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Report to
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EXAMINATION OF TALCS FROM
GOUVERNEUR DISTRICT — NEW YORK

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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, Xnc., 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 a axis perpendicular to the axis of the diffractometer which was scanned through a 2θ range from approximately 6° to approximately

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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 ($\gamma^c = 17-18^\circ$), 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 8375g.

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 Mumpton¹ indicates certain lines which may be used to distinguish the three serpentine polymorphs provided that the samples are relatively monominerallic. 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.

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 Series.

Table I
Summary of Light Microscopical Data

	Nyral 200	Nyral 400	3X	225	X	FI	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	probabl
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	83753	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%	50-80%*?	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. The primary amphibole observed is tremolite. The diffraction patterns are consistent with ASTM Card 13-437 for tremolite and with data for tremolites from the Gouverneur district. In one sample, sample 83757, a distinct side peak on the tremolite 8.410 line is identified. It is believed that this side peak is due to the presence of some talc. 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 2θ range on these samples). As is clear from these figures, in the side peak attributed to anthophyllite there is considerable overlap with the ratio of talc to amphibole in these samples. An attempt to determine this relationship in terms of percentage of amphibole present is also included 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 15-17). The electron microscopical examination concentrated on the fibrous material and an attempt was made to estimate how much of this material is in an asbestiform, that is, a fibrous amphibole as distinct from the fiber-like talc consisting of ribbons, shards, rolls, etc. This examination was done both by electron diffraction and by elemental analysis using energy-dispersive x-ray. The results of these examinations are summarized in Table 3, the listed percentages being based on occluded area. Some typical electron micrographs, diffraction patterns and energy dispersive x-ray spectra are included in the appendix. In none of these fibers examined was there a particularly high percentage of calcium noted; however, calcium was observed at the level of the 0.3% 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 amphi- bole ² (XRD)	40-50%	50-60%	40-50%	~60%	>50%	50-60%	40-50%
Total amphi- bole ³ (LM)	25-30%	15-20%	20-25%	25-30% ⁴ ;	25-30%	15-20%	25-30%
Fibrous am- phibole 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.

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Respectfully submitted,

Lucy B. McCrone

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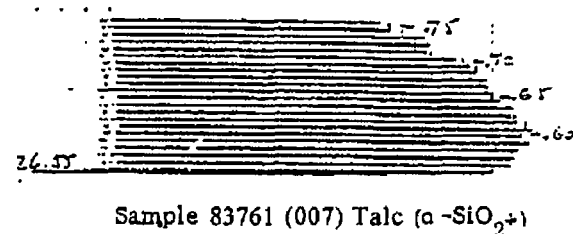
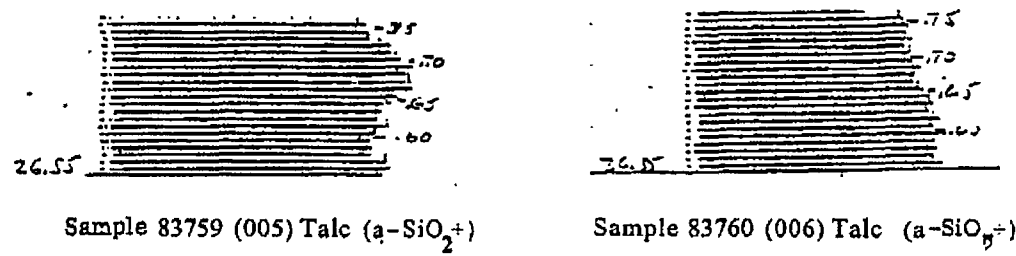
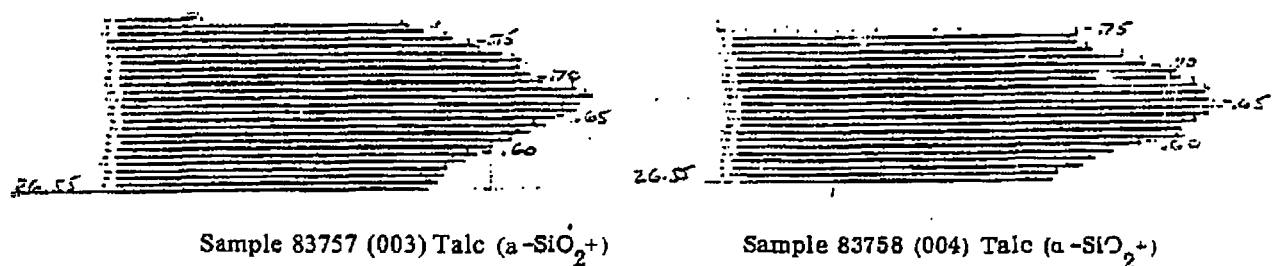
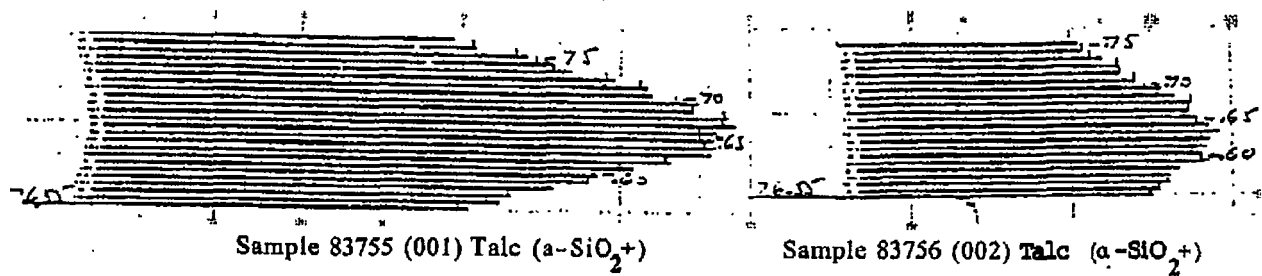
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FIGURES 1 THROUGH 4G
EXAMINATION OF TALCS FROM
GOTJVERNEUR DISTRICT — NEW YORK

Figure i Step scanning data for quartz



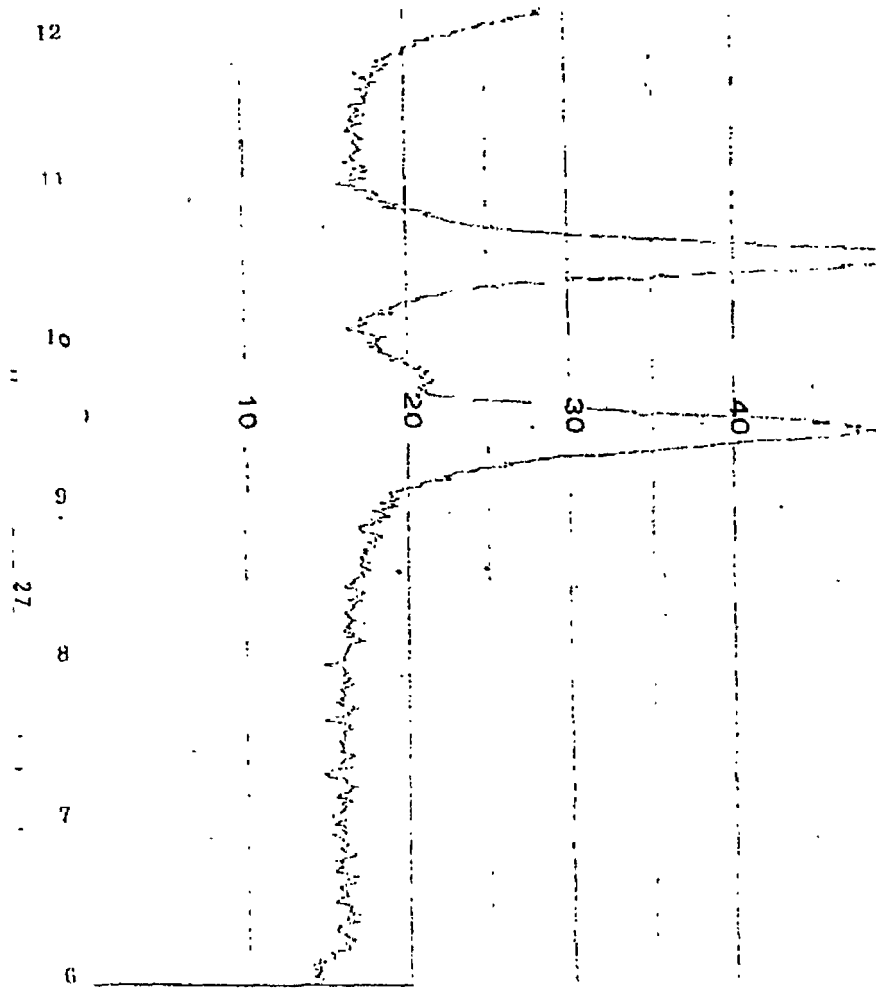
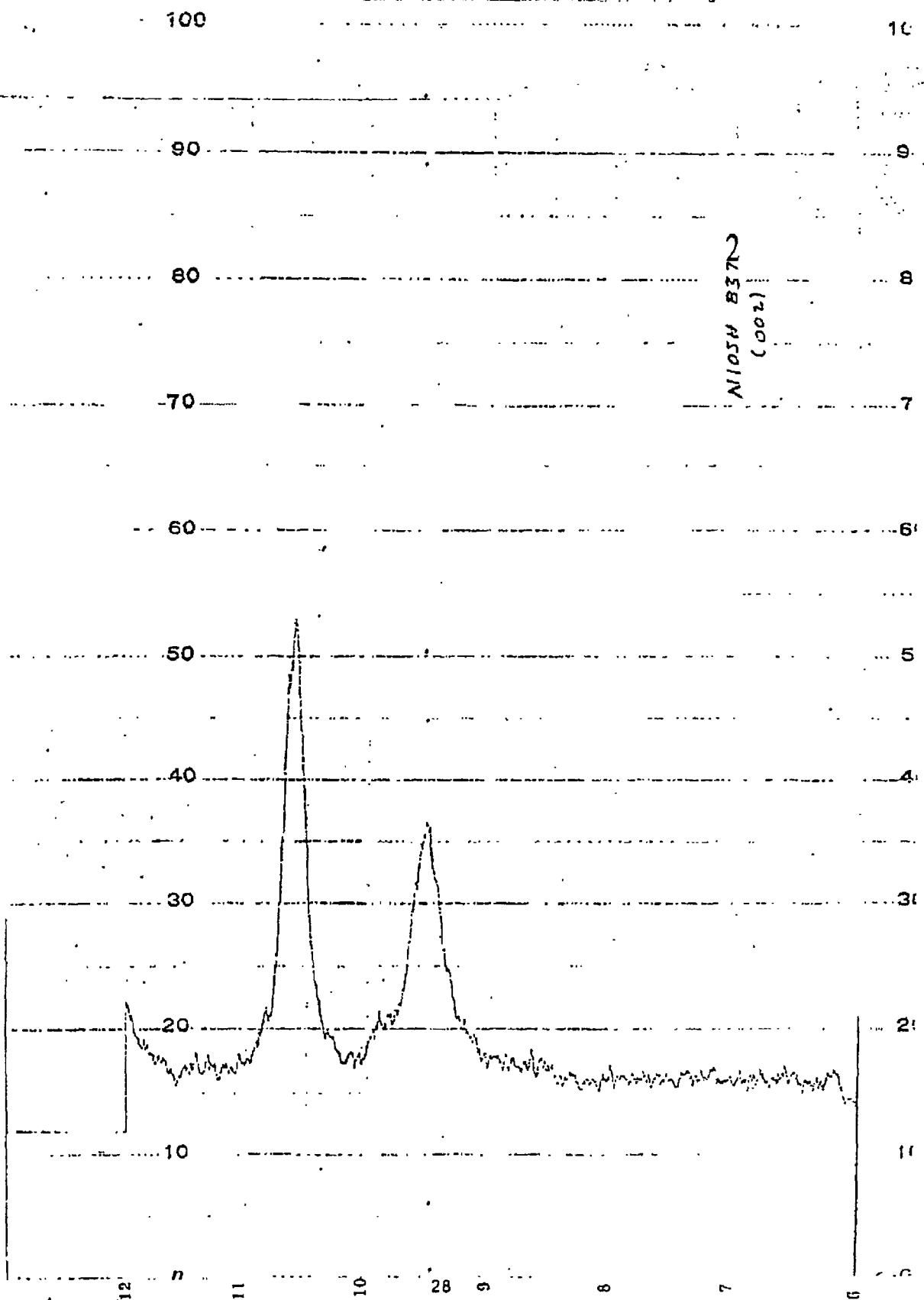


Figure 2

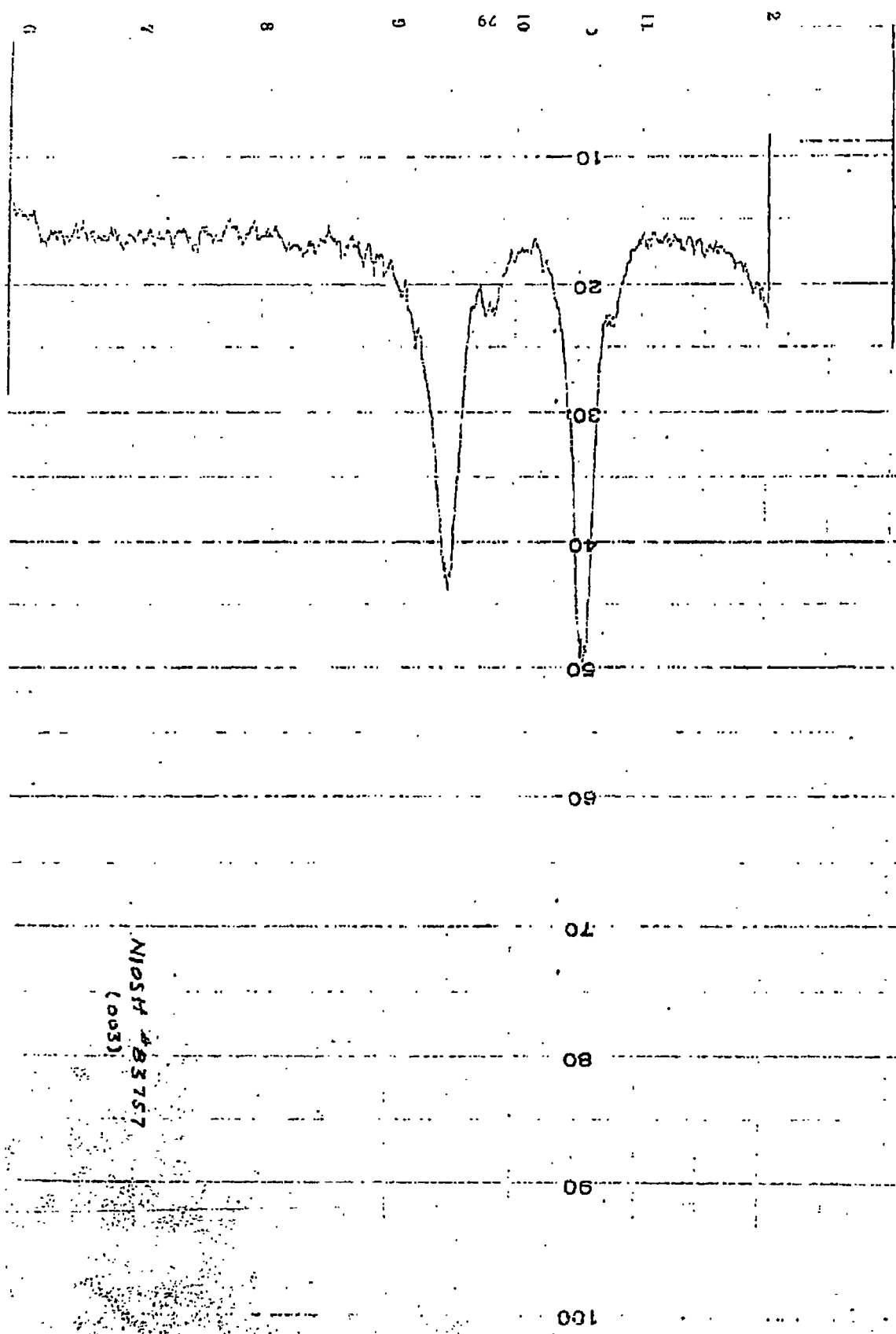
Figures 2-8 6°-12° range of 2θ for Samples 83755-83761

X-RAY DIFFRACTION DATA	
Sample No. 001	Project No. 4800
Material ALLOY 83755 Talc	
Radiation Cu	Power 42-25
Filter Ni	Scale Factor 1.000
Multiplier 2	Time Constant 2
Scan Speed 16°/min	Chart Speed 1/2" / min
Slit 1.0° - 0.25° - 1.0°	
Remarks Rotating sample	
Date 11-13-75 Operator R. J. Smith	

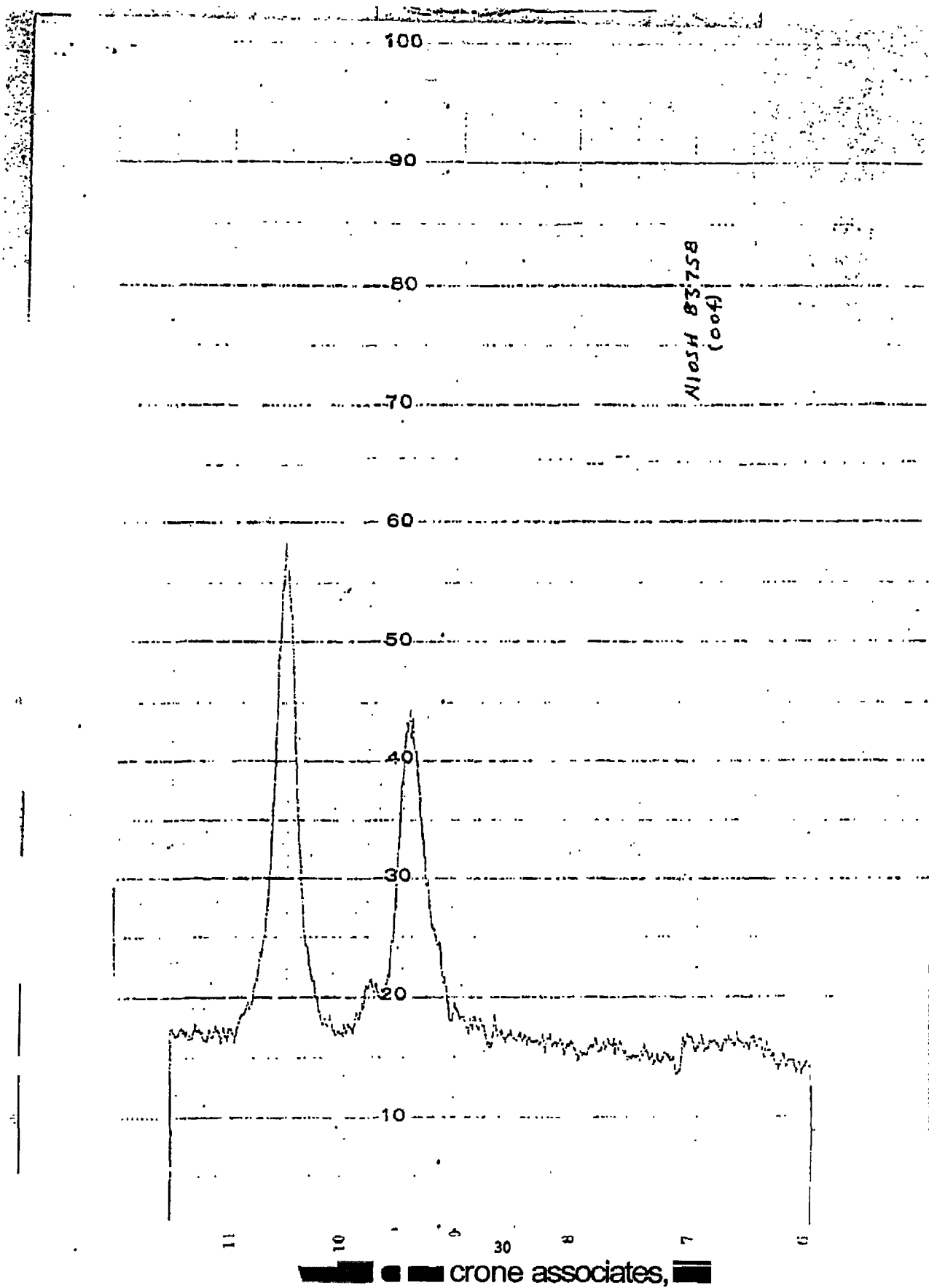


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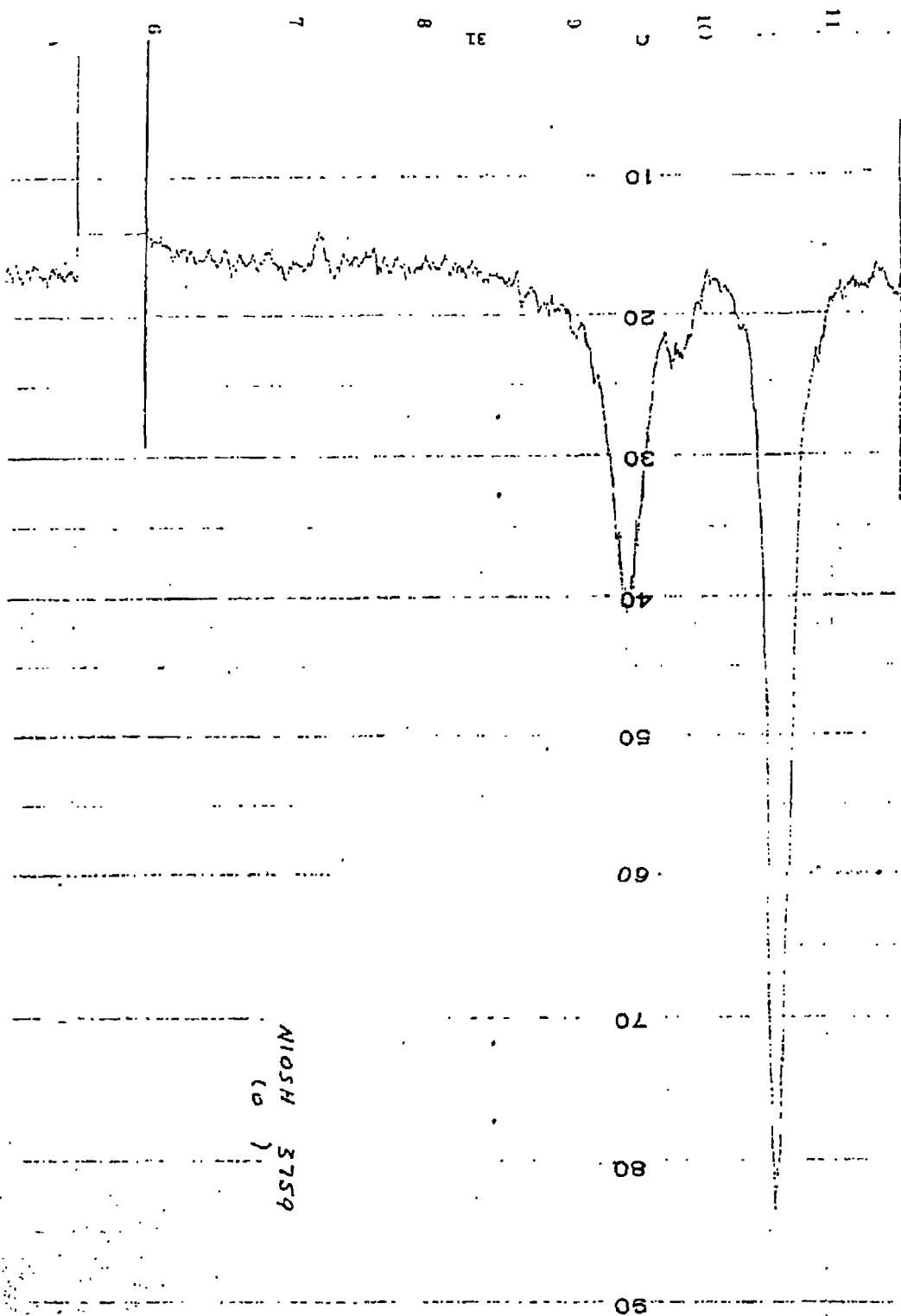


NIST # 83757
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Figure 6



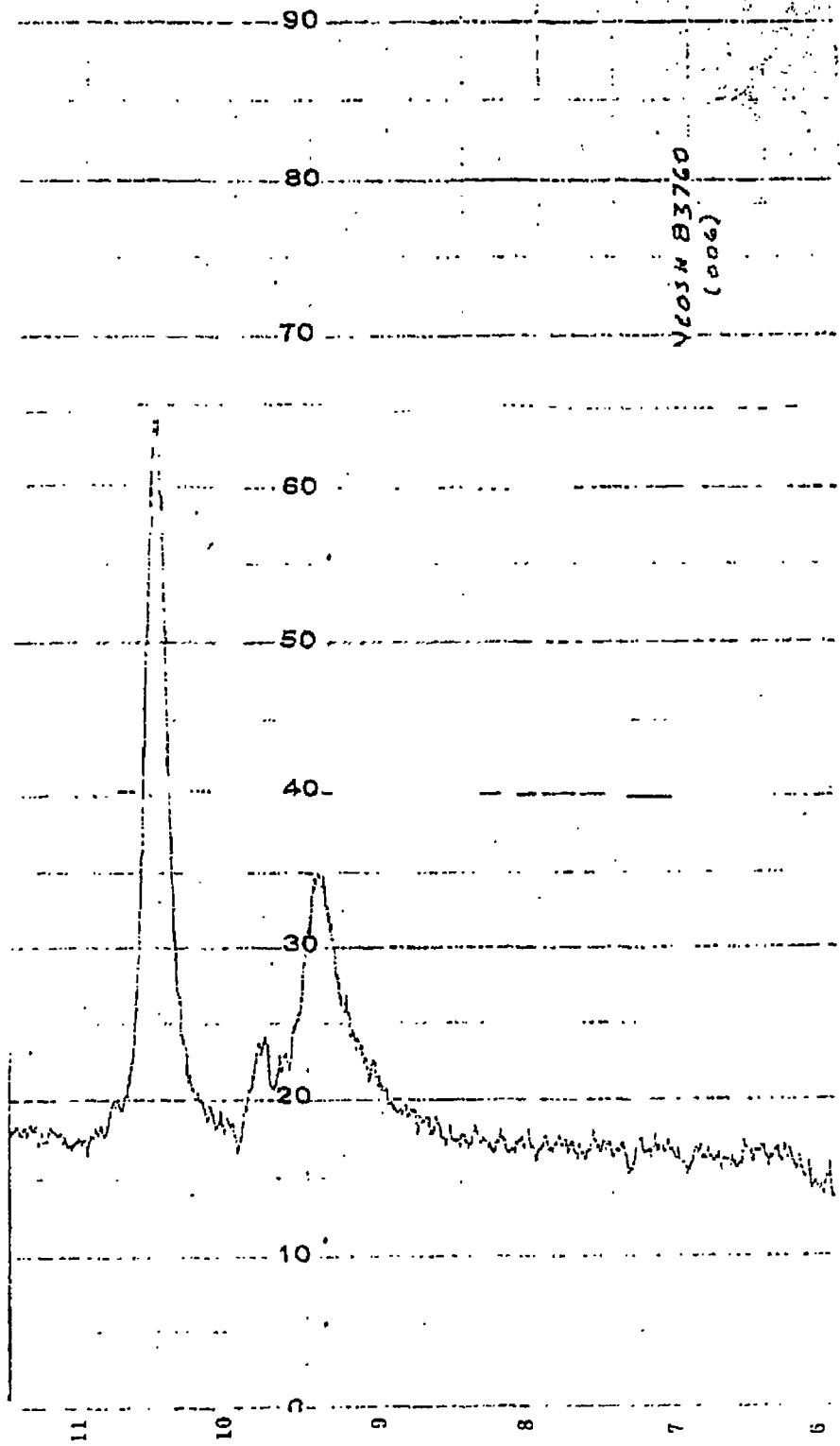


Figure 7

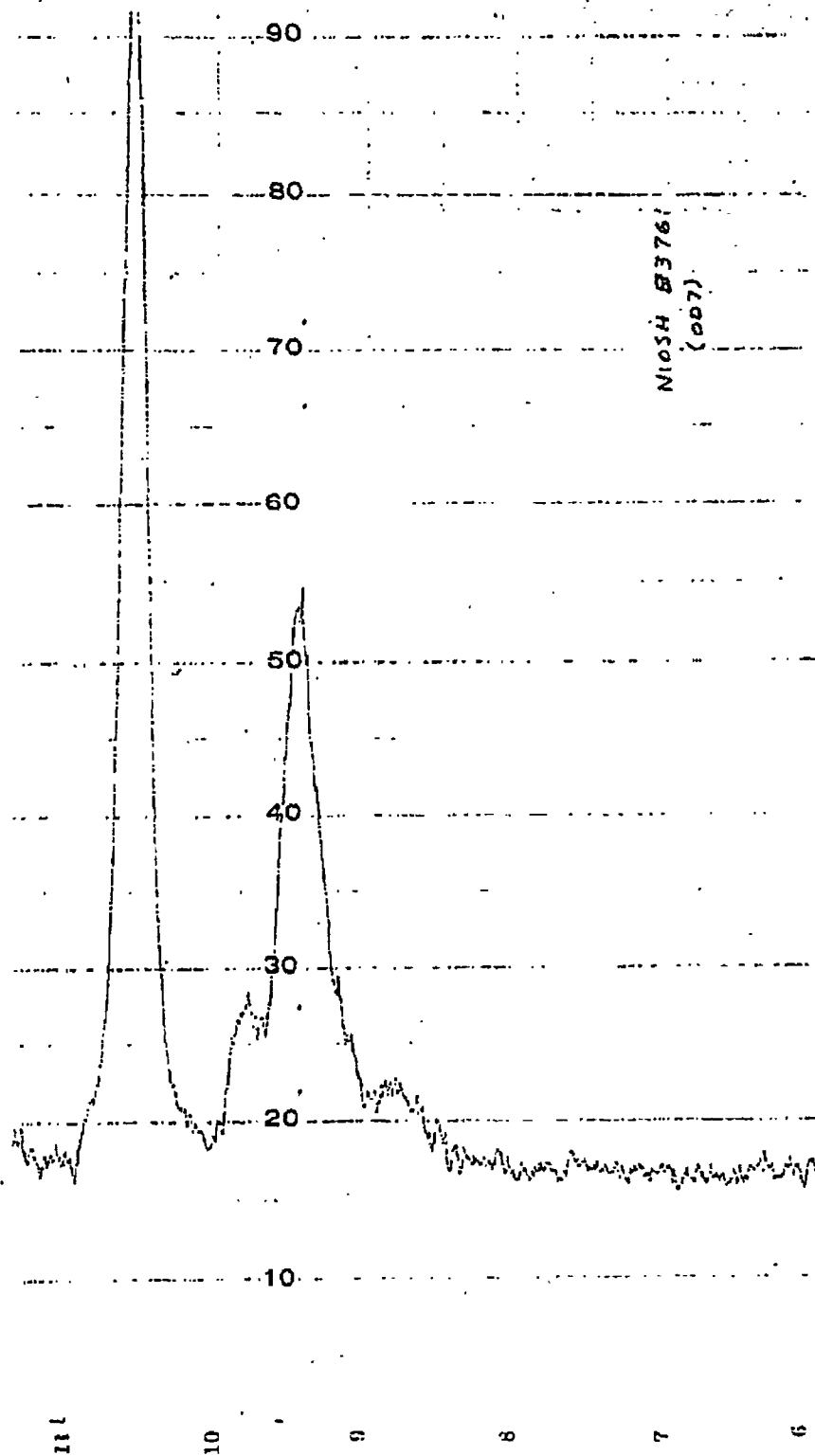


Figure 8