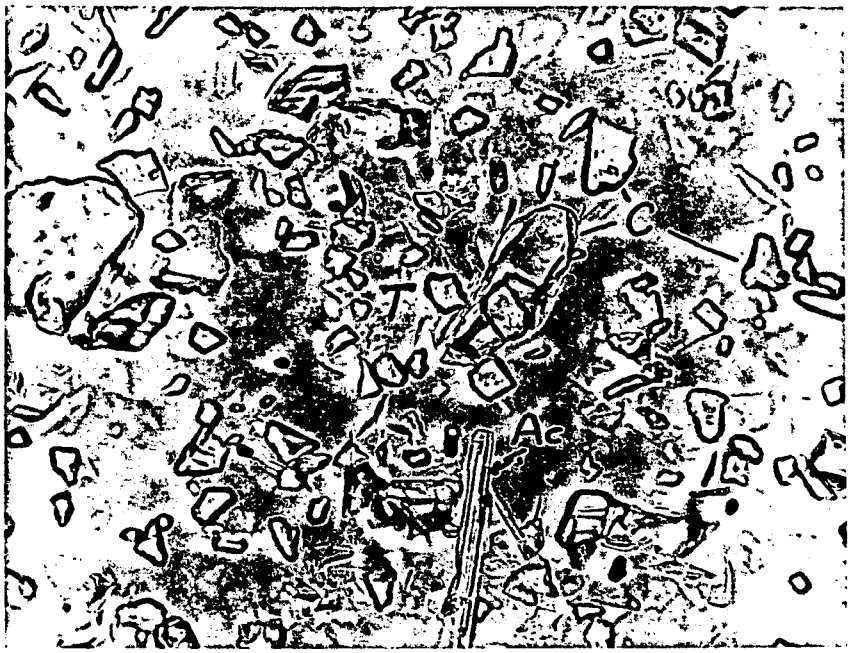


Plate 15

Talc Product, Heavy Fraction; x 400; n = 1.503
Typical field showing carbonate, talc, actinolite, and chromite.

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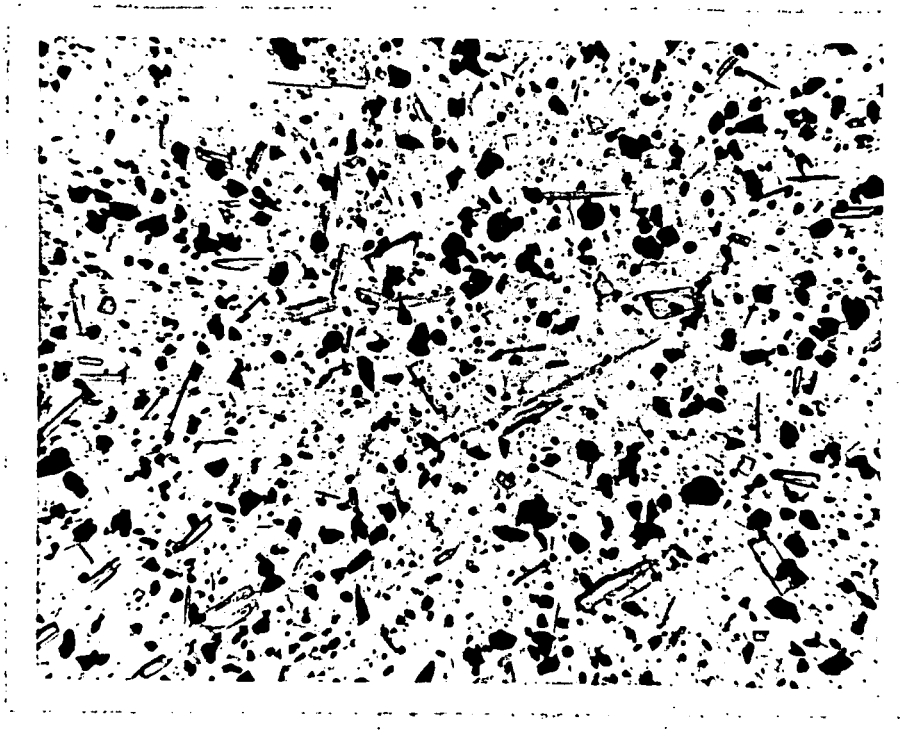


Plate 16

Talc Product, Acid Insoluble Heavy Fraction;
x 100; n = 1.571
Talc is invisible cecause of high index oil used.
Visible grains consist of chromite (opaque) and
actinolite. Note the large variation in aspect
ratio of the actinolite.

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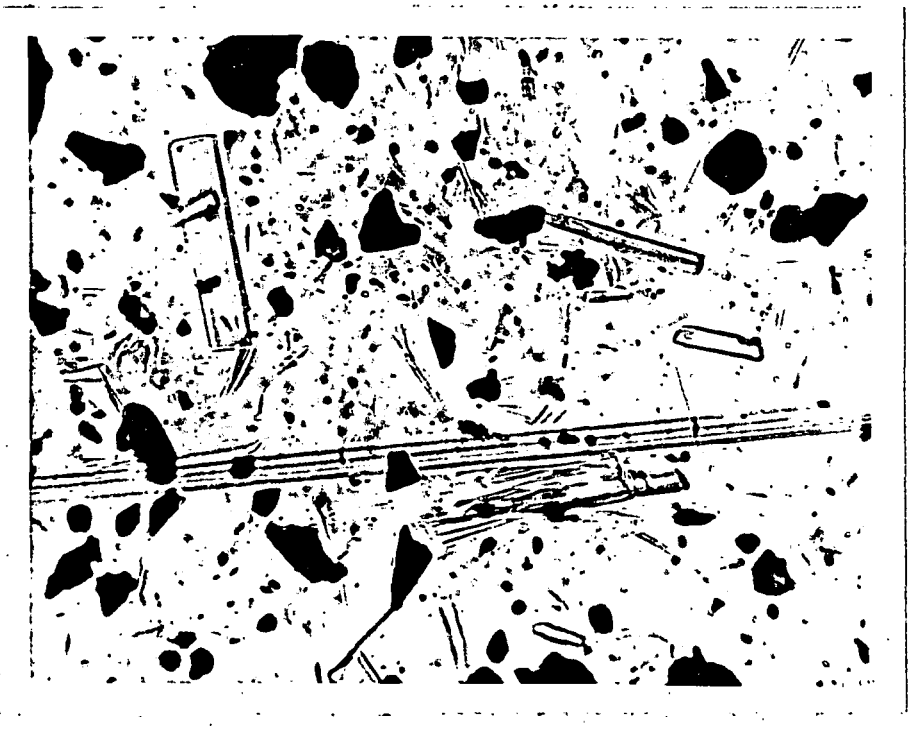


Plate 17

Talc Product, Acid Insoluble Heavy Fraction;
x 400; n = 1.571
Chromite and actinolite of varying morphology.
The characteristic striations are clearly visible
in the actinolite grains.

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Plate 18

Talc Product, Acid Insoluble Heavy Fraction;
x 400; n = 1.571
Large actinolite grain with irregular shape.

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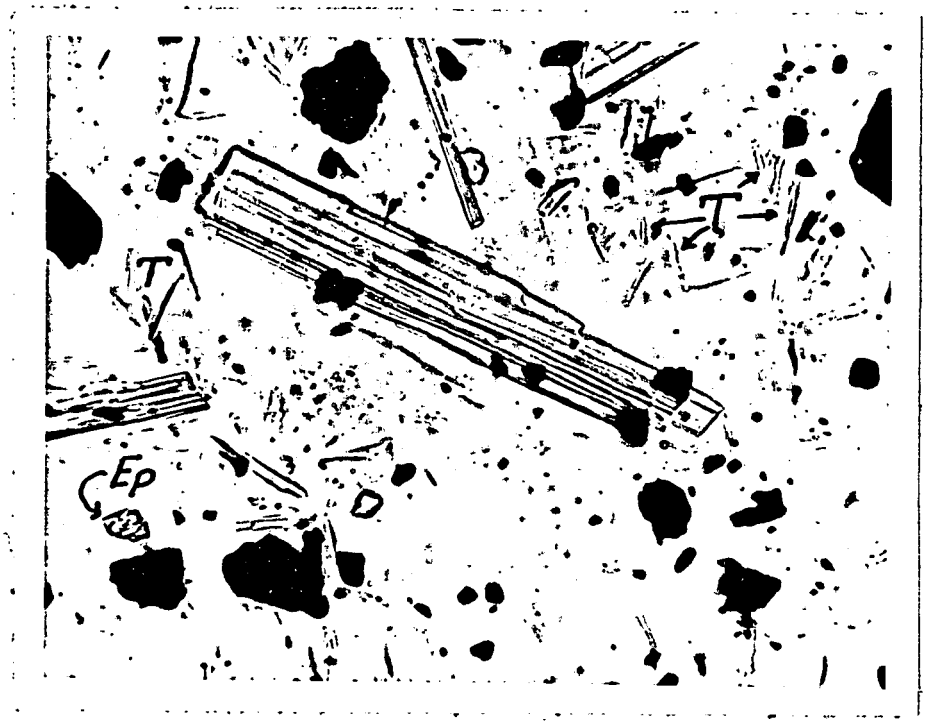


Plate 19

Talc Product, Acid Insoluble Heavy Fraction;
x 400; n = 1.571
Typical actinolite, fibrous talc, chromite and epidote.

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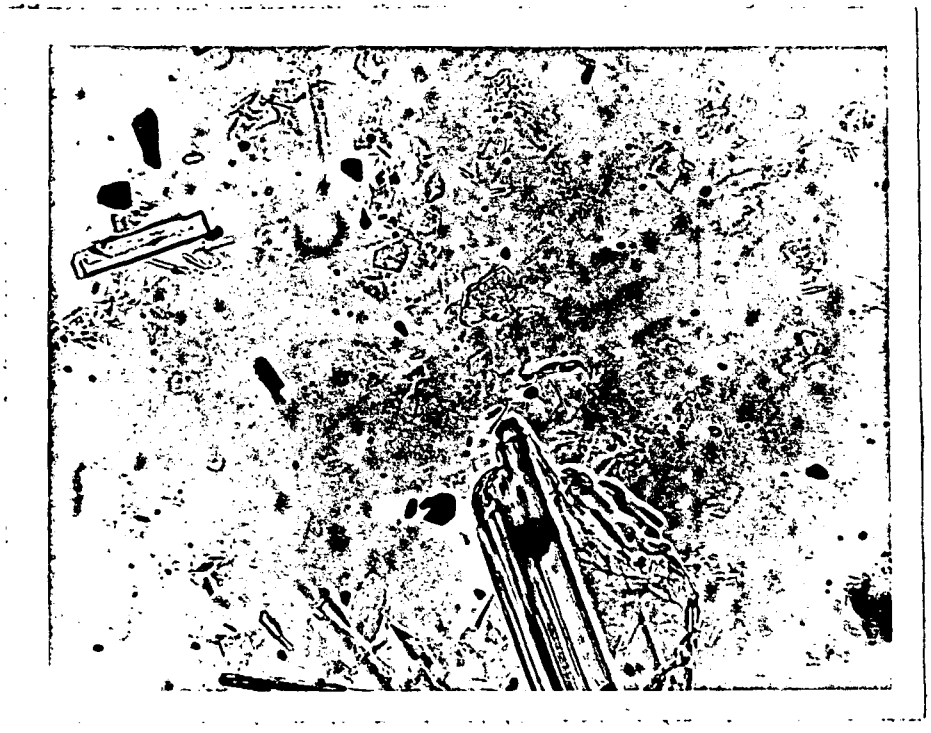


Plate 20

Talc Product, Acid Insoluble Heavy Fraction; x 400; n = 1.503
Note the large compound grain (platy talc and actinolite) at bottom center. Other minerals are platy talc (very low relief) actinolite, and chromite.

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Plate 21

Talc Product, Acid Insoluble Heavy Fraction;
x 400; n = 1.503
Small talc fibers, platy talc, and inclusion-filled talc plus chromite and actinolite. It is the presence of grains such as the inclusion-filled talc that makes grain counting analysis difficult.

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