

**MATERIALS
ANALYTICAL
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**Analysis Of Zonolite Vermiculite
Masonry Insulation**

Submitted to

J. Conard Metcalf

Williams & Trine

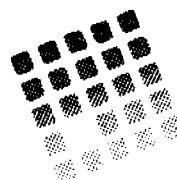
February 18, 1994

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MICROANALYSIS PROJECT REPORT



MATERIALS
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CLIENT: J. Conrad Metcalf
INSTITUTION: Williams & Trine
PROJECT: Zonolite Product Analysis
MAS NO. M10771

DATE: February 15, 1994

SUMMARY: *The objective of this study was to determine the presence or absence of tremolite/actinolite asbestos in Zonolite Masonry Block Fill that was manufactured by W.R. Grace.*

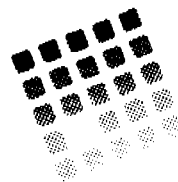
DESCRIPTION: In October of 1993, Materials Analytical Services, Inc. (MAS) received an unopened bag that was labeled "Masonry Block and Cavity Fill" that was manufactured by W.R. Grace (figure 1). In November of 1993, the Masonry Block Fill was retrieved from MAS archive storage room and analyzed by optical microscopy, scanning electron microscopy (SEM), and transmission electron microscopy (TEM).

PREPARATION AND ANALYSIS: The Zonolite bag was opened in a negative flow HEPA hood and a random sample of the vermiculite was removed for testing as shown in figures 2 and 3. The sample of the vermiculite was inspected with an Olympus SZ Zoom Stereomicroscope (figure 4) at magnifications ranging from 7X to 40X as shown in figures 5 through 7. In one of the vermiculite pellets, a fibrous gray-green mineral was observed associated with the vermiculite material, as shown by the arrows on the photomicrographs in figures 6 and 7. The vermiculite pellet containing the suspected tremolite-actinolite was then prepared for SEM analysis. Additionally, the fine vermiculite dust fraction was prepared for both polarized light microscopy (PLM) and TEM analysis.

The SEM preparation was performed by placing the associated fibrous mineral onto a SEM stub with double-sided carbon tape and carbon paint (figure 8). The sample was then carbon-coated in a Hitachi Vacuum Evaporator. The SEM analysis was performed using a Hitachi S-800 Field Emission SEM with a Tracor Northern EDXA system (figure 9). The SEM photomicrographs shown in figures 10 through 13 further show the tremolite/actinolite intergrowth in between the vermiculite leaves for this particular sample. The EDXA spectra of the mineral was consistent with tremolite-actinolite (figure 14). Both asbestiform and non-asbestiform tremolite-actinolite were observed by the SEM.

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The identity of the mineral was further verified using PLM (figure 15). The refractive indices of the individual fibers as well as other optical and morphological characteristics identified the material as tremolite-actinolite (figures 16 and 17).

The TEM preparation of the vermiculite sample was performed by suspending between 10 - 30 mg of the fine vermiculite dust fraction in reagent alcohol that was then filtered onto a 0.2 um polycarbonate (PC) filter. The filter was dried, carbon coated, and a section of the filter was placed on a 200 mesh TEM grid.

The PC filter was dissolved in a chloroform Jaffe washer and the final TEM prep examined in a JEOL1200EX II TEM (figure 18). The TEM photomicrographs in figures 19 through 20 show that the tremolite-actinolite structures that were found had an aspect ratio that was greater than 3:1, and can therefore be classified as asbestiform structures as specified by current Federal regulations. A Tracor Northern system was used to obtain EDXA spectra, as shown in figure 21, and a selected area electron diffraction (SAED) pattern was used to further positively identify the tremolite-actinolite asbestos.

DISCUSSION AND CONCLUSION: It is well known that in some areas of the country where vermiculite deposits are found, tremolite-actinolite can also be found as an associated mineral. However, there has been some suggestion that during the processing of the vermiculite ore at the manufacturing plant, the asbestiform tremolite-actinolite is somehow removed, and the final vermiculite product sold to the consumer is tremolite-actinolite free.

This study was designed to test that theory by taking a known sample of a processed vermiculite product (Zonolite Vermiculite Masonry Insulation as manufactured by W.R. Grace), and determining if it contained asbestiform tremolite-actinolite.

Our analysis showed that both asbestiform and non-asbestiform tremolite-actinolite could be found in with the vermiculite from the W.R. Grace product. The tremolite-actinolite was found both associated with the vermiculite and as free respirable fibers in the fine dust.

It is therefore our conclusion that asbestiform tremolite-actinolite does remain associated with the vermiculite after the ore is fully processed and can be found with the finished product. This was demonstrated with the W.R. Grace Zonolite Vermiculite Masonry Insulation material.

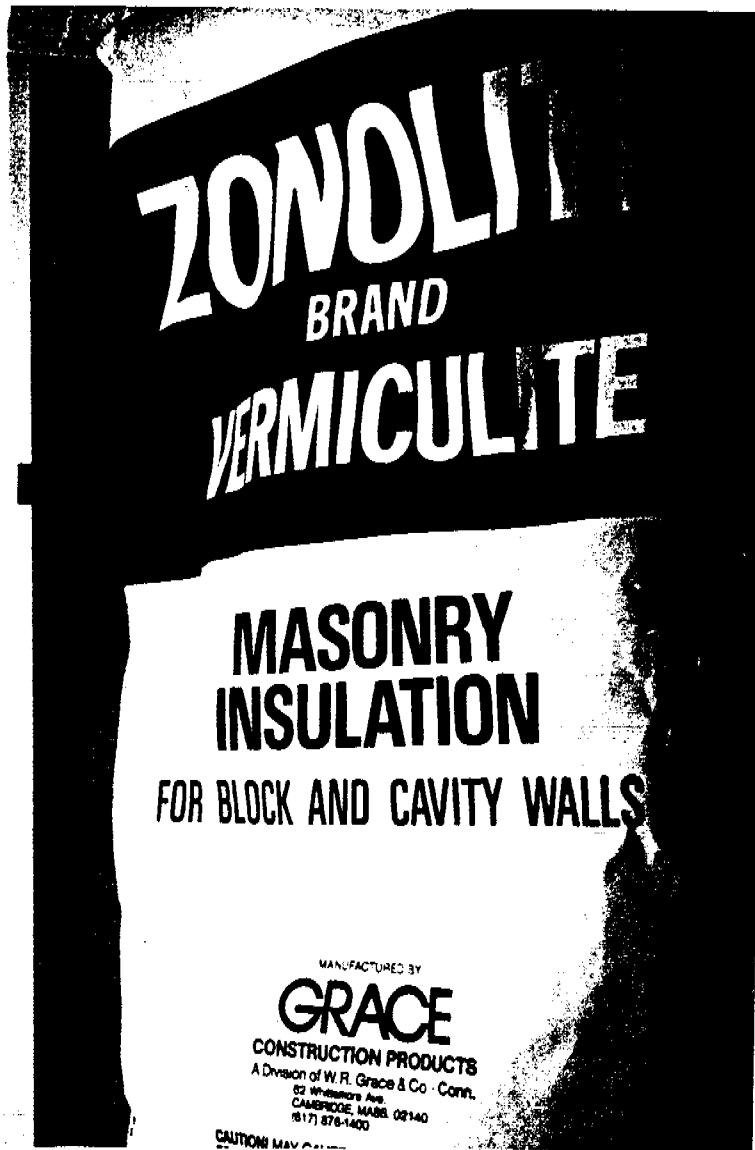


FIGURE 1

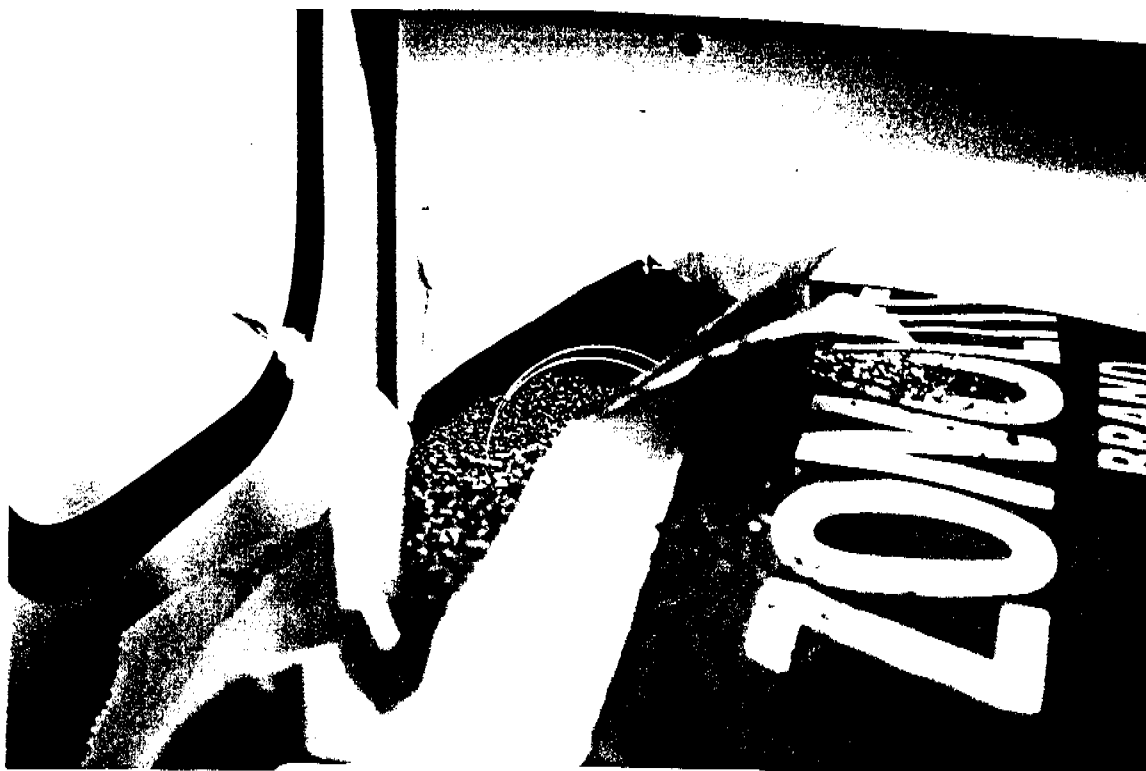


FIGURE 2



FIGURE 3

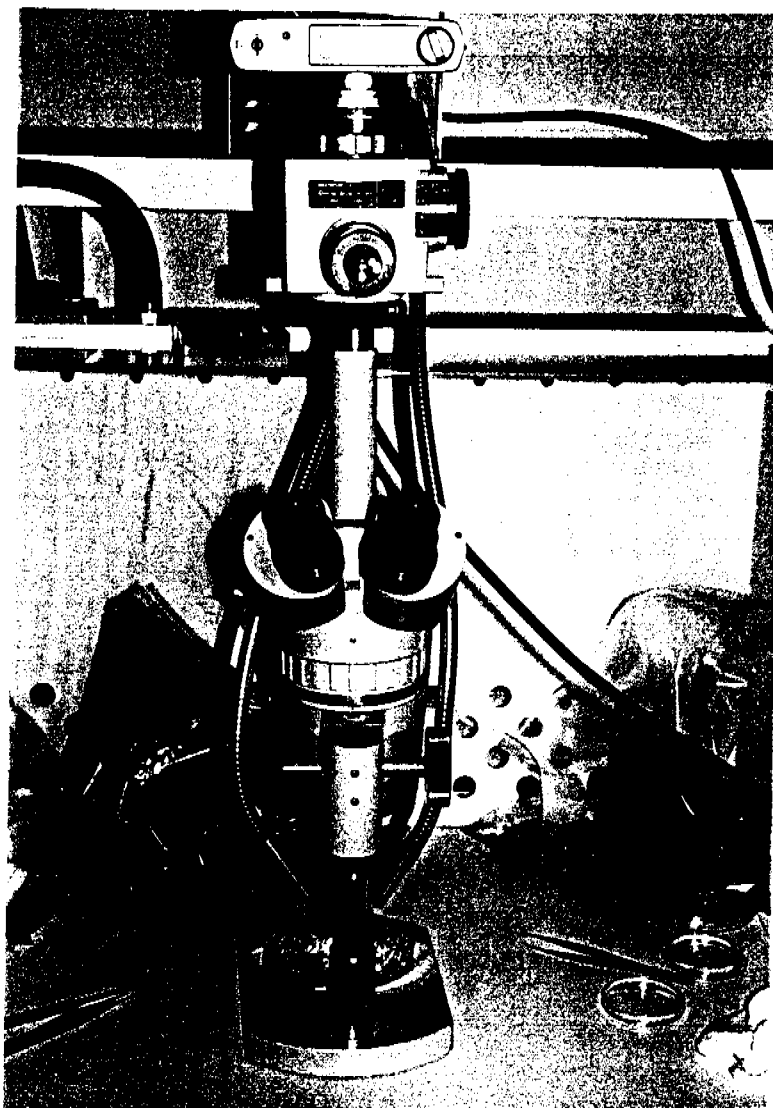


FIGURE 4

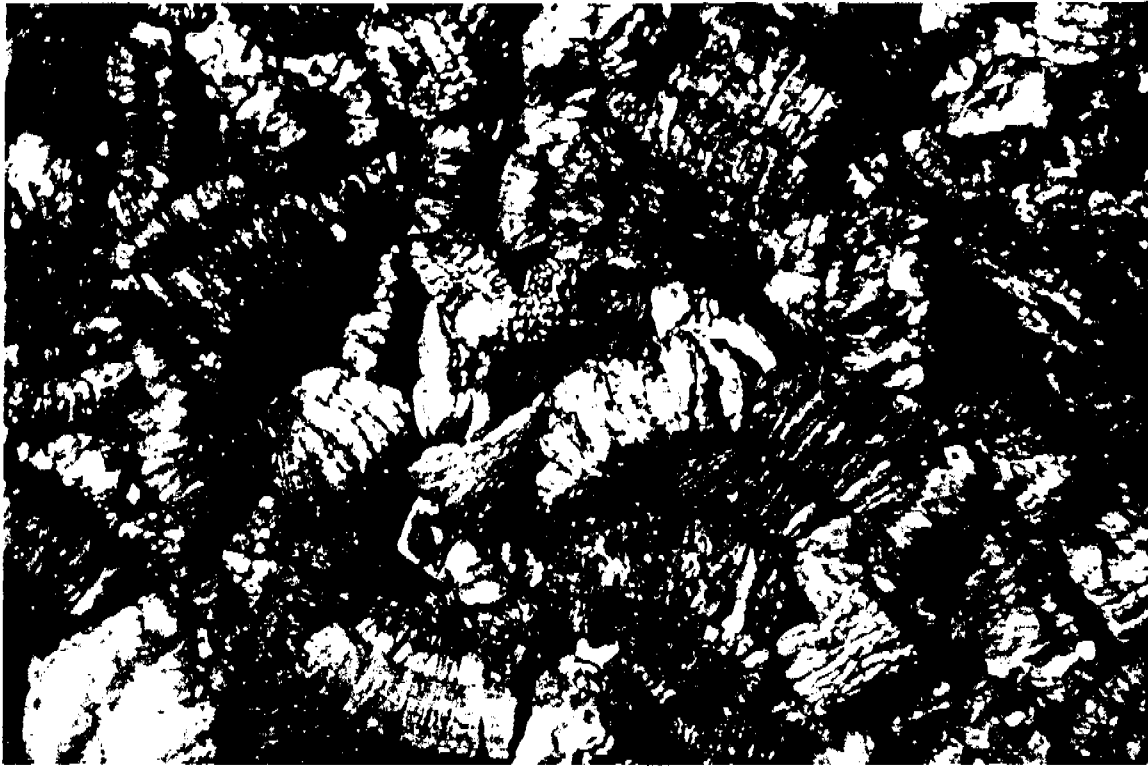


FIGURE 5



FIGURE 6

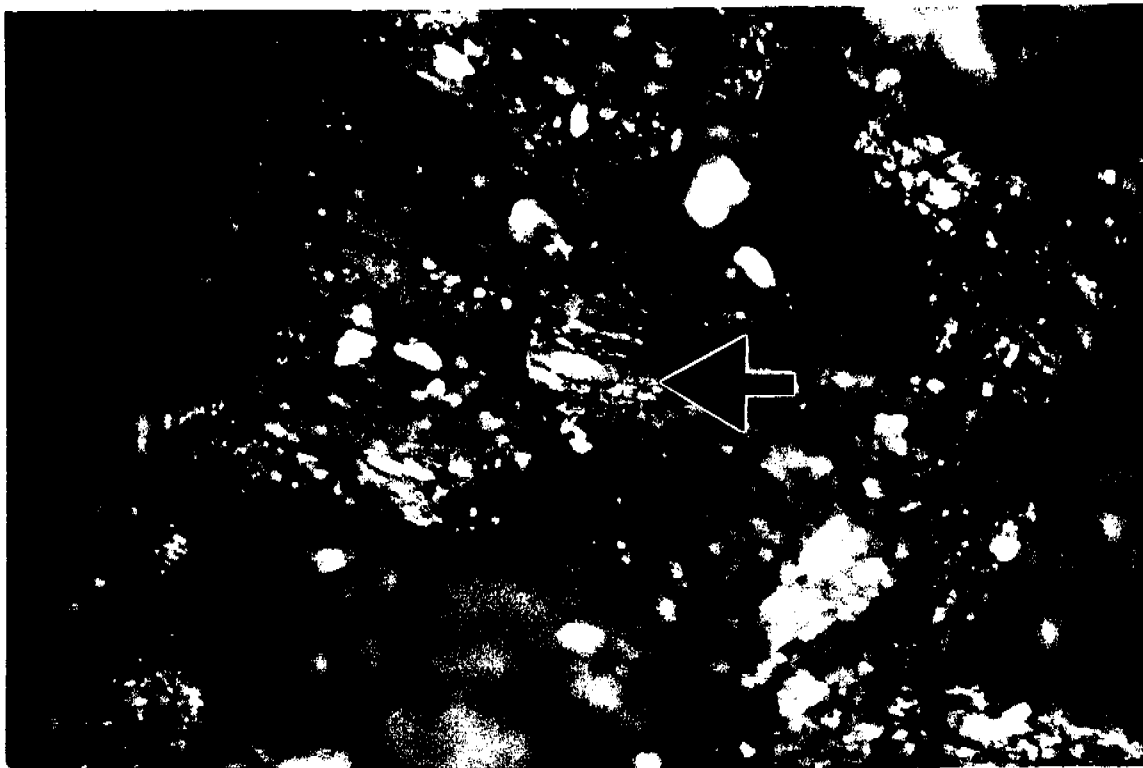


FIGURE 7

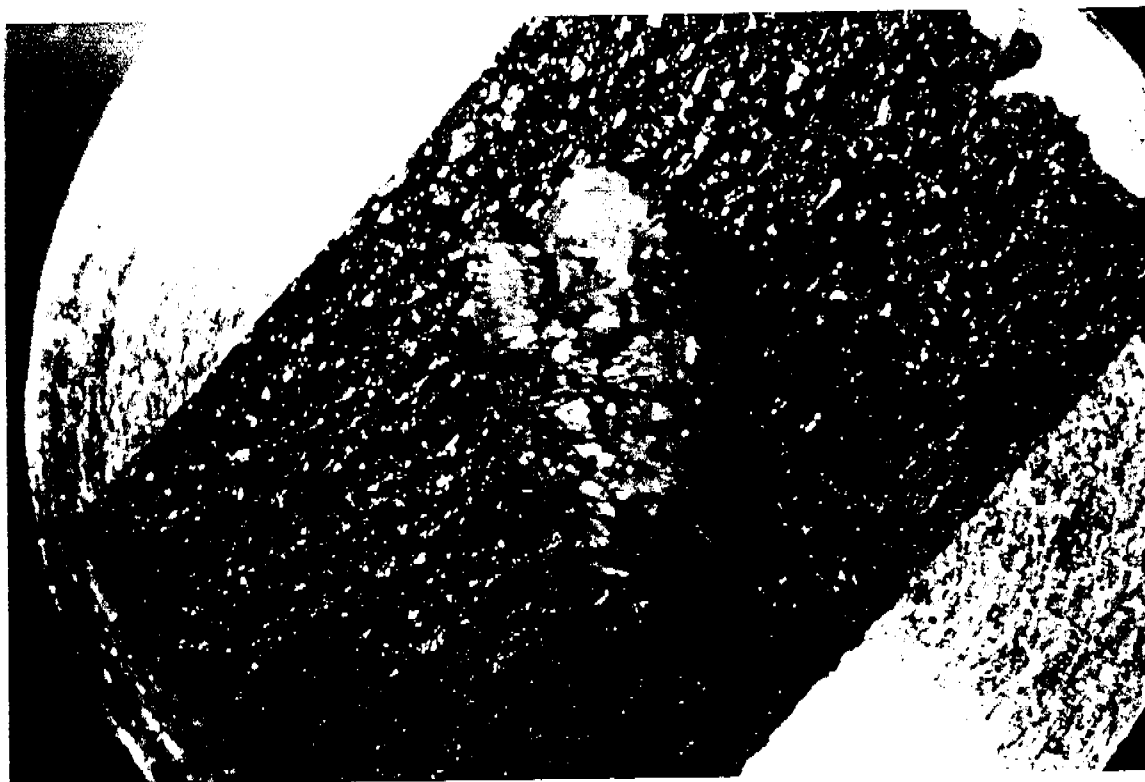


FIGURE 8

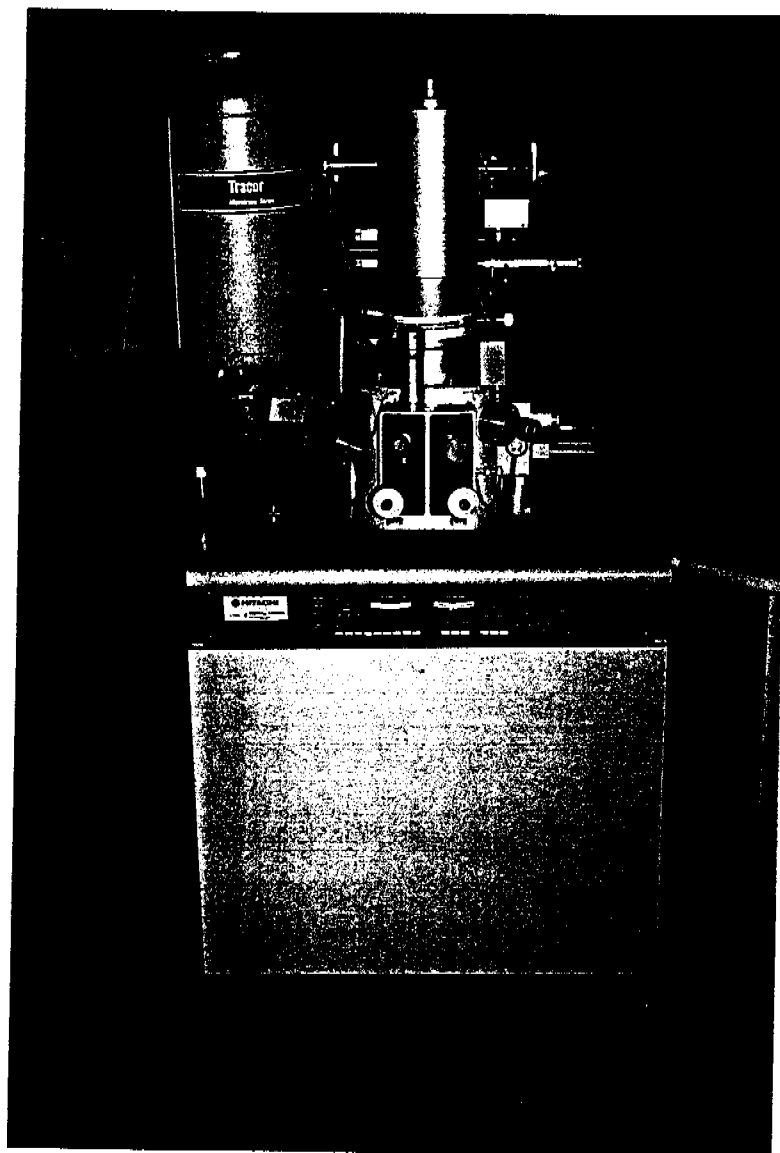


FIGURE 9



FIGURE 10



FIGURE 11



FIGURE 12



FIGURE 13

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Cursor: 0.000keV = 0

ROI (SIK α) 1.660: 1.820=99556

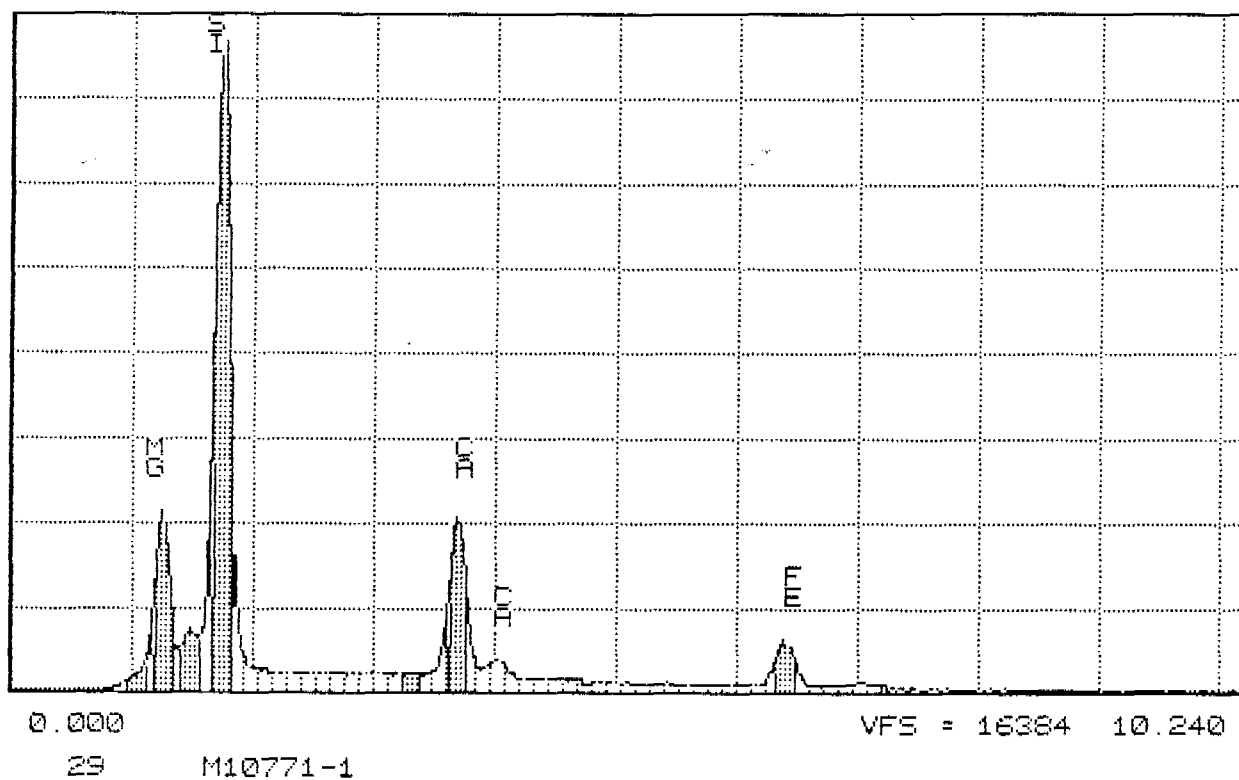


FIGURE 14

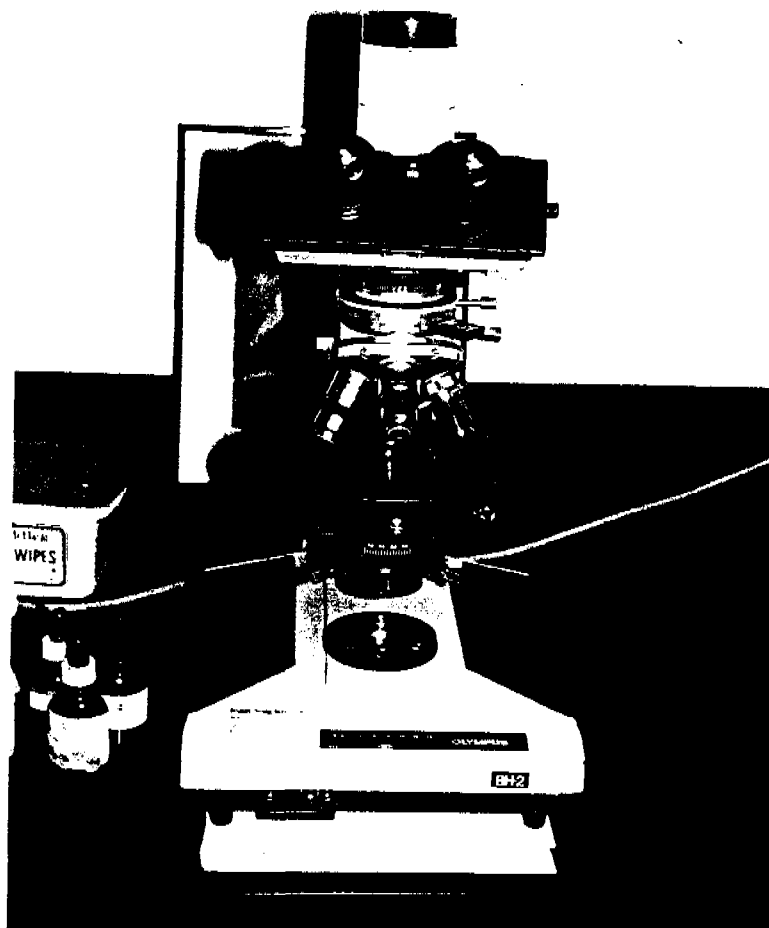


FIGURE 15



FIGURE 16



FIGURE 17

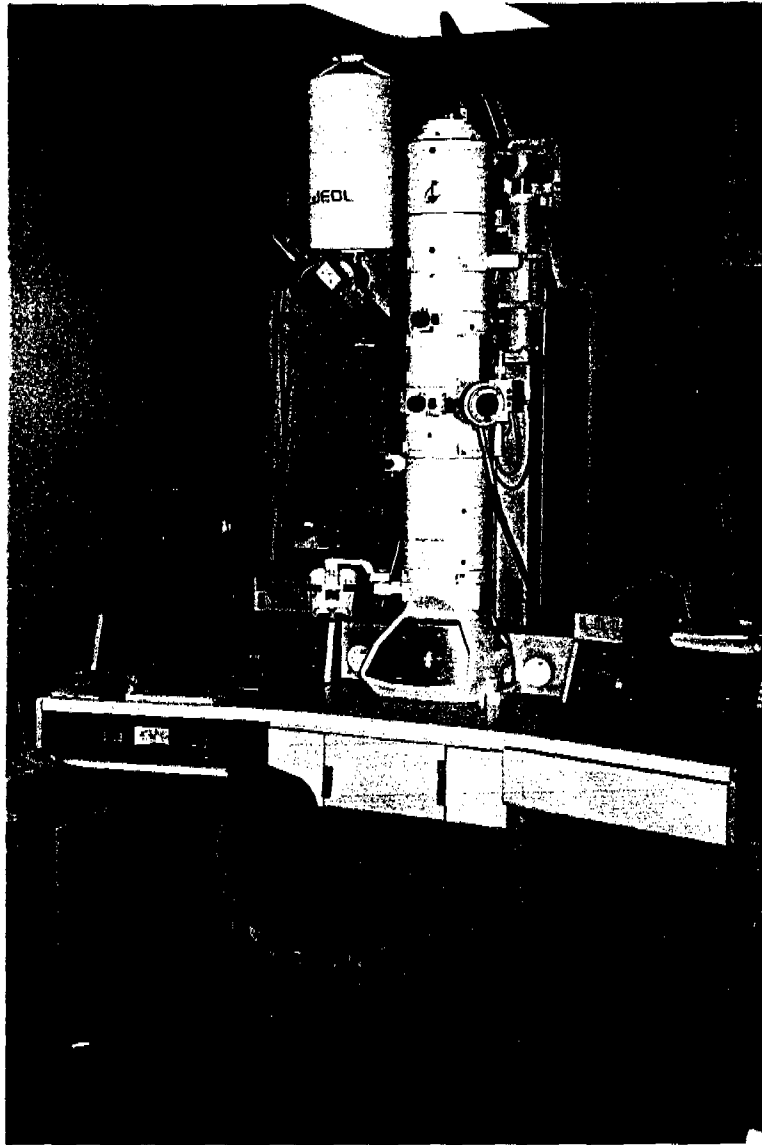


FIGURE 18

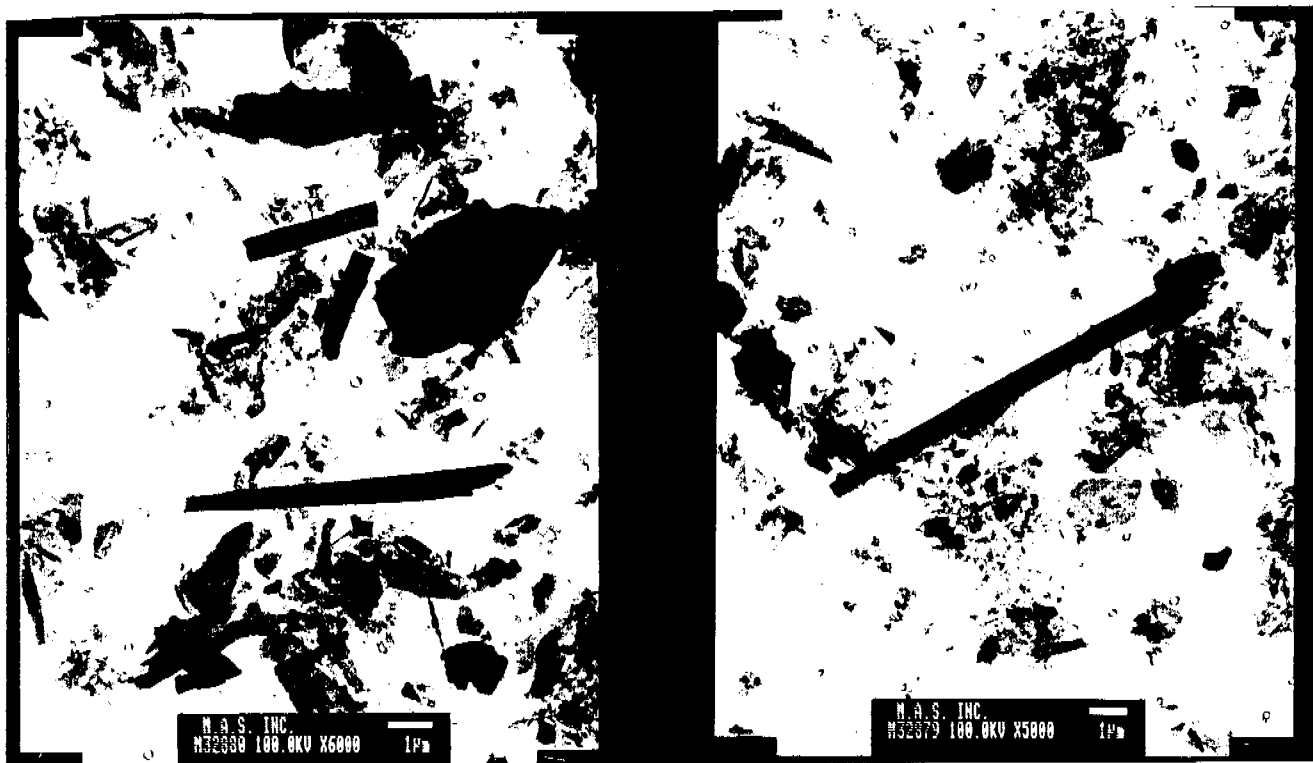


FIGURE 19

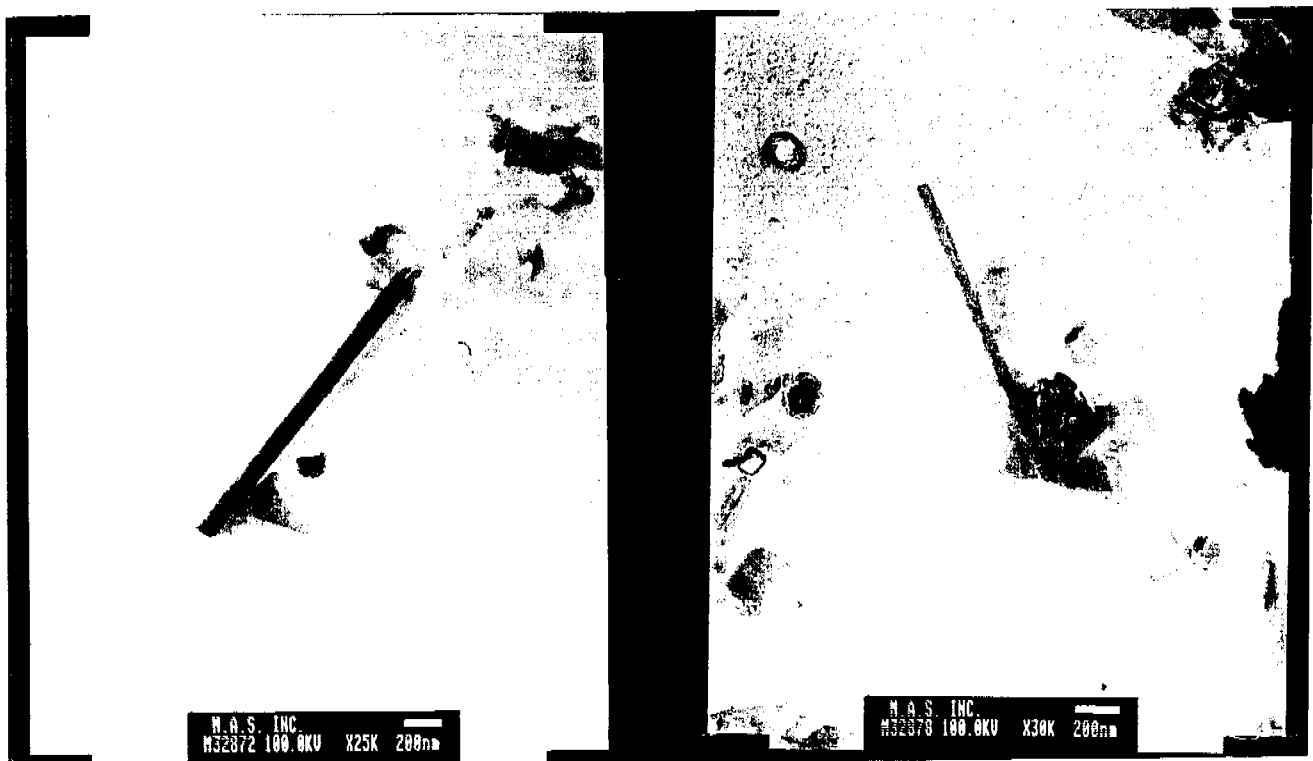


FIGURE 20

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ROI (SIK α) 1.660: 1.810=1061

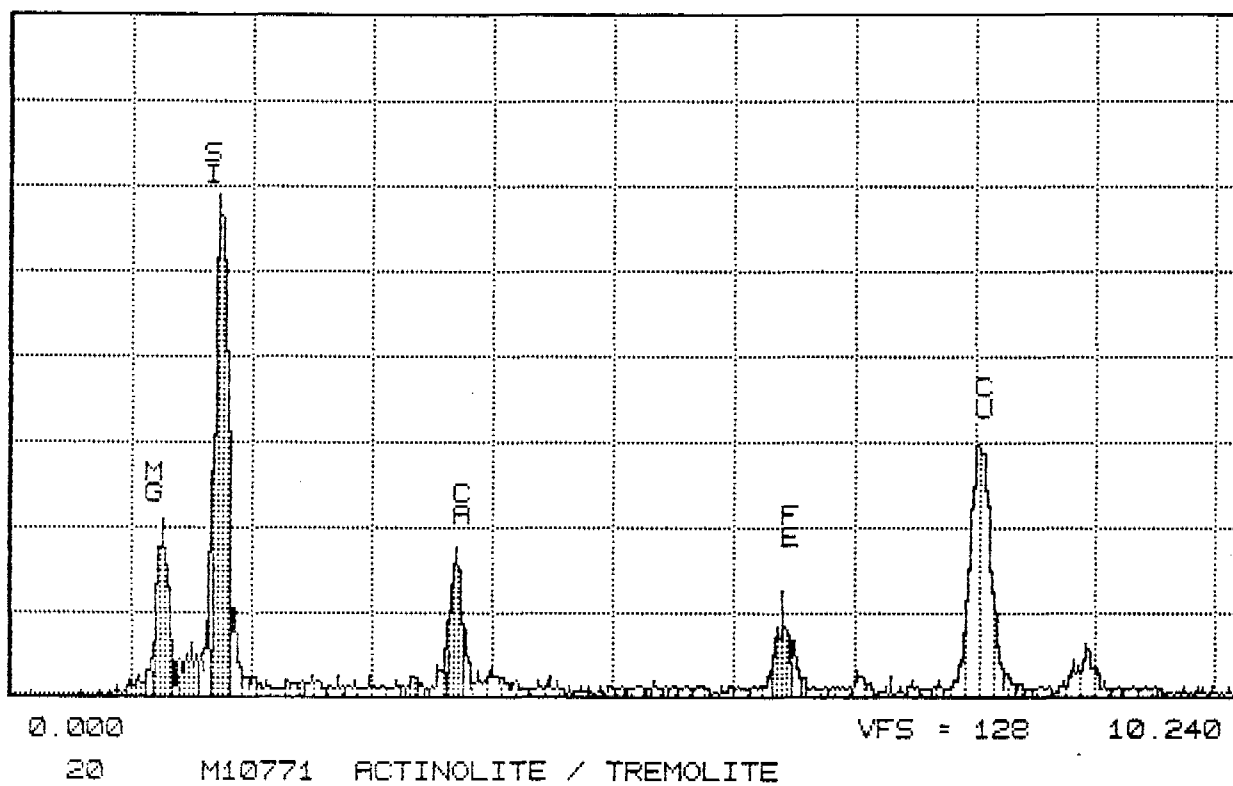


FIGURE 21