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
Mt. Sinai School of Medicine

Department of Health, Education, and Welfare
Center for Disease Control
National Institute for Occupational Safety and Health
Division of Surveillance, Hazard Evaluations and Field Studies
Cincinnati, Ohio

Electron Microscopic Analyses of R.T. Vanderbilt Talcs Collected from Talc Suppliers

Analyses Performed By

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Date
October, 1976

Department of Health, Education, and Welfare
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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 1

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

Product Name	Source	NIOSH #
Nytal 300	Crone Chemical P.O. Box 14042 Houston, Texas 77021	001
Nytal 400		002
5X	Paul Crazier Company 1115 Silver St. Houston, Texas 77007	003
325		004
X		005
FT		006
3X		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	Non Asbestos	Not* Identified
		Tremolite	Anthophyllite			
Nytal 400	3/1 to 1000/1	8	88	N.D.	4	---
Nytal 300	3/1 to 100/1	12	72	N.D.	4	12
5X	5/1 to 100/1	11	80	Trace	4	5
X	8/1 to 80/1	6	80	N.D.	2	12
3X	5/1 to 100/1	12	67	N.D.	4	16
FT	3/1 to 50/1	16	72	N.D.	4	8
325	3/1 to 50/1	4	88	Trace	7	---

* 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-Ray Specta for Typical Fibers in R.T. Vanderbilt Talcs.

MOUNT SINAI SCHOOL OF MEDICINE
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FIFTH AVENUE AND 100TH STREET • NEW YORK N.Y. 10029

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, 3X, X, FT, 300, 325 and 5X.

The seven samples were analyzed quantitatively 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 agreement, averaging about 10% standard error. The results are as follows:

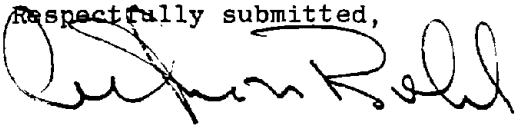
	SERPENTINE PHASE	TREMOLITE	ANTHOPHYLLITE	QUARTZ
	%	%	%	%
NYTAL 400	14-18	24	2-3	7-10
" 3X	31-35	32	not detected	7-8
" X	26-30	50-60	not determined	9-10
" FT	26-30	17	not detected	not detected
" 300	14-18	18	4-5	9-10
" 325	19-23	24	5-7	3-4
" 5X	14-18	27	7-9	8-9

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-

chemical analysis of fibers on the SEM shows that anthophyllite is present. X-ray diffraction indicated the presence of serpentine phases in all samples, but the specific mineral species of this group which includes chrysotile, antigorite and lizardite, could not be positively identified although the latter most closely fitted the x-ray patterns. Examination by TEM and SEM with energy dispersive analysis system confirmed that virtually all of the sperpentine phase consists of lizardite. Chrysotile was detected by electron microscopy in the samples. (See accompanying photomicrographs).

The samples were prepared for analysis by transmission electron microscopy by means of a technique which did not alter the size distributiin of particles. Two squares from three EM grids were scanned at 25,000x magnification on a Hitachi HU 125 microscope. Fibrous particles were abundant in all seven samples. Morphological and crystal cleavage characteristics indicated the presence of amphiboles. This was verified by observing a typical layer-type selected area electron diffraction pattern on the fibrous particles. The layer line distance normal to the long fiber axis was consistently observed to be 5.3Å, which is characteristic of the c-axis repeat of amphiboles. These particles were defined as amphibole and their location on a grid facsimile recorded. The specimens were transferred to a scanning electron microscope (CWIKSCAN) equipped with an energy dispersive x-ray spectrometer. The amphibole particles were analyzed by point count and were found to fall into two general morphological-chemical types of populations which permits them to be defined as different mineral species. The long, thin fibers (aspect ratio 10/1 or more) showed Si/Mg in ratios consistent with anthophyllite composition. These fibers contained little or no iron. Short prismatic fibers with aspect ratios from about 3/1 to 5/1 contained Si/Ca/Mg proportions consistent with tremolite. No iron was found in tremolite.

Respectfully submitted,



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

ANR:sl
Enc.

Chrysotile- talc standard dilution - step-scanned 0.02 degrees two theta over 3.66 A (004) reflection (2×10^3)

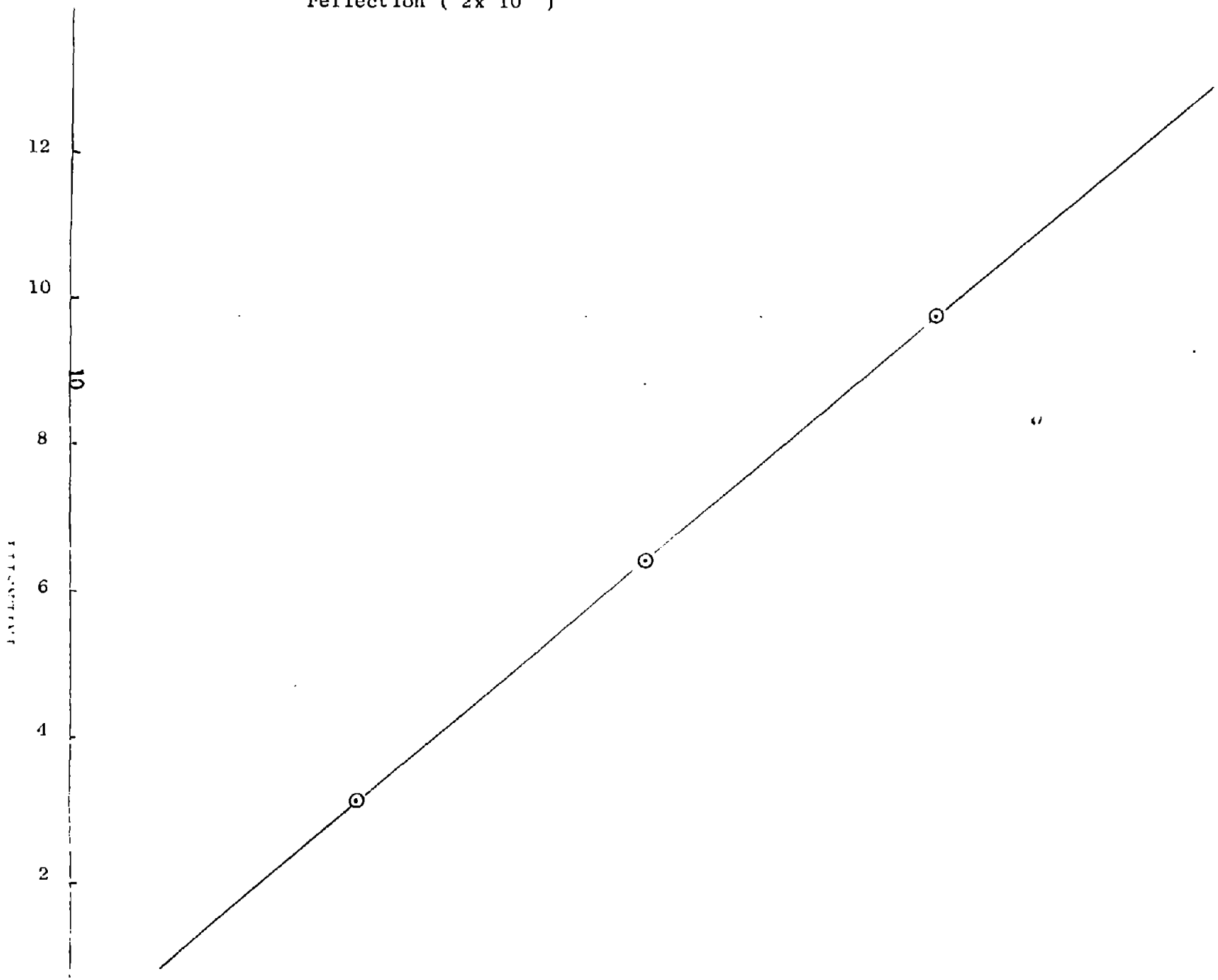
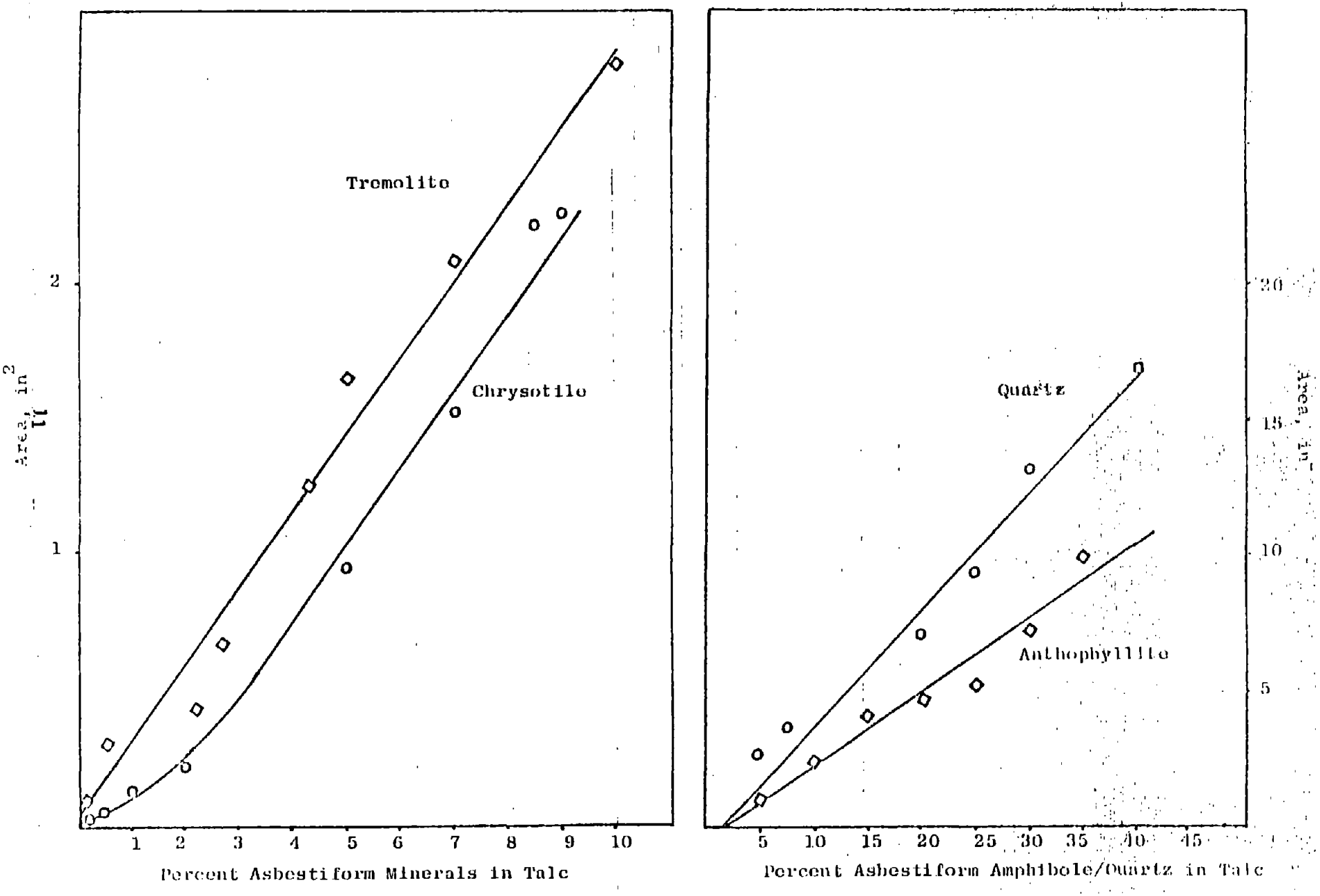
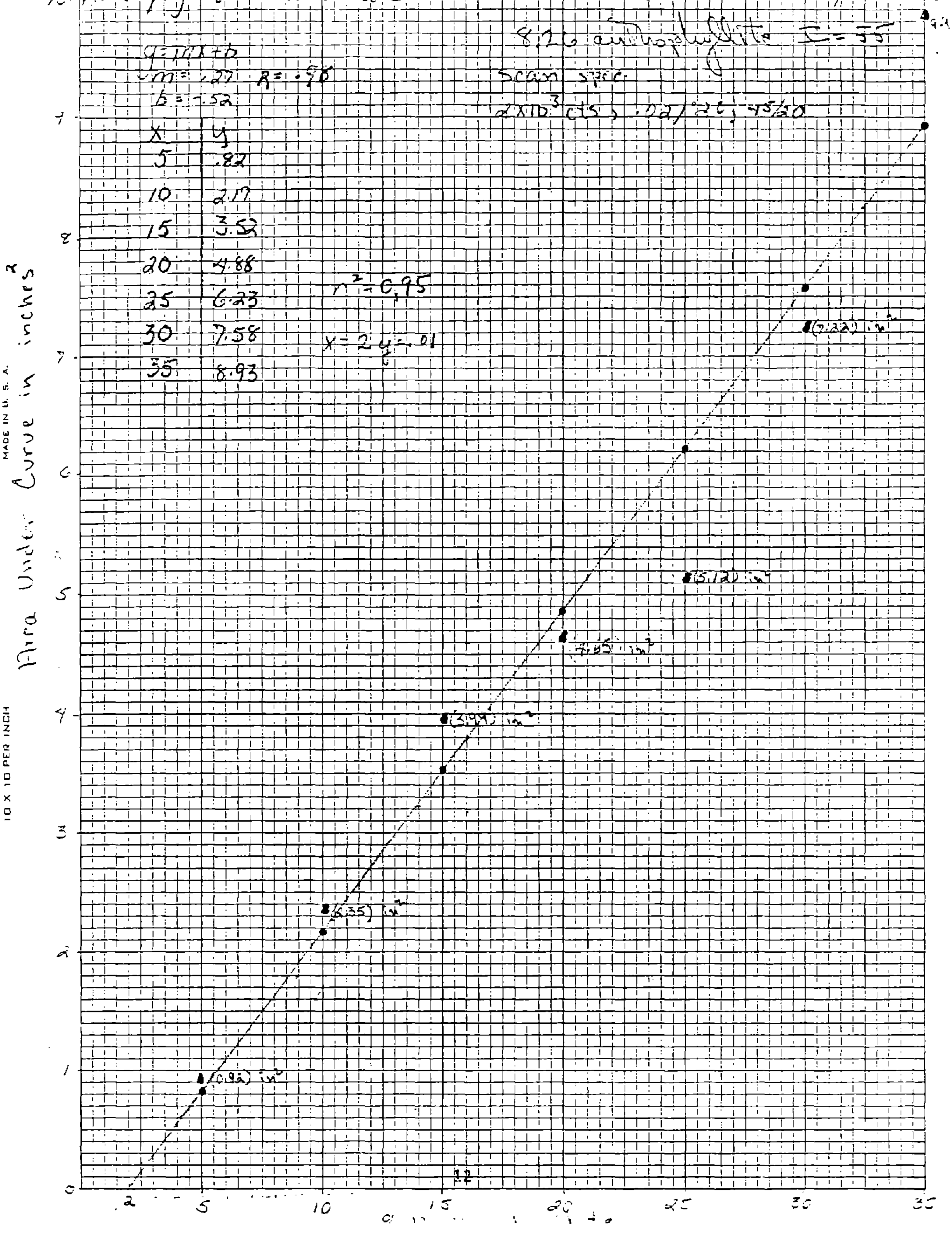


Figure 2: Calibration of Asbestiform Amphiboles and Quartz in Talc



Anthophyllite Standard Dilution in Merck talc. total sample wt = 500



Report to

Dr. John Dement

NIOSH
Cincinnati, Ohio 45202

EXAMINATION OF TALCS FROM

GOUVERNEUR DISTRICT — NEW YORK

Date: 12 December 1975

MA Number: 4800

Copy 3 of 4

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2820 SOUTH MICHIGAN AVENUE • CHICAGO, ILLINOIS 60616

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EXAMINATION OF SEVEN TALCS FROM THE GOUVERNEUR DISTRICT, NEW YORK

SUMMARY

An extensive examination has been carried out of seven talcs submitted by the National Institute of Occupational Safety and Health, to determine their asbestos content. These talcs all originated from the Gouverneur District of New York State and were identified by number code in the series 83775-83761.

Examination was carried out by a combination of light microscopy, using both polarized light and dispersion staining, x-ray diffraction, and electron microscopy combining electron diffraction and elemental analysis.

The results of the examination showed the presence of asbestiform amphibole in all seven samples. The amounts of the total amphibole and of the fibrous amphibole differed between the various samples. In all cases the principal asbestiform amphibole was identified as tremolite, but this was of a low calcium variety, containing approximately 2-3% calcium.

In Sample 83757, x-ray diffraction examination suggested that a small percentage of anthophyllite was also present. Examination by light microscopy, however, could not conclusively differentiate between anthophyllite and tremolite in this sample. The identification of tremolite rather than a calcium bearing anthophyllite in all samples was based on the extinction angle, observed under crossed polars, of the fibrous amphibole.

INTRODUCTION

The National Institute of Occupational Safety and Health is currently sponsoring a study of occupationally related disease in the talc industry. As part of this study, Walter C. McCrone Associates, Inc., has been contracted to study and characterize the raw talc ores. The present work is related to this major program and is concerned with a study of seven specific samples from the Gouverneur talc ore body.

Material and Method of Conducting Tests

Seven talc samples were submitted to Walter C. McCrone Associates, by John Dement of NIOSH. These samples were identified by numbers running in sequence from 83755-83761. No indication was given of the sample location within the Gouverneur talc body. The samples were examined by light microscopy, using both polarized light and dispersion staining, x-ray diffraction, and transmission electron microscopy combined with electron diffraction and elemental analysis.

Light microscopy

Permanent mounts were prepared for all seven samples, using Aroclor 5442 as a mounting medium. These preparations were examined under partially uncrossed polars to obtain a general indication of the mineral types present together with their shapes and sizes. Samples for examination by dispersion staining were prepared using Cargille high dispersion refractive index liquids with n values of 1.550 and 1.605. The lower refractive index was used to examine the serpentine content of the talcs; the higher value was chosen to enable identification of the amphibole present. Examination by dispersion staining was at a magnification of approximately 200X in plane polarized light.

X-ray diffraction

For x-ray diffraction examination, samples of the powders were packed into aluminum cup holders and placed in the Norelco diffractometer. The samples were rotated around an axis perpendicular to the axis of the diffractometer which was scanned through a 2θ range from approximately 6° to approximately

60° with a scanning speed of 1° per minute and a chart speed of 1/2 inch per minute. Cu radiation, nickel filtered, was used at an accelerating voltage of 42 kV, and a current of 25 milliamps. The quartz content of the samples was determined by step scanning through the 3.35 Å line for quartz and comparing the resulting data with that for "spiked" standards of quartz in talc. The step increment was 0.01°, 2θ with a counting time per step of 50 secs., an 8 sec. time constant and a chart speed of 1/8 inch per minute.

Electron microscopy

For electron microscopic examination samples of the powder were suspended in isopropanol and lightly ultrasonerated. A drop of this suspension was placed on carbon coated Nylon grids. Examination of the samples was carried out in a combined electron-microscope-microprobe-analyzer, EMMA-IV. The samples were examined and photographed at several different magnifications, electron diffraction patterns were obtained from typical fibers present and energy dispersive x-ray analysis was performed on these fibers.

RESULTS

1) Light microscopy

The following general statements can be made:

All the samples contained a serpentine which, on the basis of dispersion staining and morphology, has been identified as lizardite. Chrysotile (the asbestiform variety of serpentine) and antigorite were not detected.

All the samples contain tremolite identified by dispersion staining and polarized light microscopy and, although it would be possible to miss a small percentage of anthophyllite particles, since most anthophyllites give nearly the same dispersion staining colors as tremolite, appreciable amounts of anthophyllite would not be missed because a significant number of particles would lie in the proper orientation for positive identification. With the exception of an occasional particle in Sample 83757, all the particles which showed the tremolite/anthophyllite dispersion colors showed oblique extinction under crossed polars ($\gamma \wedge c$ = 17-18°), thus identifying them as tremolite.

All samples contain talc in a form which looks like fibers. These give typical talc colors by dispersion staining. The majority of these talc fibers are apparently long talc rods. These talc fibers are especially long in Sample 83759.

Most samples are believed to contain a little quartz on the basis of the light microscopical examination. It is difficult to put an accurate value to the amount of quartz present, since it is extremely difficult to distinguish between quartz and lizardite under dispersion staining conditions.

A calculation of the percentage of amphibole present and of the portion of this amphibole which was fibrous was made as follows. First the portion by volume of the total tremolite present as fibers was estimated taking cognizance of both particle size and numbers present. Subsequently, 30 fields in each sample were examined in detail and the percentage by area of the total particle area occupied by amphibole particles was estimated.

Table 1 summarizes the observations made by light microscopy.

X-ray diffraction

All samples showed the presence of talc, an amphibole and a serpentine. Inconclusive evidence of quartz was detected by x-ray diffraction on the normal scan. By step scanning, however, low levels of quartz were found. The quartz data are presented in Table 2 and Figure 1.

The serpentine minerals are difficult to distinguish from each other by x-ray diffraction. A paper by Mumpton[1] indicates certain lines which may be used to distinguish the three serpentine polymorphs provided that the samples are relatively monomineralic. Specifically, Mumpton mentions that lizardite is characterized by a strong doublet at 1.531 Å. This doublet has indeed been observed on our x-ray diffraction trace, thereby confirming the light microscopical identification of lizardite by dispersion staining.

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[1] Characterization of chrysotile asbestos and other members of the serpentine group of minerals, Siemens review XLI (1974) 7th special issue, X-ray and Electron Microscopy News.

Table I
Summary of Light Microscopical Data

	Nytal 300	Nytal 400	3X	225	X	FH	3X
Sample	83755	83756	83757	83758	83759	83760	83761
Total tremolite (% of sample by area)	25-30	15-20	20-25	25-30	25-30	15-20	25-30
Fibrous tremolite (estimated % by vol. of total tremolite)	5	25	8-10	5	1	25	10
Quartz	+	possible	+	+	-?	+?	+?
Serpentine	+	?	-	+?	+	probable	probable
Talc "fibers" (rolls, shards etc.)	+	+	+	+	+(long)	+	+
Comments				large tremolite plates	large particle size	small particle size	

Table II
Tremolite and Quantity Content of Talc Samples

Sample	83755	83756	83757	83758	83759	83760	83761
Quartz Content**	2%	1/2-1%	~1 1/2%	~1 1/2%	<1/2%	below detection limit	1/2-1%
Content tremolite	40-50%	50-60%*?	40-50%*	~60%	>50-60%*	50-60%	40-50%

** Step scanning data.

* Indicates possible anthophyllite present in addition to tremolite.

This leaves only the amphibole to be characterized in all seven samples. The primary amphibole observed is tremolite. The diffraction lines observed are consistent with ASTM Card 13-437 for tremolite and with published data for tremolites from the Gouverneur district. In one sample, however, Sample 83757, a distinct side peak on the tremolite 8.410 line is observed and it is believed that this side peak is due to the presence of some anthophyllite. Less distinct evidence of this side peak is also present on Sample 83756 and possibly also 83759 (see Figures 2-8 for a comparison of the 6°-12°, 2θ range on these samples). As is clear from these figures, in addition to the side peak attributed to anthophyllite there is considerable variation in the ratio of talc to amphibole in these samples. An attempt to quantitate this relationship in terms of percentage of amphibole present is also given in Table 2.

Electron microscopical examination

Electron microscopical examination showed that all seven samples contain substantial amounts of fibrous material. This was highest in Sample 83757 and lowest for Samples 83756 and 83761. Several typical electron micrographs showing general areas of the samples are appended to this report (Figures 9-17). The electron microscopical examination concentrated on the fibrous material and an attempt was made to estimate how much of this material was in fact asbestiform, that is, a fibrous amphibole as distinct from the fiber forms of talc consisting of ribbons, shards, rolls, etc. This examination was conducted both by electron diffraction and by elemental analysis using energy dispersive x-ray. The results of these examinations are summarized in Table 3, the estimated percentages being based on occluded area. Some typical electron micrographs, diffraction patterns and energy dispersive x-ray spectra are appended. In none of these fibers examined was there a particularly high percentage of calcium noted; however, calcium was observed at the level of the order of 1-3% in all the amphibole fibers examined. The only conclusion which

Table III
Electron Microscopical Estimates of Fiber Content

Sample	83755	83756	83757	83758	83759	83760	83761
Total fibers	~10%	~10%	~40%	~30%	~5-10%	~20%	~10%
Fibrous amphibole (as % of total sample)	<5%	<5%	15-20%	10-15%	<5%	~5%	<5%

Table IV
Overall Data Summary

Sample	83755	83756	83757	83758	83759	83760	83761
Total fibers (TEM)	10%	10%	40%	30%	5-10%	20%	10%
Amphibole fibers[1] (TEM)	<5%	<5%	15-20%	10-15%	<5%	5%	<5%
Total amphibole[2] (XRD)	40-50%	50-60%	40-50%	~60%	>50%	50-60%	40-50%
Total amphibole[3] (LM)	25-30%	15-20%	20-25%	25-30%	25-30%	15-20%	25-30%
Fibrous amphibole[4] (LM)	1-2%	3-5%	1-3%	1-2%	<1/2%	3-5%	2-3%
Serpentine[2,5] (XRD)	10-15%	10-15%	10-12%	10-12%	~15%	~20%	~30%
Quartz[2,6] (S.S. X-RD)	2%	1/2-1%	~1 1/2%	~1 1/2%	<1/2%	B.D.L.	1/2-1%

NOTES:

1. Based on occluded area
2. Estimated weight percentage
3. Based on occluded area
4. Derived from estimated % of total amphibole which was fibrous. See also Table I.
5. Identified as lizardite by light microscopy
6. S.S. XRD = Step scanning x-ray diffraction

can be drawn from the electron diffraction and energy dispersive x-ray analysis is that one has a fibrous amphibole of the tremolite or anthophyllite type. It is generally not possible to distinguish between tremolite and anthophyllite by electron diffraction and one would therefore be tempted to suggest that the fibers were anthophyllite with a trace amount of calcium. Taken in conjunction with the light microscopical examination, however, during which a diligent search was made, particularly on Sample 83757, for fibers which might be anthophyllite rather than tremolite, one is forced to the conclusion that, rather than being a calcium bearing anthophyllite, the fibers are in fact a low calcium tremolite. The aspect ratios of many of the fibers observed are quite high and numerous fibers, such as those shown in Figures 20, 22, 24 and 30 showed the classic fibrous structure of the asbestos minerals. There is no doubt in our minds that these are indeed asbestos fibers rather than cleavage fragments of a more massive amphibole. Massive forms of the amphibole are, however, present and the fibrous amphibole content represents only a fraction of the total amphibole content of the samples.

CONCLUSIONS

Table 4 summarizes the results of all the investigations which we have carried out. In answer to the specific question, "Do these particular talc samples contain any asbestos minerals?", the answer must be an unequivocal yes. The asbestos mineral present appears to be a low calcium tremolite and is definitely asbestiform by any definition of the word. The asbestos content in the samples varies from less than 5% to approximately 20%, based on occluded areas observed in the transmission electron microscope.

Respectfully submitted,

Ralph J. Hinch, Jr.
Research Chemist

Lucy B. McCrone
Senior Research Scientist

Richard J. Shimps
Research Chemist

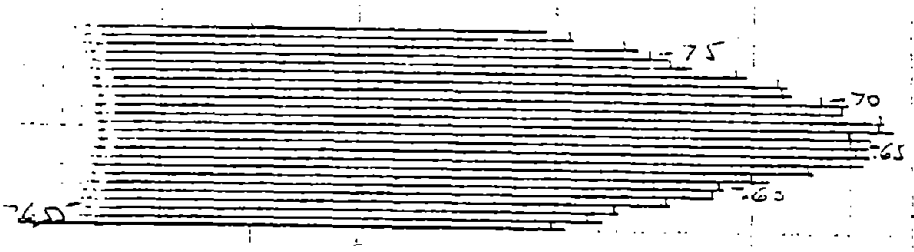
Ian M. Stewart
Manager, Electron Optics Group

FIGURES 1 THROUGH 40

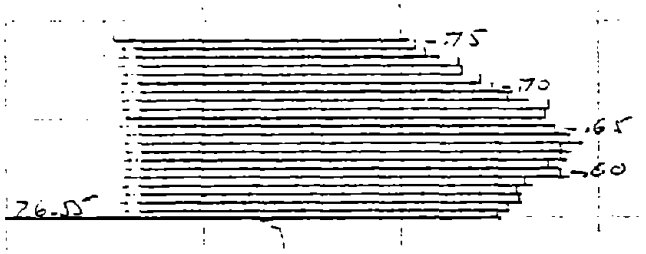
EXAMINATION OF TALCS FROM

GOUVERNEUR DISTRICT — NEW YORK

Figure 1 Step scanning data for quartz



Sample 83755 (001) Talc (α-SiO_2+)



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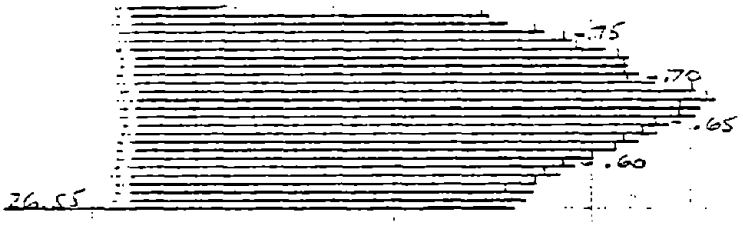

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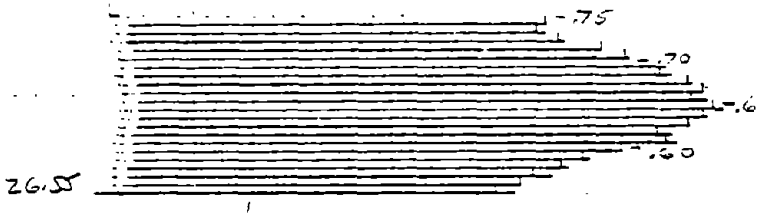
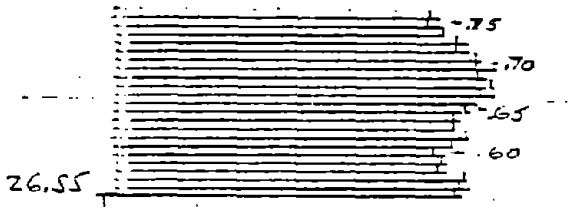
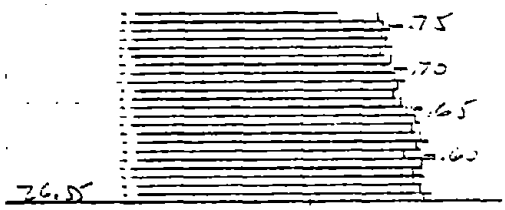


Sample 83758 (004) Talc (α-SiO_2+)



Sample 83759 (005) Talc (α-SiO_2+)



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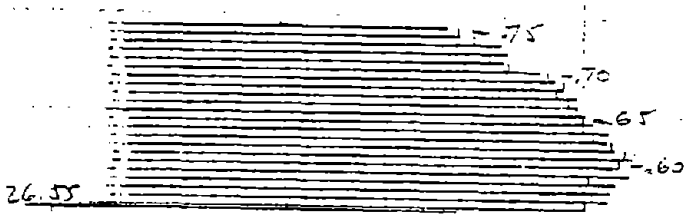
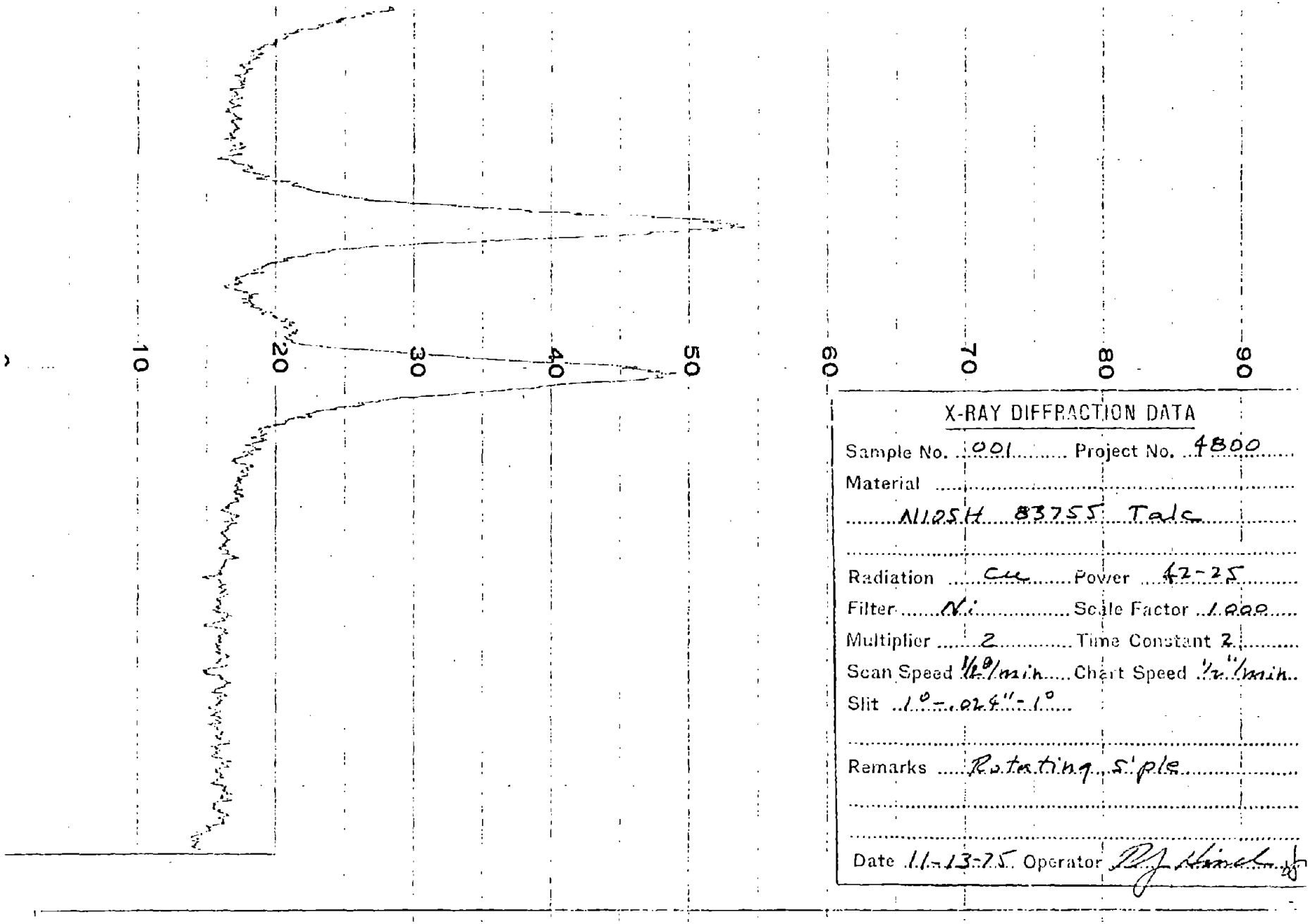
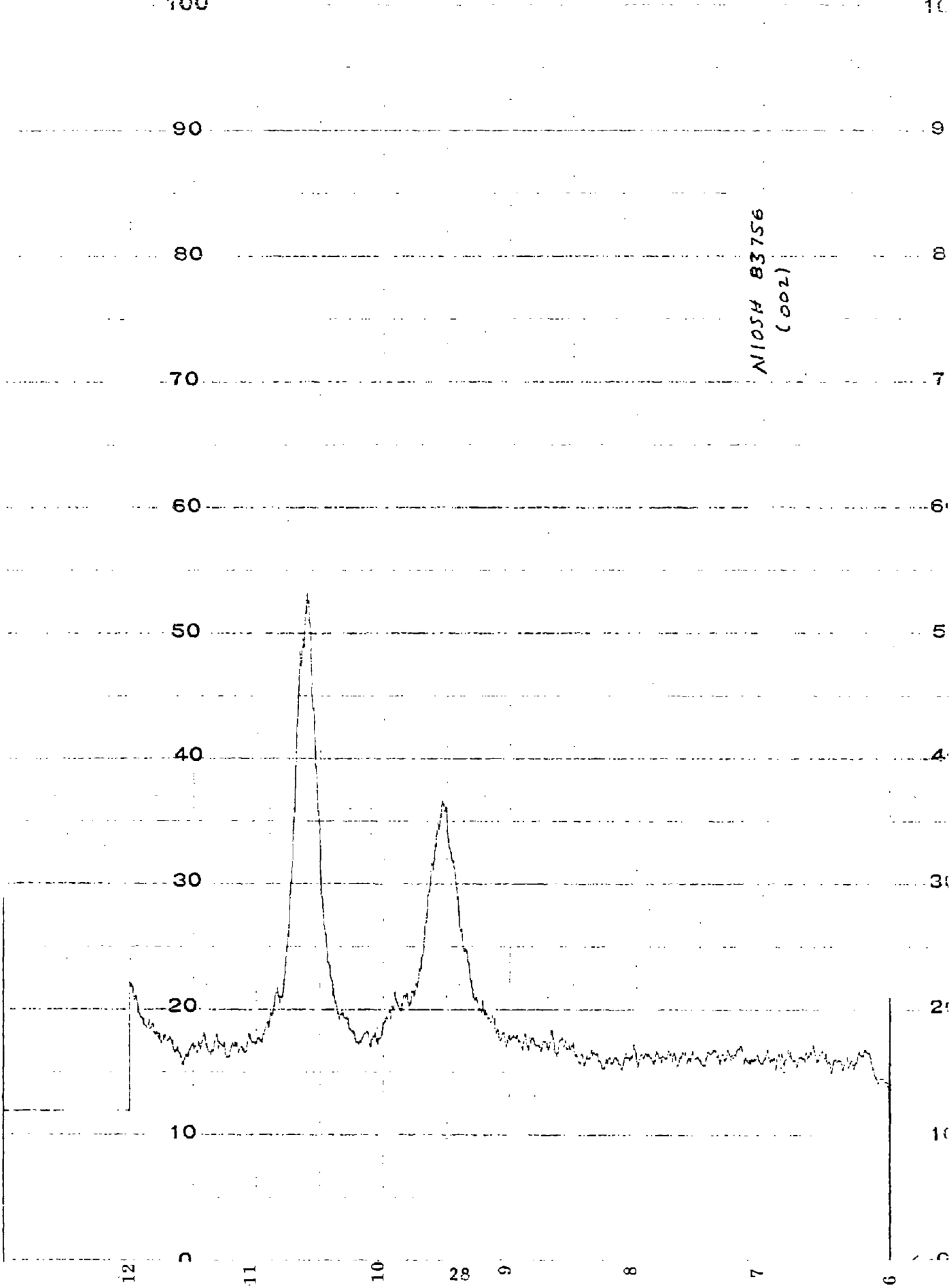


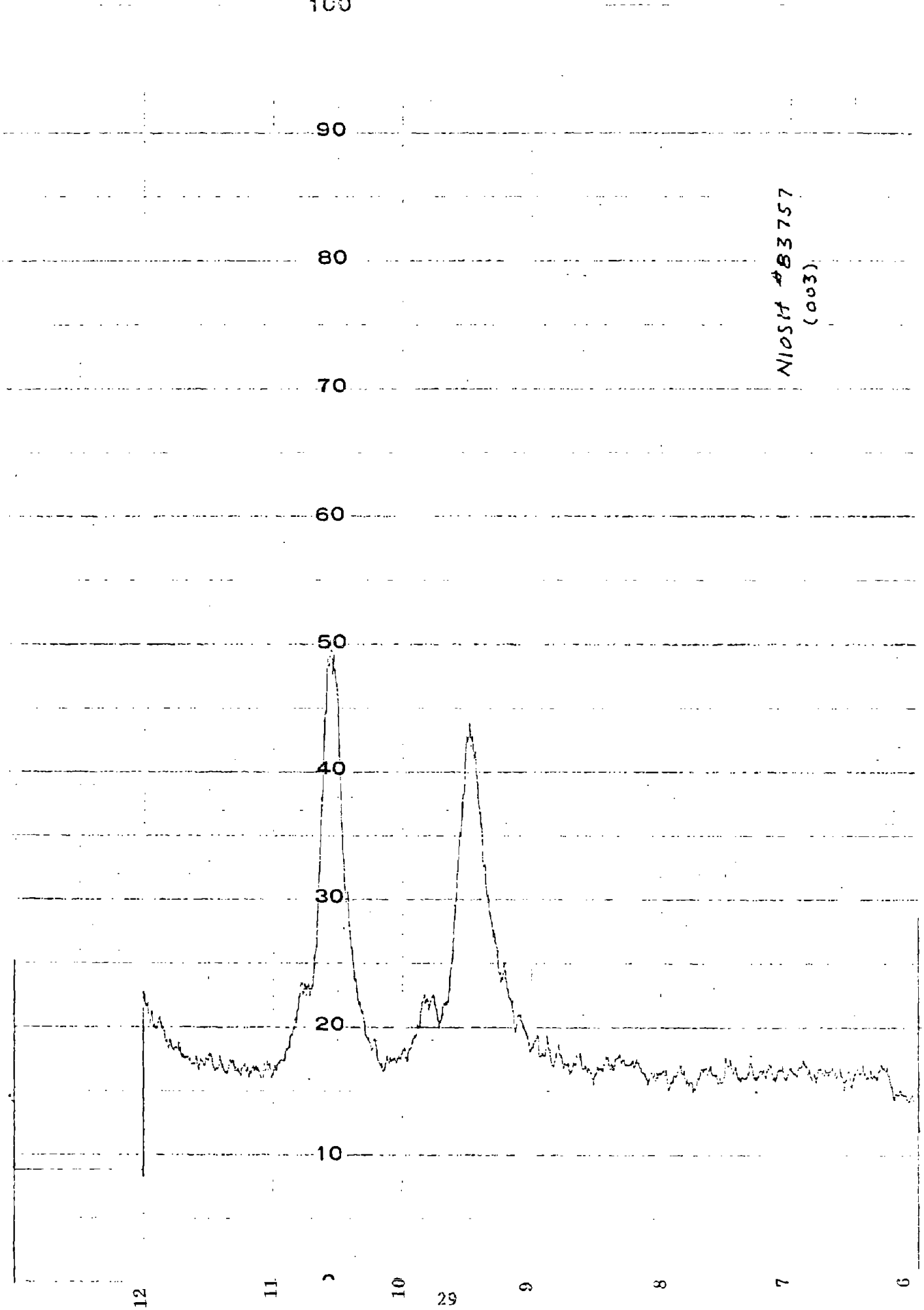
Sample 83761 (007) Talc (α-SiO_2+)



Figures 2-8 6°-12° range of 2θ for Samples 83755-83761



walter c. mccrone associates, inc.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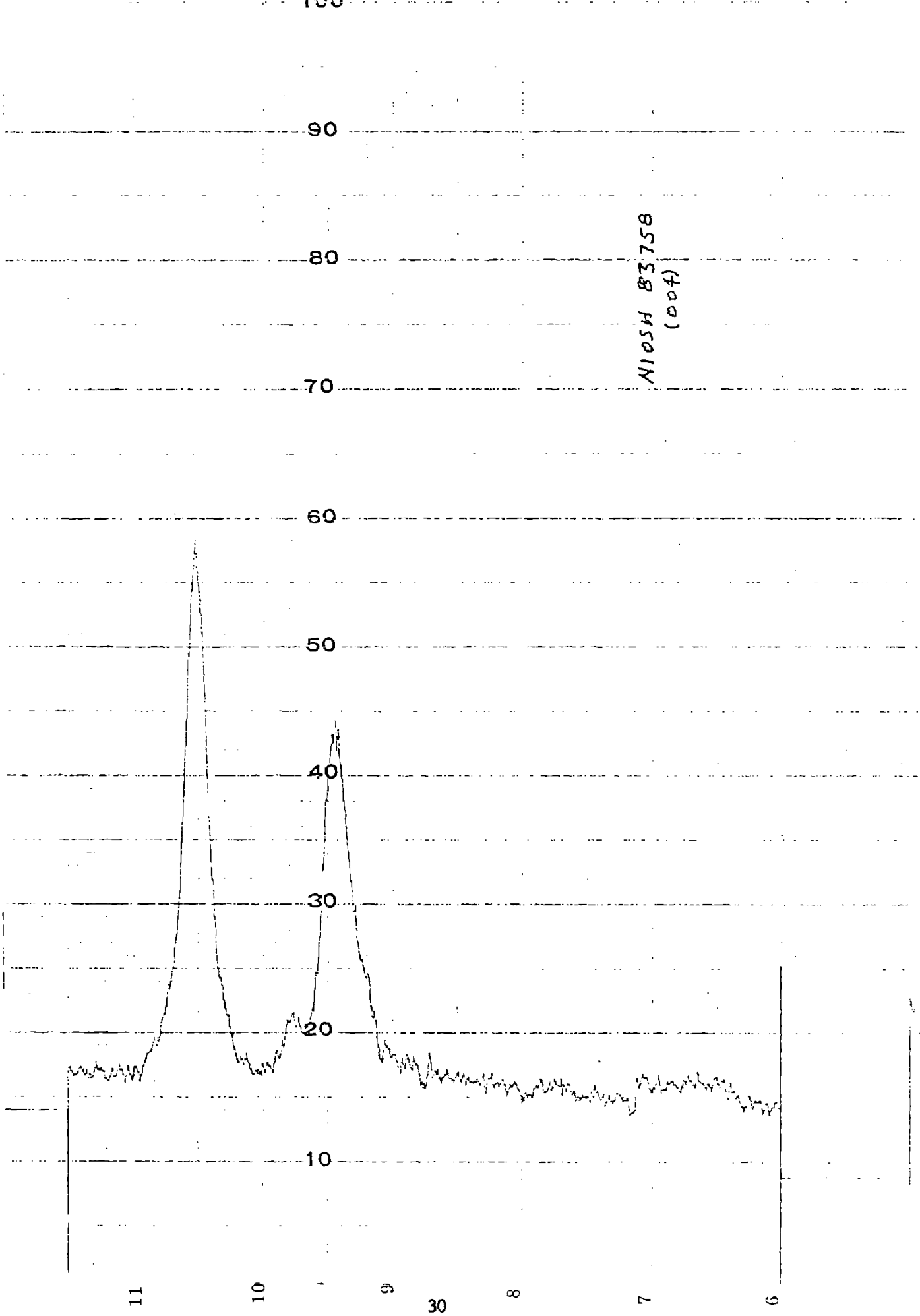
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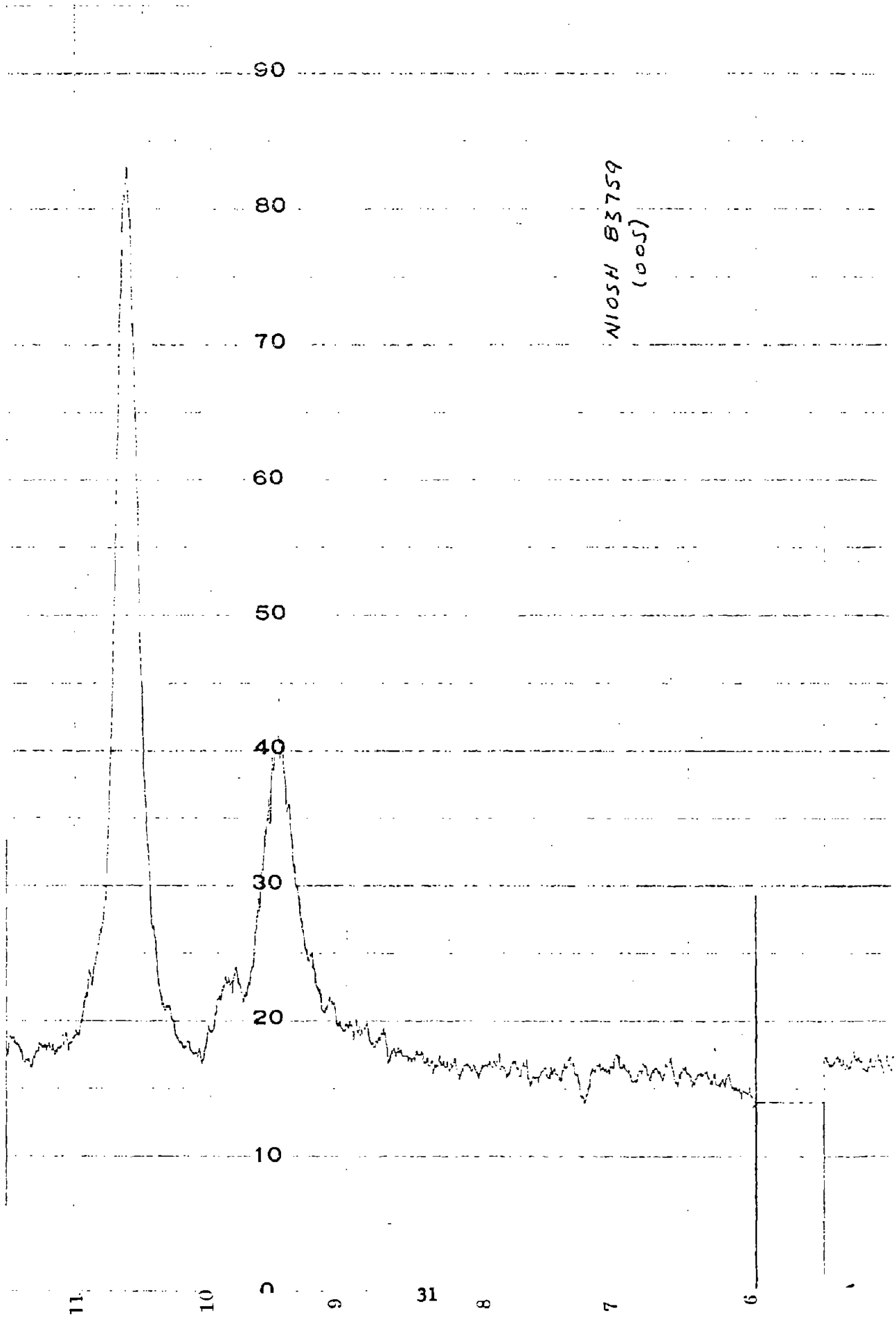

Figure 6

walter c. mccrone associates, inc.

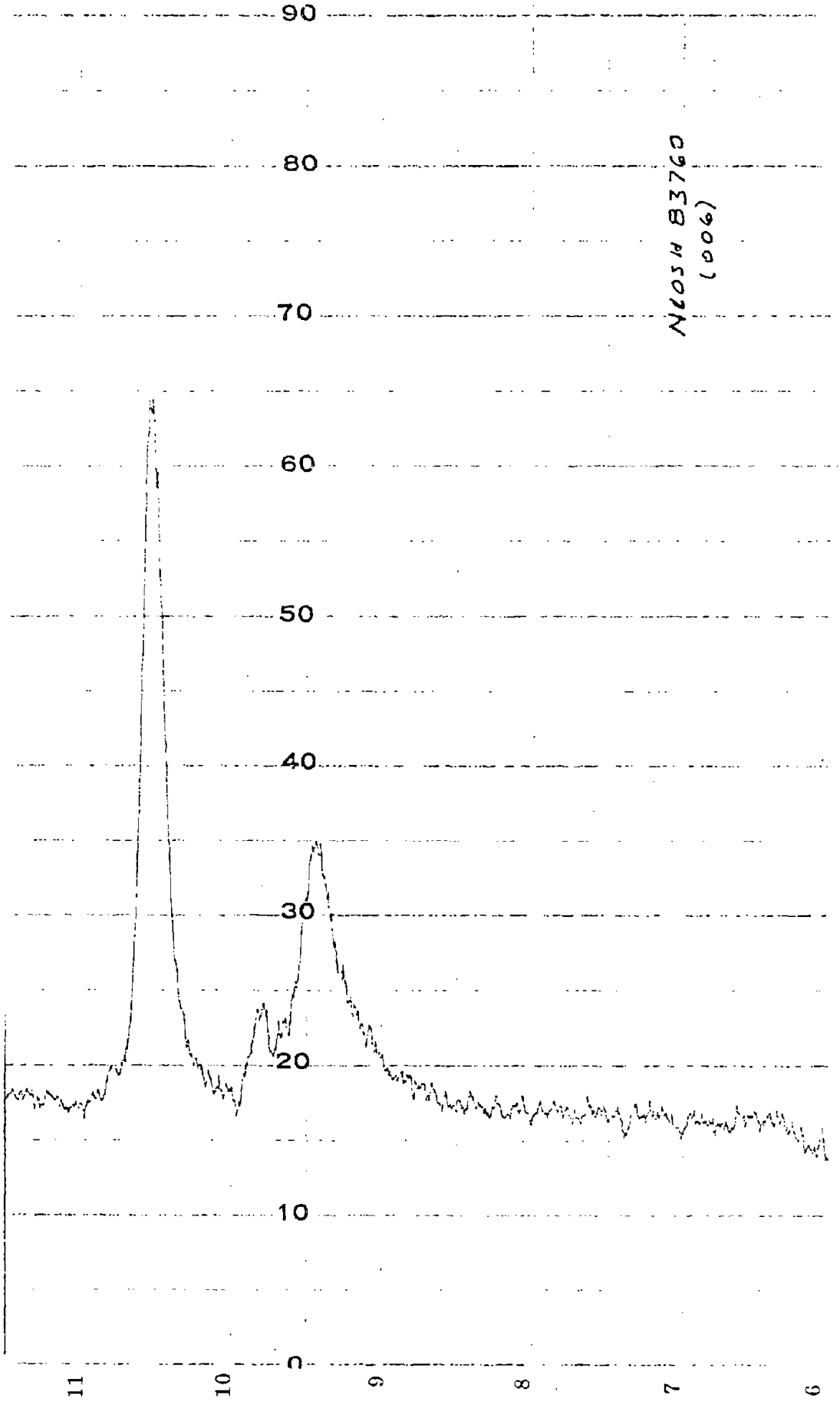

Figure 7

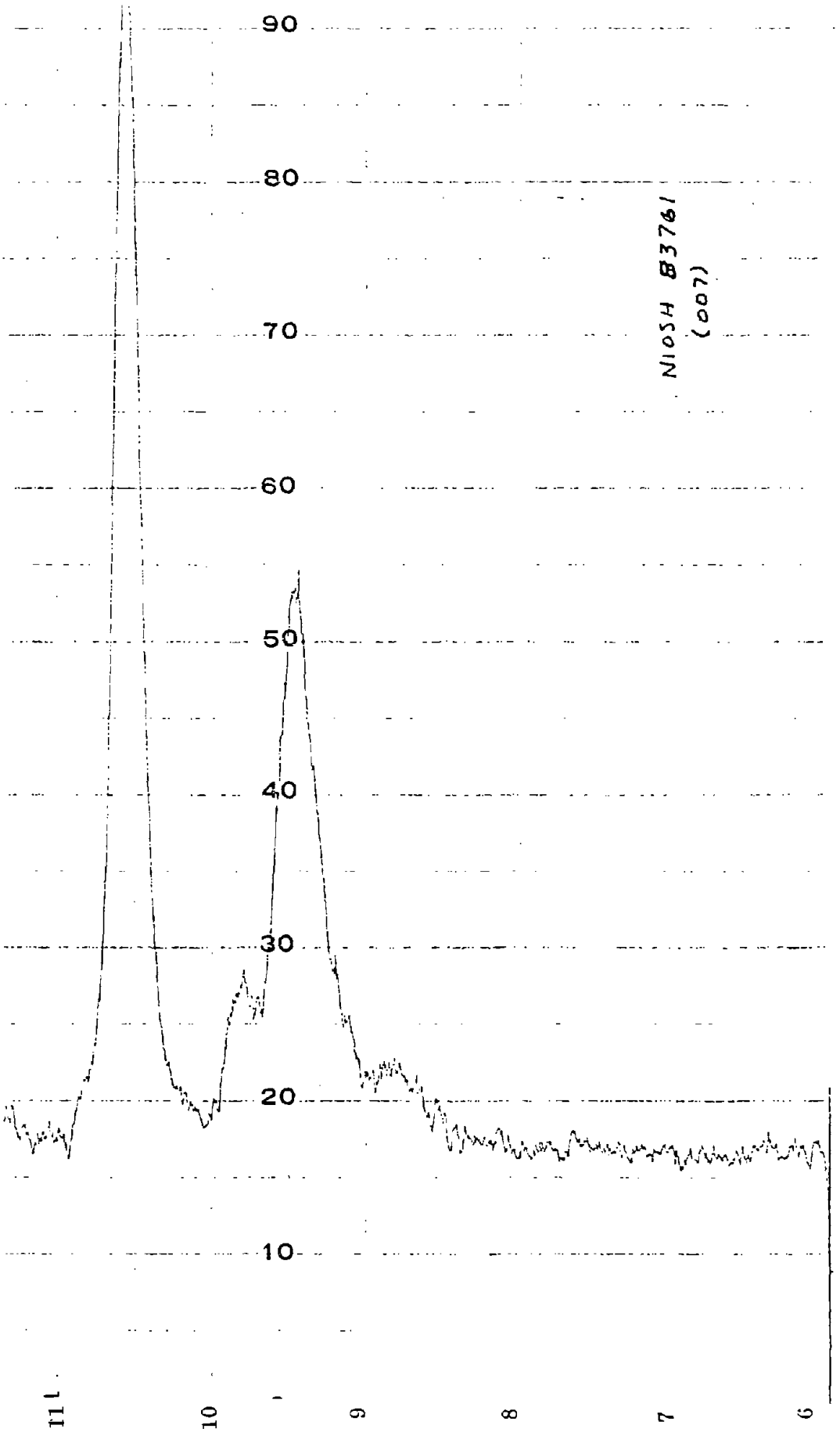

Figure 8