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walter c. mc crone associates, inc.

CONSULTING: ULTRAMICROANALYSIS • MICROSCOPY • SMALL PARTICLE PROBLEMS • SOLID-STATE CHEMISTRY

22 December 1975

Mr. Martin Abell
Room 206
1014 Broadway
Cincinnati, Ohio 45202

Dear Marty:

Enclosed are two copies of our first sample data report. Six more are nearly finished, but cannot be mailed until after the first of the year. I will return to the office on January 5.

With best wishes,

0.2
Sincerely,

Lucy B. McCrone

Lucy B. McCrone
Senior Research Scientist

LBM:de
enclosure
ref: 4800

- 0.2*
- ✓ (1) Calibration curves? ✓
 - ✓ (2) get portions of gasolines and quartz dilution standards for TEM
 - ✓ (3) Want 1/2 dozen curves used for the micro-micro

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PLAINTIFF'S
EXHIBIT
CAM-231

Data Summary, Sample No. 83755 (001)
average particle size: 2.97 μ m

mineral	detected	not detected	amount	qualified by	comments
talc	+		30-40%	XRD line scan	rolled talc "fibers" present
anthrophyllite	+		<3%	XRD step scan	identification uncertain
chrysotile		+	0		
quartz	+		2%	XRD step scan	
tremolite	+		40-50%	XRD step scan	about 3% by weight of the tremolite is fibrous
amosite		+	0		
calcite	+		trace	light microscope	
ehlorite		+	0		
crocidolite		+	0		
dolomite	+		trace	light microscope	
magnesite		+	0		
lizardite	+		10-15	XRD line scan	
antigorite		+	0		

lower limit

Particle size distribution. The particle size distribution was determined by light microscopy with a manual count based on Martin's diameter. Results are plotted in Figure 1. Since the average particle size is $2.97 \mu\text{m}$ grinding the sample is unnecessary for x-ray diffraction.

Data summary. The first ten minerals following talc in the data summary are those specified for analysis in Contract No. 210-75-0063. (These will be tabulated in that order in all subsequent Sample Data Reports). Since no chrysotile or chlorite could be found microscopically to explain the peak at 12.1° on the x-ray line scan we examined the sample for the two serpentines, antigorite and lizardite.

Talc. The weight percent of talc was calculated by difference to be between 30 and 45%. Some fibrous talc is present as rolled up sheets. In Cargille high dispersion liquid 1.550 they show talc colors by dispersion staining with $\lambda_{\text{0}} = 430$ to 440 nm and $\lambda_{\text{0}} = 610-620$ nm.

Anthophyllite. Analysis for anthophyllite is somewhat hampered because we have not yet found a completely satisfactory standard to use in preparing calibration curves for x-ray diffraction and dispersion staining. In general, the dispersion staining colors of anthophyllites are close to those of tremolites and the two are distinguishable only by the extinction positions of the $\alpha-\gamma$ view: tremolites show oblique extinction of from about 10° to about 21° , while anthophyllites show parallel extinction.

We found a very few particles in the sample which appeared to have parallel extinction on the $\alpha-\gamma$ view but complete confidence is lacking because there were too few particles to be sure. The three principal x-ray diffraction lines for the

most reliable anthophyllite pattern in the ASTM file (16-401) occur at 10.61°, 27.59° and 29.16°. Another pattern in the ASTM file (9-455) gives the same lines as 10.70°, 27.50° and 29.26°. The UICC standard sample contains a major proportion of anthophyllite, confirmed optically, as well as various impurities. A line scan of the appropriate regions gives the three anthophyllite lines at 10.85°, 27.70° and 29.45°. Ignoring possible instrumental errors of our own we must assume that peaks within the ranges 10.61°-10.85°, 27.50°-27.70° and 29.16°-29.45°, could be due to anthophyllite. Tremolite overlaps in the regions around 10.55° and possibly 27.25°. Calcite overlaps at 29.40°.

Step scans of these regions are given in Figure 2. Anthophyllite peak centers are marked in red, tremolite in green. We cannot say that these are in fact anthophyllite peaks but they could be, especially since a peak appears in each of the three regions. Without calibration curves we only suggest the quantity represented by the peaks as less than 3%. The optical results suggest a lower value.

Serpentines. The peak at 12.1° in the line scan, Figure 5, is not due to chrysotile but to the nonfibrous serpentine, lizardite. No chrysotile fibers were found by dispersion staining in samples mounted in Cargille high dispersion liquid 1.550. Particles showing λ_0 's of 540 and 610 nm were identified as lizardite rather than antigorite, which shows 500 and 520 nm under the same conditions. The quantity of lizardite was estimated to be 10-15% from the x-ray line scan.

Quartz. The dispersion staining colors for quartz and lizardite are different by only 10 to 40 nm in the high dispersion Cargille liquid of choice, 1.550, thus they are difficult to distinguish optically, since they have similar morphology. They can be so distinguished but the procedure is slow.

The strongest x-ray diffraction peak, at 26.84° , is relatively free of interferences. Step scanning, gives the amount of quartz as about 2% (see Figure 3).

- calibration curves?

Tremolite. So much tremolite is present, 40-50% by weight estimated from the x-ray line scan (Figure 5), that step scanning was unnecessary. Step-scanned tremolite peaks at 10.55° and 27.26° are shown, however, in Figure 1.

Using dispersion staining the proportion of fibrous tremolite was estimated at 3% of the total tremolite. Because of the great differences in weight between the massive particles and the fibers the percentage could not be derived from fiber counts. It was obtained by estimating areas occluded by fibers and by massive particles, then converting to volume (weight) percent by dividing the percent fibers by four.

For ten fields examined at 200X the averaged percent of the tremolite present as fibers by area was 13.6%, which would be about 3% by volume.

Other minerals. A few particles of calcite were identified optically by their dispersion staining colors and refractive index relative to the mounting media in Cargille Liquids 1.605, 1.650 and 1.670. The principal calcite x-ray peak at 29.40° is buried in the presumed anthophyllite peak (Figure 2). Not enough calcite was observed optically to account alone for this peak.

A few particles of dolomite but no magnesite were detected in Cargille liquid 1.670 by dispersion staining.

X-ray line scans. The total sample is represented by Figure 5, the x-ray line scan from 2θ of 5° to 70° . Conditions were: Cu radiation with Ni filter, at 42 kV and 25 ma. Scale factor was 1000, time constant was 2, multiplier 2, scan speed 1° per minute, chart speed $1/2$ inch per minute, slits 1° , $0.024''$, $1''$

The sample was rotated in an aluminum holder which contributes a large peak at 38.5° and a smaller one at about 44.5°. Alignment of the goniometer is checked daily using a quartz standard. A slower scan of the region between 6° and 12° was also run for better resolution. This scan, Figure 4, was made at the same operating conditions except that the scan speed was 1/2° per minute.

Respectfully submitted,

Jan N. Stewart

for

Lucy B. McCrone
Senior Research Scientist

LBM:fe
ref: 4890

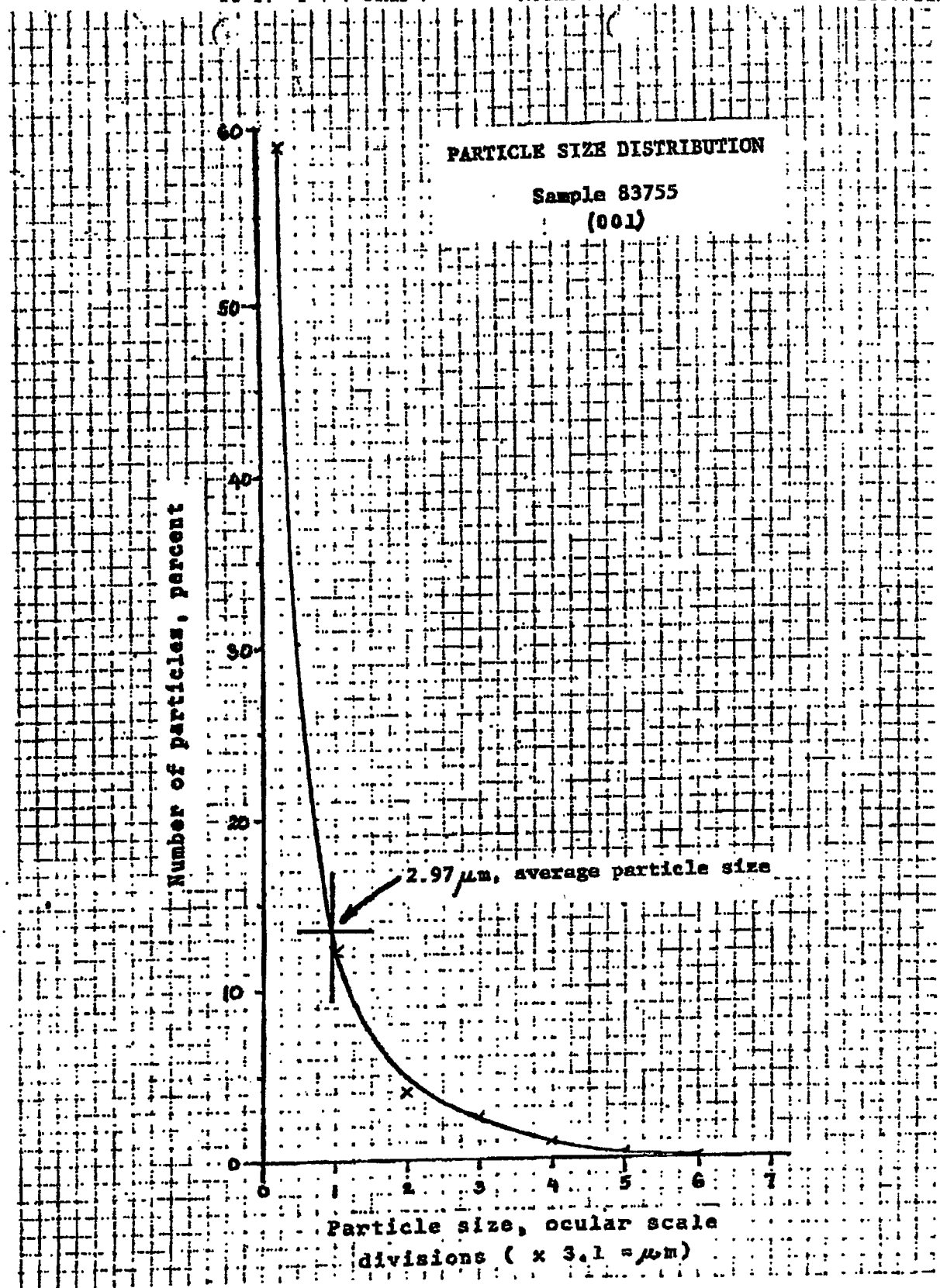
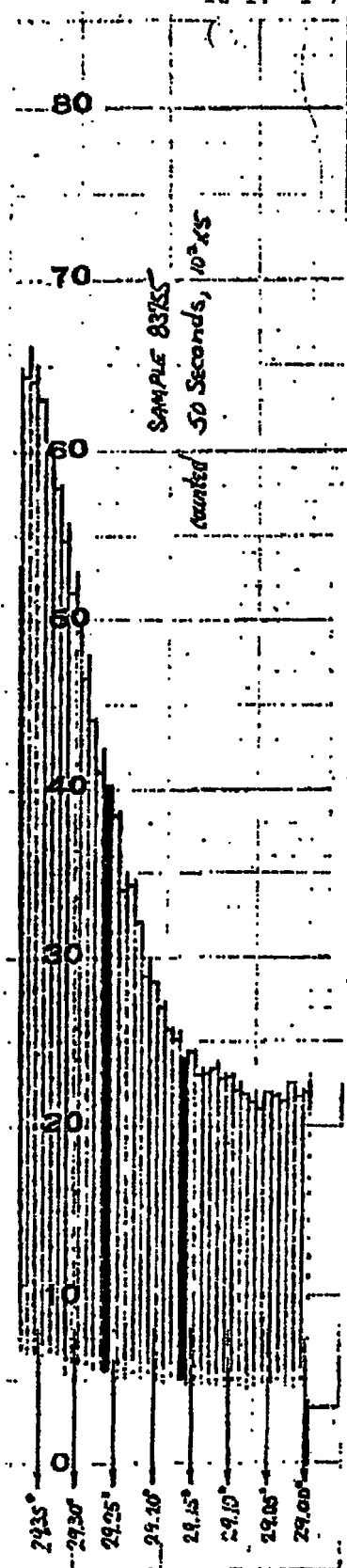


FIGURE 1.



Instrumental conditions:

500 counts/sec., 8 second time
constant, 10, 20 or 50 second
counts, 0.01° 2θ steps, 1/8"/min.
c chart speed, 0.024" slit.

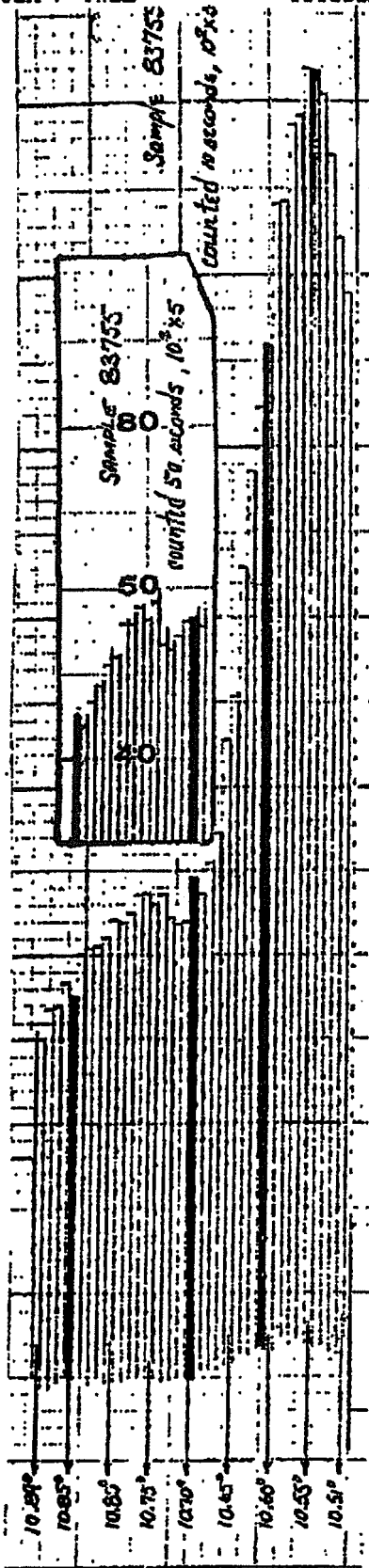
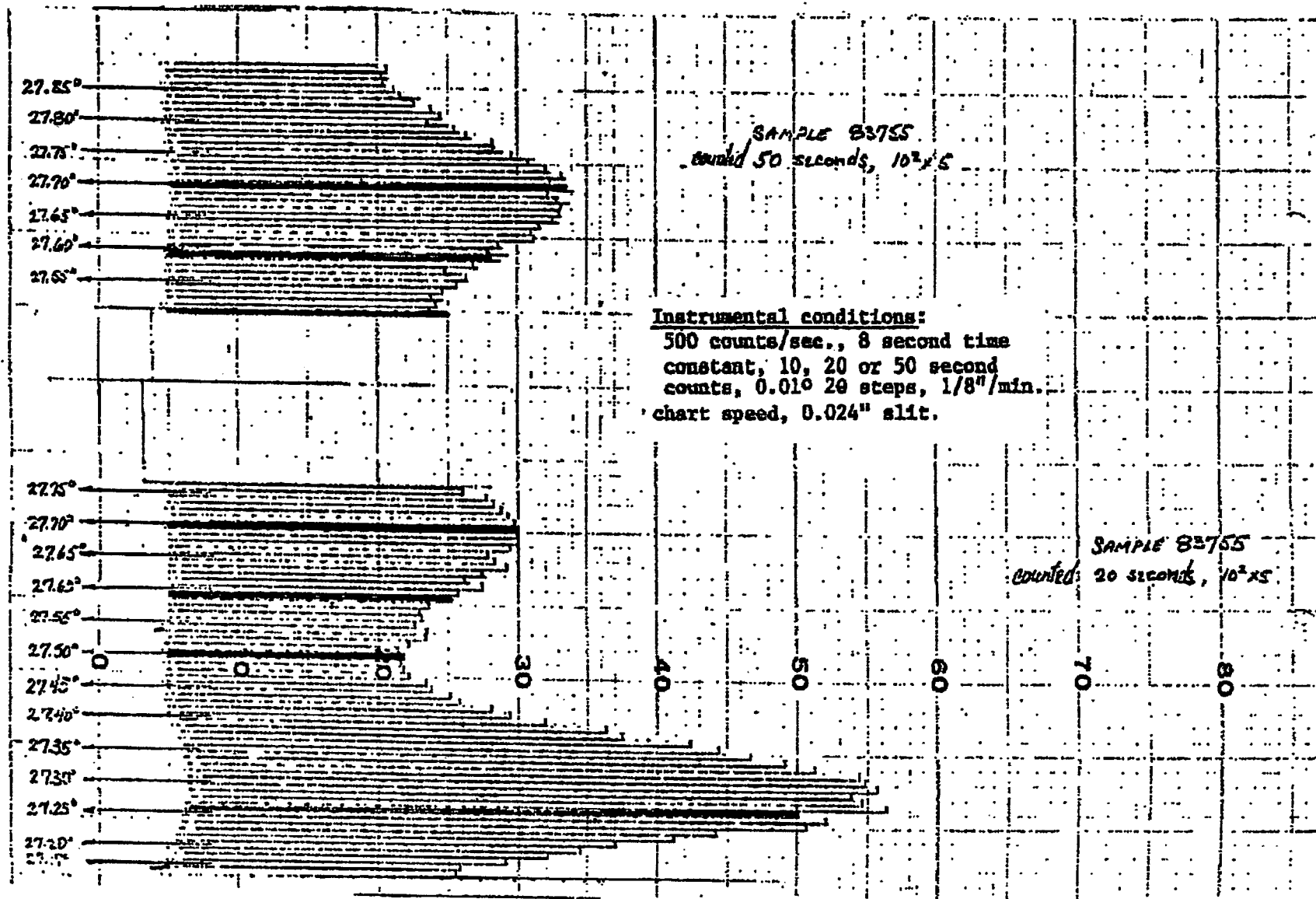
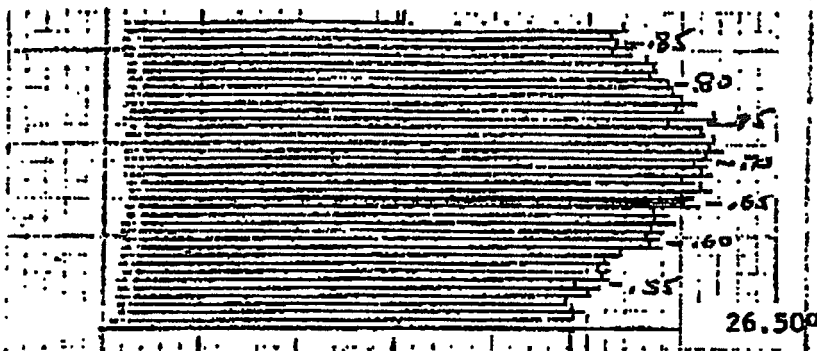


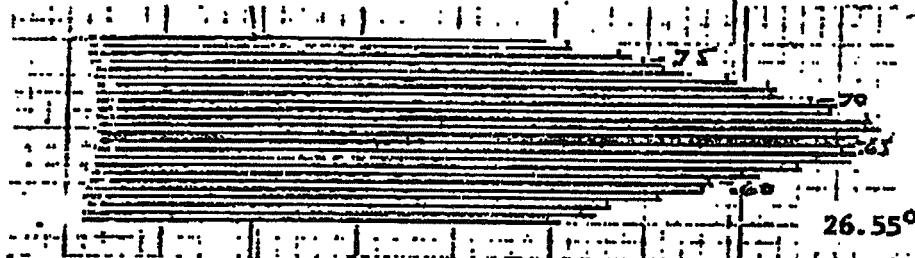
FIGURE 2A. X-ray step scans at
anthophyllite lines in the regions
of 10° and 29° 2θ.

FIGURE 2B. X-ray step scans at
anthophyllite lines near $27^{\circ} 2\theta$.

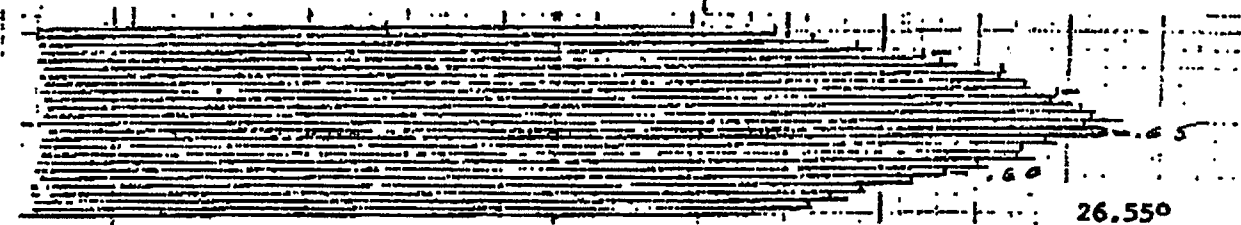




Standard: 1% quartz in talc



Sample 83755 (001): 2% quartz, approximately



Standard: 3% quartz in talc

Instrumental conditions:

500 counts/sec., 8 second time
constant, 50 second counts,
steps of 0.01° 2θ, 1/8" / min.
chart speed, 0.024" slit.

FIGURE 3.

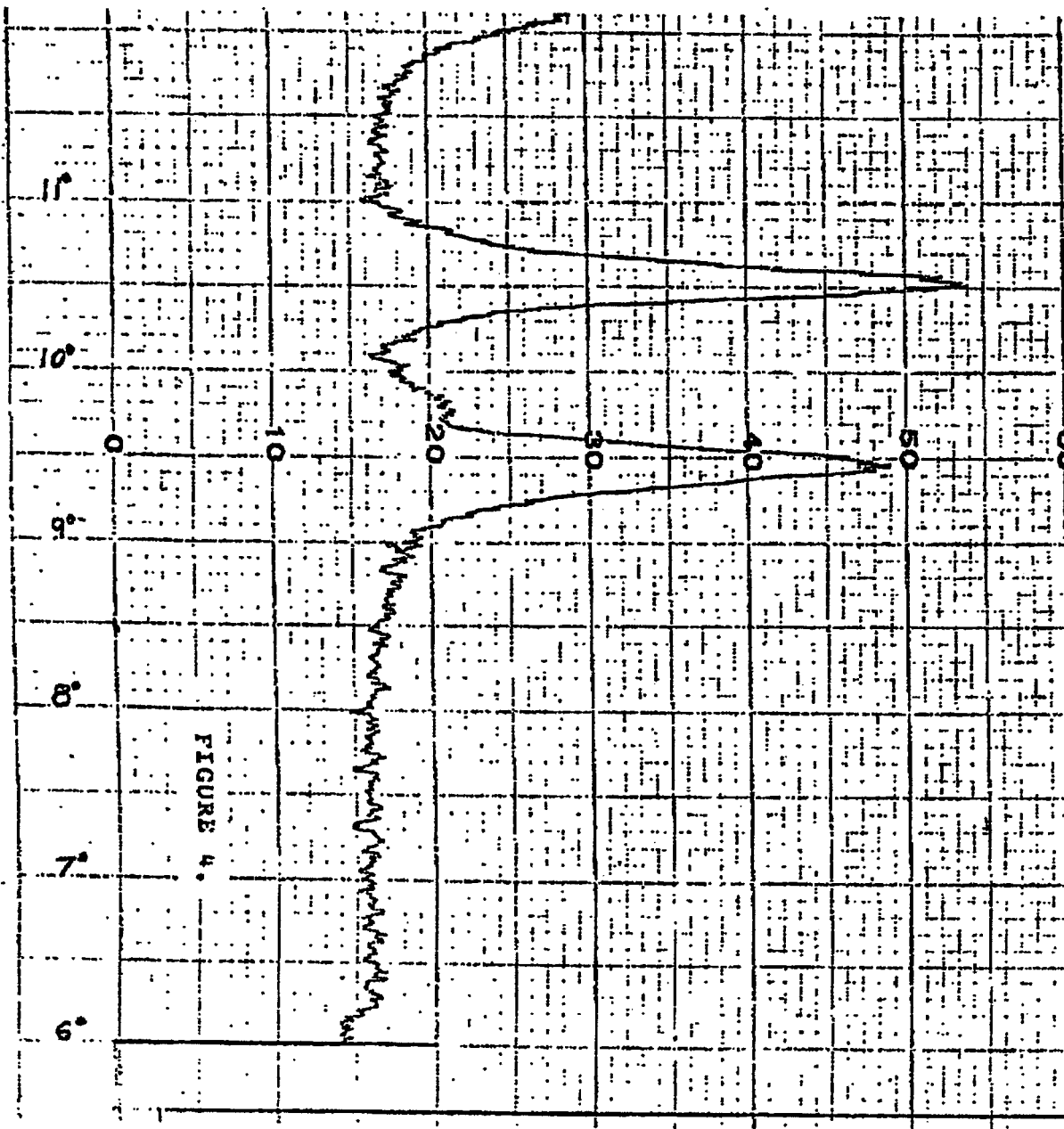


FIGURE 4.

X-RAY DIFFRACTION DATA

Sample No. 00 Project No. 4800
 Material NIOSH B3755 Tals
 Radiation Cu Power 62-25
 Filter Ni Scale Factor 1.00
 Multiplier 2 Time Constant 2
 Scan Speed 1/4°/min Chart Speed 1/4" / min
 Slit 1° - 0.25" - 1°
 Remarks Rotating s'ple
 Date 11-13-75 Operator RJ. Hinch