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FINAL REPORT

INDUSTRIAL HYGIENE STUDY OF THE GOUVERNEUR TALC COMPANY, NUMBER ONE MINE AND MILL

Balmat, New York

Volume II
Talc Bulk Sample Analyses
By NIOSH, W.C. McCrone and
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Center for Disease Control
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Electron Microscopic Analyses of R.T. Vanderbilt Talcs Collected from Talc Suppliers

Analyses Performed By

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Date
October, 2976

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INTRODUCTION

As a part of ongoing research by the National Institute for Occupational Safety and Health (NIOSH) concerning health effects of occupational exposures to talc and as requested by the Occupational Safety and Health Administration, seven bulk talcs produced by the R.T. Vanderbilt were analyzed for asbestos content. These talcs were obtained from independent talc suppliers and have been "certified" by Vanderbilt not to contain asbestos. Samples were analysed for asbestos content by analytical electron microscopy. The following paragraphs describe sample preparation and analytical methods employed along with results.

METHODS

Given in Table 1 are descriptive data for the 7 talcs obtained and analyzed including source and NIOSH sample number assigned.

Samples for transmission electron microscopic analyses were prepared by dispersing the powder samples in ethyl acetate by ultrasoneration. Approximately 10 mg of each powder was placed in 10-20 ml. of ethyl acetate followed by vigorous ultrasoneration for 10-15 minutes using a cell disrupter. Samples were prepared using a micro capillary tube to place a drop of the solution onto 200 mesh Formvar/carbon substrate copper electron microscope grids. The ethyl acetate was allowed to evaporate in a laboratory clean hood. If necessary, an additional drop of the solution was used to insure sufficient material for efficient analysis by electron microscopy. Blank grids were prepared in the same manner as the samples.

Samples were analyzed by first scanning the entire grid at low magnification to ascertain the suitability of the preparation for analysis. At a magnification of approximately 17,000X, all particles with an aspect ratio (length to diameter) of 3 to 1 or greater were identified using both selected area electron diffraction and energy dispersive microchemical analysis for fiber identification. A total of 25 to 50 individual fibers were so analyzed for each sample. A JEOL, JEM 100B transmission/scanning electron microscope equipped with an EDAX energy dispersive x-ray analyzer was used for all analyses. Electron micrographs of typical fibers along with their diffraction patterns and x-ray spectrum were recorded for each sample.

Table i

Sources of R.T. Vanderbilt Talcs Produced at the Gouverneur Talc Company, Number One Mine and Mill and Obtained from Suppliers

Nytal 300 Nytal 400	Crone Chemical P.O. Box 14042 Houston, Texas 77021	001 002
5x 325 X FT 3x	Paul Crazier Company 1115 Silver St. Houston, Texas 77067	003 004 005 006 007

RESULTS AND DISCUSSION

Results of all analyses are shown in Table 2 and typical micrographs of fibers in these samples along with electron diffraction patterns and x-ray spectra are shown in the Appendix. Based on their selected area electron diffraction patterns, fibers were classified as positive amphiboles, positive chrysotile, non-asbestos or "ambiguous" meaning that the pattern was not sufficiently clear for positive identification. The energy dispersive x-ray spectra were used to classify the amphiboles as to type and for further confirmation of chrysotile identification by electron diffraction.

As shown in Table 2, results of these analyses show all talcs analyzed to be of essentially the same fiber composition. The major fiber component is asbestiform anthophyllite, ranging from 67 to 88% of the fibers present in addition to fibrous tremolite (4 to 12%). Aspect ratios for these fibers ranged up to 1000 to 1, with the longer, thinner fibers being anthophyllite. Trace quantities of chrysotile were found in two of the samples. Chrysotile fibers observed were small in diameter (< 0.1 μm) and short in length (most < 1.0 μm).

Table 2

Summary of NIOSH Electron Microscopic
Analyses of Talc Produced at the
Gouverneur Talc Company
Number One Mine and Mill
Samples Obtained From Talc Suppliers

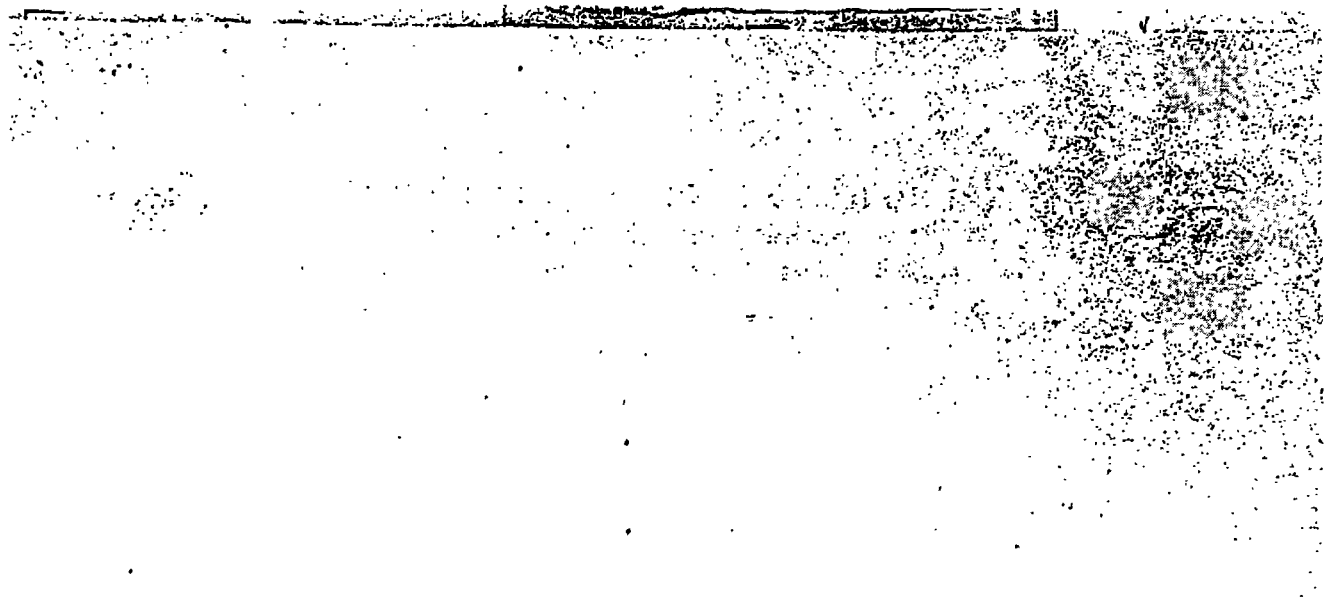
Talc Sample	Range of Fiber. Aspect Ratios	Fiber Identification (Percent)		
		Positive Amphiboles		Positive Chrysotile
		Tremolite	Anthophyllite	
Nytal 400	3/1 to 1000/1	8	88	N.D.
Nytal 300	3/1 to 100/1	12	72	N.D.
5x	5/1 to 100/1	11	80	Trace
X	8/1 to 80/1	6	80	N.D.
3x	5/1 to 100/1	12	67	N.D.
FT	3/1 to 50/1	16	72	N.D.
325	3/1 to 50/1	4	88	Trace

* Selected area diffraction patterns not sufficient for positive identification

ND - None Detected

CONCLUSIONS

Using present "state-of-the art" techniques for asbestos fiber analyses all 7 talcs analyzed demonstrated large quantities of asbestiform tremolite and anthophyllite to present. Only trace quantities of chrysotile were detected in 2 of the 7 talcs. Based on these analyses, all talcs analyzed should be labeled with the OSHA asbestos warning label.



APPENDIX

Electron Micrographs, Electron Diffraction Patterns and X-Kay Specta for Typical Fibers in R.T. Vanderbilt Talcs.

MOUNT SINAI SCHOOL OF MEDICINE
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Department of Community Medicine

April 29 '1976

Mr. John Dement
NIOSH Room 527
Post Office Building
Fifth and Walnut Street
Cincinnati, Ohio 45202

Dear Mr. Dement:

This report covers the results of analyses of seven NYTAL samples; 400, 35, X, FT, 300. 325 and 5X.

The seven samples were analyzed quantitativelp for three varieties of asbestos minerals - chrysotile, anthophyllite and tremolite and also for quartz. Analytical techniques included polarized optical microscopy, x-ray diffraction, transmission electron microscopy and scanning electron microscopy with energy dispersive analytical systems.

Quantitative determinations by x-ray diffraction of the four minerals in question were made by comparison with dilution standards of these minerals. A uniform method of back mounting in which the sample is remounted and re-run four times at identical instrumental settings over diagnostic reflections has been shown to be reproducible and accurate. Quantitation of these minerals was also done by x-ray diffraction in the step-scan, fixed count mode.

Integrated intensities for diagnostic x-ray reflections were measured using both the back-mounting and step-scan techniques. When referred to calibration curves prepared by each technique, the NYTAL unknowns showed egreement, averaging about 10% standard error. The resnlts are 2s follows:

	SERPENTINE PHASE	TREMOLITE	ANTHOPHYLLITE	QUARTZ
	%	%	%	%
NYTAL 400	14-18	24	2-3	7-10
" 3x	31-35	32	not detected	7-8
" X	26-30	50-60	not determined	9-10
" FT	26-30	17	not detected	not detected
" 300	14-18	18	4-5	9-10
" 325	19-23	24	5-7	3-4
" 5x	14-18	27	7-9	8-9

Because of the large amounts of tremolite (50-60%) present in sample X anthophyllite could not be quantitatively determined by x-ray diffraction. However, micro-

[redacted]

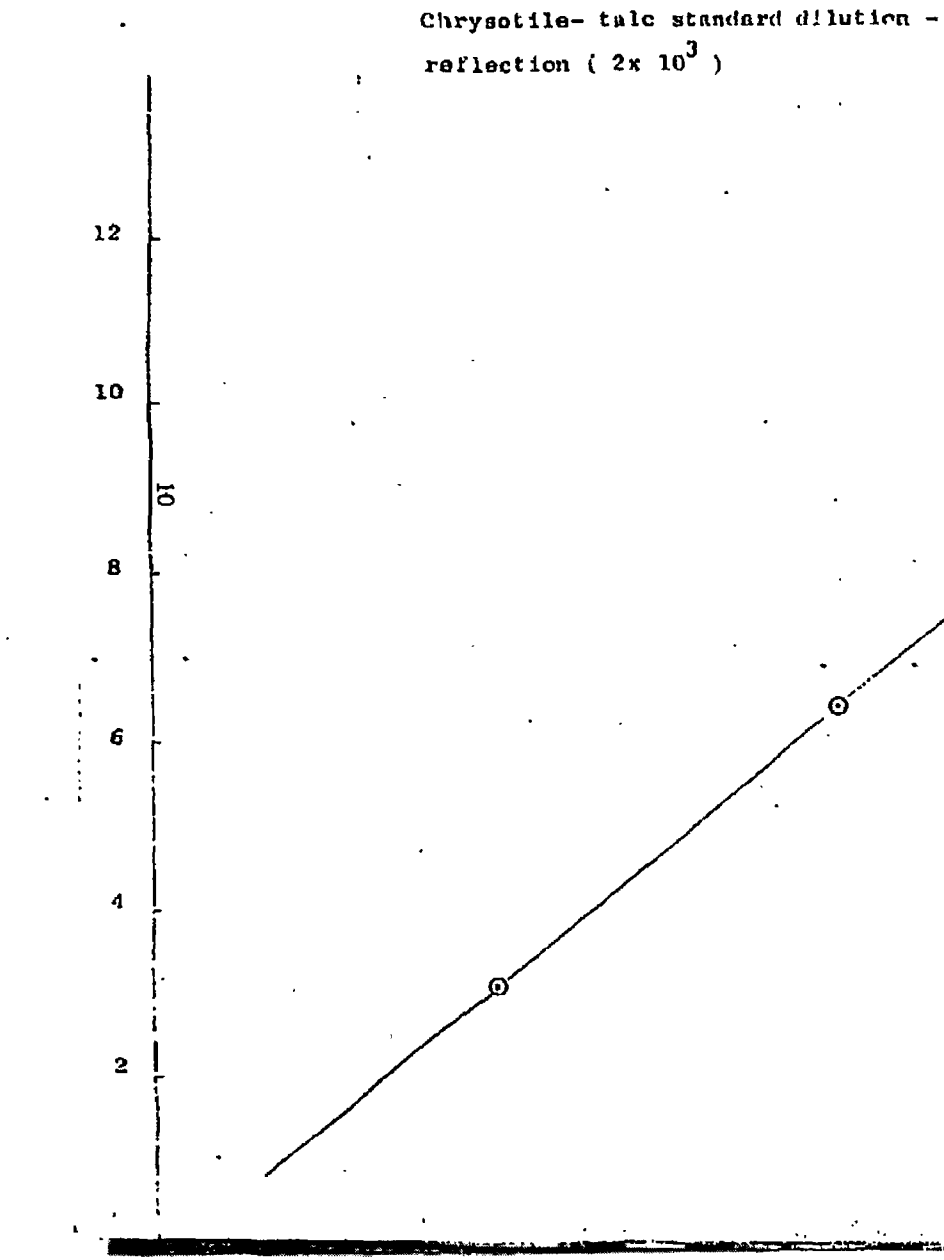
:nt

.ysis of fibers on the SEM shdws that anthophyllite is present. X-ray
.ndicated the presence of serpentine phases in all samples, but the
:ral species of this group which includes chryaotile, antigorite and
)uld not be positively identified although the latter most closely
·ray patterns. Examination by TEM and SEM with energy dispersive
em confirmed thst virtually all of the sperpentine phase consists
Chrysotile was detected by electron microscopy in the samples,
:ying photomicrographs).

·er: prepared for analysis by transmission electron microscopy by
·chnique which did not alter the size distributiin of particles. Two
three EM grids were scanned at 25,000x magnification on a Hitachi
:cope. Fibrous particles were abundant in all seven samples. Morphol-
·ystal cleavage charccteristics indicated the presence of amphibvles.
fied by observing a typical layer-type selected area electron diffrac-
on the fibrous particles. The layer line distance normal to the
:is was consistently observed to be 5.3Å, which is characteristic of
:peat of amphiboles. These particles were defined as amphibole and
n on a grid facsimile recorded. The specimens were transferred to
ectron microscope (CWIKSCAN) equipped with an energy dispersive x-ray
The amphibole particles were analyzed by point count and were found
two general morphological-chemical types of populations which permits
·fined as different mineral species, The long, thin fibers (aspect
· more) showed Si/Mg in ratios consistent with anthophyllite composi-
fibers contained little or no iron. Short prismatic fibers with
. from about 3/1 to 5/1 contained Si/Ca/Mg proportions consistent
e. No iron was found in tremolite.

Respectfully submitted,

Arthur S. Rohl, Ph.D.
Environmental Sciences Laboratory


Chrysotile- talc standard dilution -
reflection (2x 10^3)
12
10
8
6
4
2
10

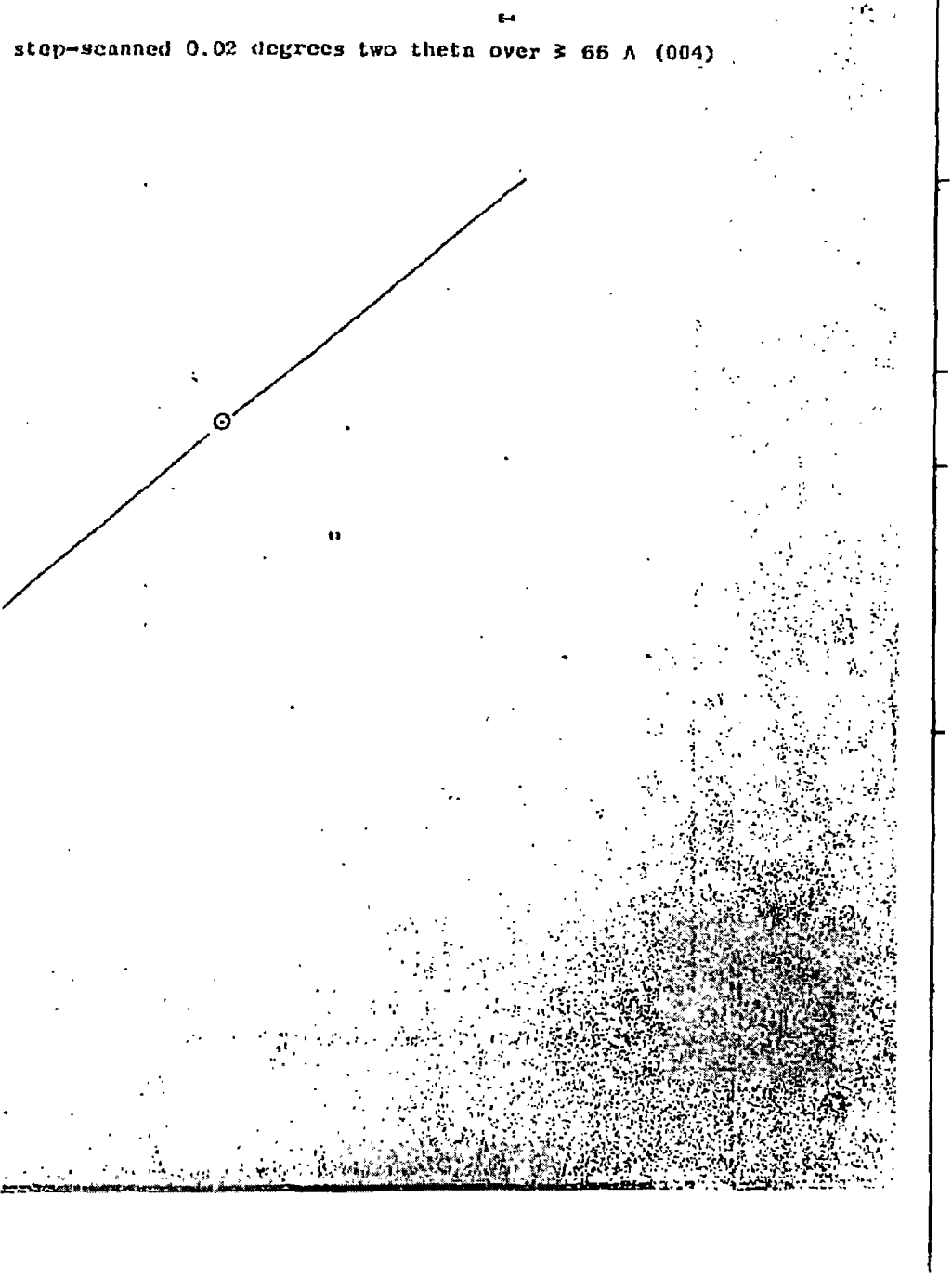
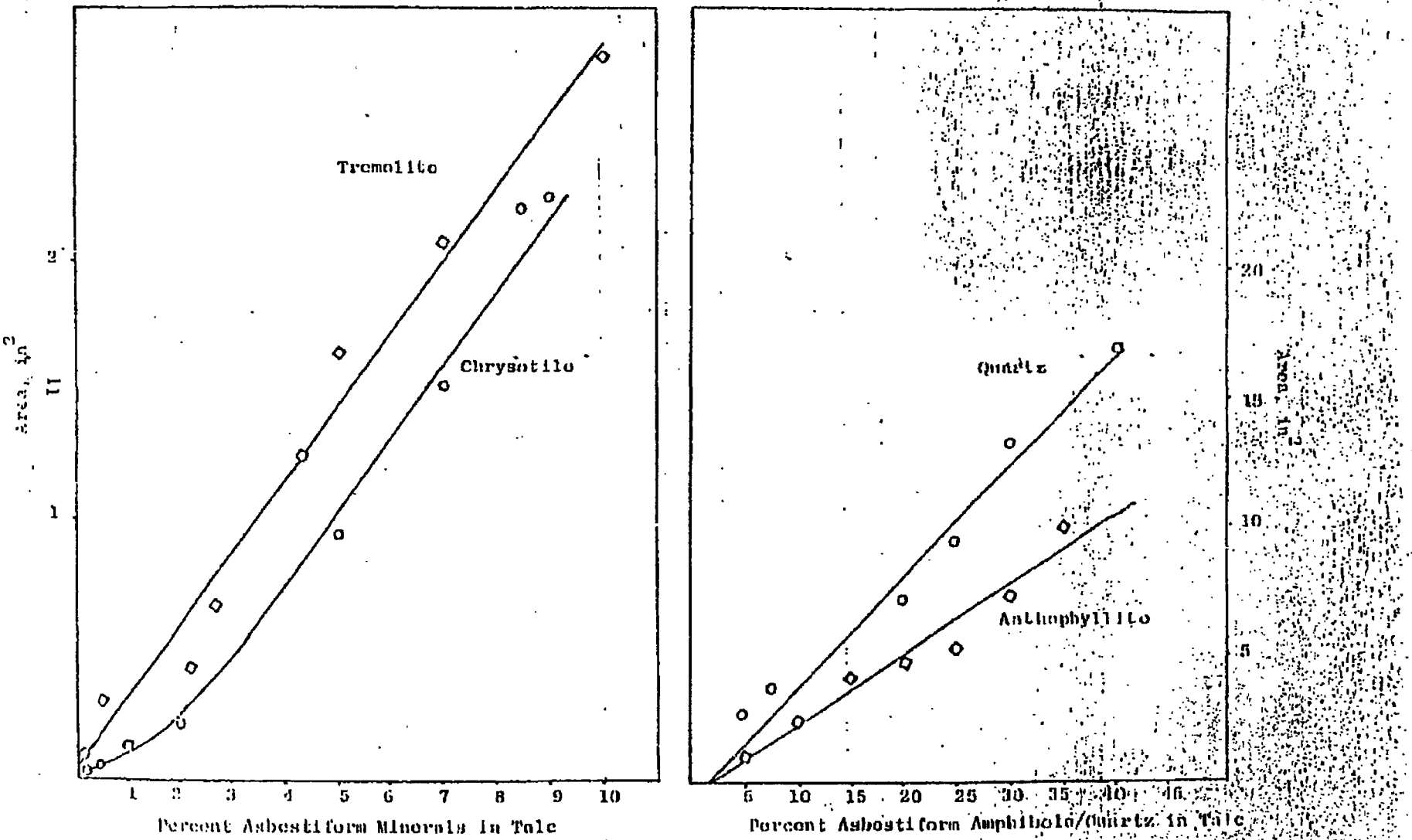
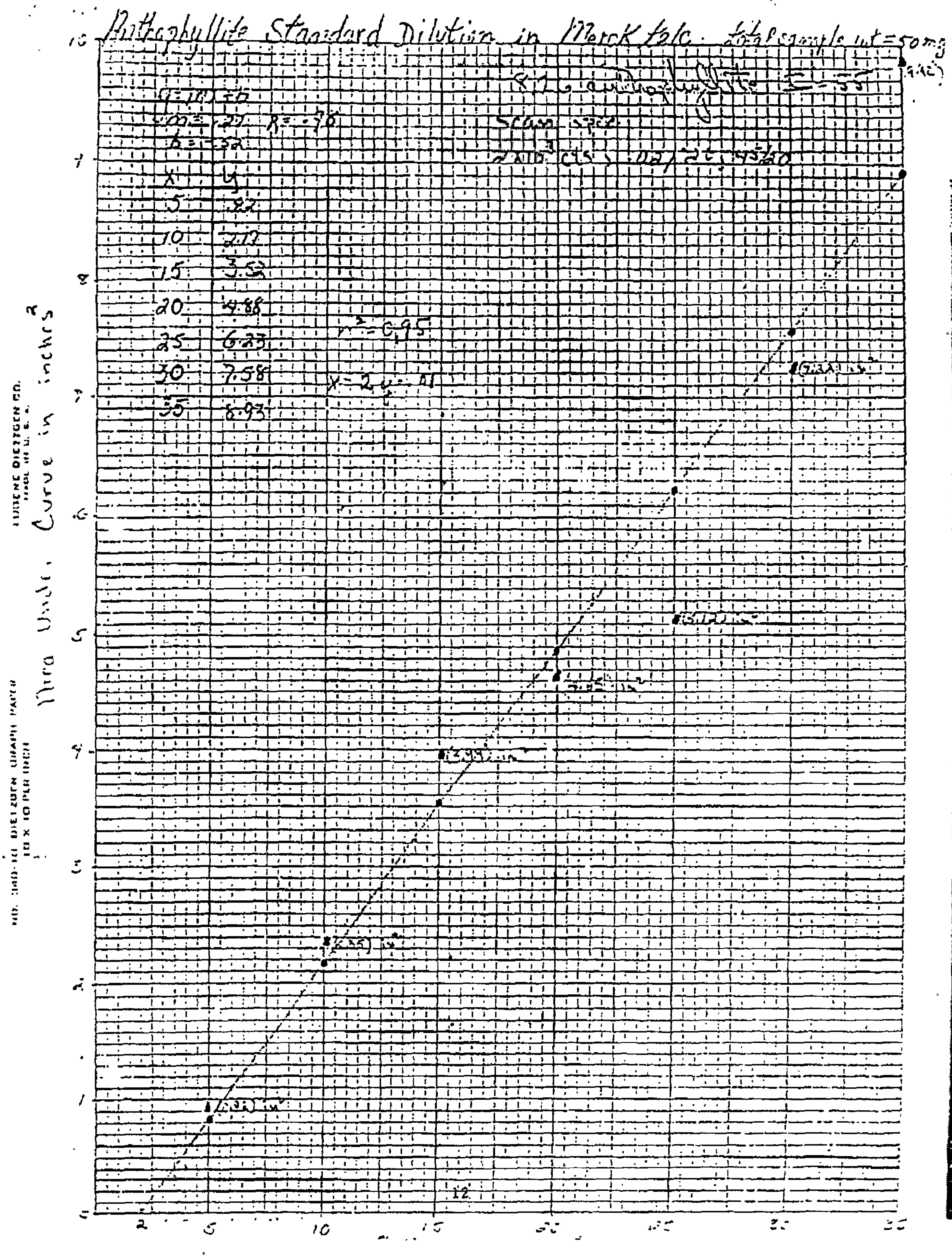

step-scanned 0.02 degrees two theta over ≥ 66 Λ (004)

Figure 2: Calibration of Asbestiform Amphiboles and Quartz in Talc





Anthophyllite Standard Dilution in Merck talc. total sample wt=50mg
x
y
5
.82
10
2.17
15
3.52
20
4.88
25
6.23
30
7.58
35
8.93
r² = 0.95
Nira Unit. Curve in inches²
EUGENE DIETZGEN CO.
10 X 10 PER INCH