COLORADO SCHOOL OF MINES RESEARCH INSTITUTE
P.O. Box 112
GOLDEN, COLORADO 80401

October 27, 1972

Project C10704

Dr. Al Goudie
5 Finley Road
Edison NJ 08817

Dear Dr. Goudie:

In compliance with your request I have made X-ray diffraction step scans on Baby Powder Talc Samples 108T and 109T for the purpose of determining if serpentine (possibly chrysotile) is present. It is my understanding that a contention has been made that these samples contain approximately 3% chrysotile.

The results of these studies do not substantiate this. They do show that the sample contains chlorite which give similar, but distinguishable X-ray diffraction peaks. If the chlorite peaks were mistakenly assumed to result from the presence of chrysotile, then one would mistakenly assume that the samples contain about 3% chrysotile based on the diffraction peak intensity resulting from the addition of 3% chrysotile to the samples.

Asbestosform chrysotile is but one species of the mineral serpentine. Other species are not asbestosform. However, all species give similar X-ray diffraction patterns so that X-ray diffraction can only indicate the presence or absence of serpentine. If X-ray diffraction indicates the presence of serpentine, other methods must be used to determine if it is chrysotile. However, according to the FDA guidelines, if a talc sample yields no diffraction data which could be ascribed to serpentine (including chrysotile) then the talc is regarded as acceptable relative to chrysotile content.

The most effective way to ascertain if a serpentine mineral may be present in a sample by diffraction is to first determine if the material yields a diffraction peak in the vicinity of 7 Å. This is the most intense diffraction peak for serpentine. However, this diffraction peak cannot be used indiscriminately since other non-asbestos minerals have their most intense diffraction peaks in the same region. An awareness of the geological and mineralogical associations of talc immediately suggests the chlorite minerals. They are commonly associated with talc and yield their most intense diffraction peak at essentially the same place as that of serpentine

(including chrysotile). Fortunately there is a way to distinguish between the serpentine minerals and chlorite minerals by X-ray diffraction. Chlorite gives a diffraction peak at about 14 Å and serpentine does not. Thus if a suspect 7 Å peak is observed then the sample must be checked for a 14 Å peak. If a 14 Å peak occurs, the 7 Å peak represents chlorite, not serpentine. The X-ray diffraction must be determined by accumulating counts for a few minutes at each of small increments across the peaks. Continuous counting or counting rates give neither adequate counting statistics nor sufficient peak discrimination.

Step scanning of several splits of Samples 108T and 109T revealed the presence of a peak in the 7 Å region as shown on the accompanying charts. However, they also show the presence of peaks in the 14 Å region which are in the proper position to show that the 7 Å peaks result from the presence of chlorite and not serpentine.

Since the contention was made that Samples 108T and 109T contain about 3% chrysotile, they were spiked with 2.7% and 2.8%, respectively, of finely milled chrysotile which was obtained from Johns-Manville. As may be seen from Charts 2 and 8 the chlorite and chrysotile peaks are distinguishable and distinctive. The chlorite peak occurs at about 7.06 Å. The chrysotile peak is quite broad and shows at least 2 maxima at approximately 7.32 Å and 7.38 Å. It was first thought that this broadening may have resulted from overgrinding, however, relatively coarse unground material gave the same pattern. It is possible that the peak broadness results from different varieties of serpentine in addition to chrysotile.

Step scans of Samples 108T and 109T spiked with 2.7% and 2.8% chrosotile, respectively, show that the area under the chrysotile peak is comparable to the area under the chlorite peaks. If one were to mistakenly assume that the 7.06 Å peak resulted from chrysotile instead of chlorite, he would calculate that the samples contain about 3% based on increased diffraction intensities after the addition of chrysotile. This would be especially true if step scanning were not used since the chlorite and serpentine peaks would not be resolved. It is probable that the contention that the two talc samples contain about 3% chrysotile is mistakenly based on the magnitude of the 7 Å chlorite peak. If this is so, it could have been avoided by step scanning the proper regions.

Sincerely,

W. T. Caneer
Assistant Manager
Mining Division

WTC/psk

Dr. A. J. Goudie
Johnson and Johnson
Research Center
501 George Street
New Brunswick, New Jersey 08901

EXAMINATION

OF

JOHNSON AND JOHNSON'S BABY POWDER

Date: 27 October 1972

MA Number: 2546

Copy 2 of 4

walter c. mccrone associates, inc.
2820 SOUTH MICHIGAN AVENUE • CHICAGO, ILLINOIS 60616

EXAMINATION OF JOHNSON AND JOHNSON'S BABY POWDER

Summary

Two samples of Johnson and Johnson's Baby Powder, batch number 108T and 109T, which correspond to the samples examined by Professor Seymour Z. Lewin of New York University on behalf of the FDA have been examined by x-ray diffraction, light microscopy, transmission electron microscopy and electron diffraction to determine whether they contain any asbestiform minerals.

Both samples contained an insignificant amount of tremolite — a few isolated crystals. Neither sample contained chrysotile.

Introduction

On behalf of the FDA, Professor Seymour Z. Lewin of New York University is examining a number of commercial talcum powders for the presence of asbestiform minerals. Two of the samples which he has examined are samples of Johnson and Johnson's Baby Powder, batch number 108T and batch number 109T. Johnson and Johnson therefore requested Walter C. McCrone Associates to examine samples from the same batches to determine whether they contained any asbestiform minerals.

Materials and Method of Conducting Tests

Two samples were submitted, identified as Johnson and Johnson's Baby Powder, batch numbers 108T and 109T.

For x-ray diffraction examination, the samples were examined on a Phillips-Norelco verticle diffractometer using CuKα radiation and a scanning speed of 1° per minute. The dispersion staining technique was used for the light microscopical examination and the electron microscopy-electron diffraction examination was carried out using procedures previously described (MA report 2330-1; dated 10 August 1971).

Results

X-ray Diffraction

The diffractograms were carefully examined in the vicinity of the major peaks of chrysotile and tremolite. Neither mineral was present. The presence of peaks in the vicinity of 12.0-12-5° 2θ, the region in which one of the principal lines of chrysotile may be found, was correlated with peaks in the vicinity of 6° 2θ and are thus attributable to chlorites. No significant peaks were observed in the 24° region which would be required were chrysotile present.

Light Microscopy

Using the dispersion staining technique and a liquid of refractive index 1.550, the samples were examined for chrysotile particles and fibers, but none could be found. Using a similar technique with a liquid of refractive index 1.605, the samples were similarly examined for the presence of tremolite and a few individual crystals were found, some rod shaped.

Electron Microscopy and Electron Diffraction

Several electron microscope grids from both samples were examined in their entirety and although some fibers were observed these were shown by electron diffraction to be shards of talc or rolled talc. No chrysotile fibers were found.

Conclusion

A detailed examination of two samples of Johnson and Johnson's Baby Powder, batch numbers 108T and 109T has shown this material to be substantially free of asbestiform minerals. A few tremolite rods were observed in both samples. No chrysotile has been detected.

Respectfully submitted,

Ian M. Stewart

Ian M. Stewart
Manager, Electron Optics Group

University College, Cardiff

Postal Address: University College, Newport Road, Cardiff CF2 1TA.
Telephone Cardiff 44211 Telegrams: Coleg Cardiff

From Dr. F.D. Pooley

Department of Mineral Exploitation

AIR MAIL

FDP/MM

8th November, 1972.

Dr. A.J. Goudie,
5, Finley,
Edison,
New Jersey 08817,
U.S.A.

Dear Dr. Goudie,

We have extensively examined the sample 108/T powder for chrysotile asbestos but can find no evidence of any of the mineral at all. Both electron microscope and X-ray techniques have been employed and we have also applied a chrysotile concentrating technique which we have developed to the sample which enables us to definitely demonstrate chrysotile at the 0.10% level and nothing was found. I will be communicating a number of simple tests to you in the near future for demonstrating chrysotile at low levels.

Yours sincerely,

F.D. Pooley

X-ray Study of Johnson & Johnson's Baby Powder
Retain 108T (4-17-72)

Gordon E. Brown
Princeton University

Retain 108T (4-17-72) of Johnson & Johnson's Baby Powder was examined by slow, continuous scanning x-ray techniques. A Norelco vertical diffractometer with a LiF crystal monochrometer and sample spinner was used in this work. Other pertinent experimental conditions were as follows: CuKα radiation (40KV, 20mA); scan speed = 1/4° 2θ/min.; scale factor = 4; time constant = 4; 1° slits; scan range = 5-80° 2θ; PHA (12V baseline, 12V window); amount of powder = 0.1 gr. (< 325 mesh).

The diffraction tracing is attached to this report and a list of d spacings is given in Table 1. Twenty-four of the recorded peaks have been assigned to talc using cards 19-770A and 13-558 from the ASTM powder diffraction file. Seven additional peaks were also recorded and all but one have been attributed to chlorite, magnesite, chalcopyrite and rutile. The unknown peak represents a d-value of 1.48Å.

No evidence of chrysotile or tremolite was found in the x-ray data outlined above. The two strongest chrysotile peaks (7.31 and 3.65Å) as well as the three strongest tremolite peaks (8.38, 3.12 and 2.70Å) were unobserved. The peak at 3.12Å is attributed to talc ($006, 11\bar{5}$) and cannot be a tremolite peak. Peaks due to CuKβ were also searched for but were not found.

It is concluded that retain 108T (4-17-72) from Johnson & Johnson's Vermount talc mine is a very pure talc with less than 5% impurity due mainly to chlorite and magnesite. A careful search for chrysotile and tremolite impurities proved negative.

Gordon E. Brown

Table 1: X-ray Data for Johnson and Johnson's Baby Powder Retain 108T (4-17-72)

Mineral	hkl	d(Å)	2θ(deg.)
Talc	002	9.45	9.36
Chlorite	002	7.14	12.40
Talc	004	4.69	18.93
Talc	020,11$\bar{1}$	4.57	19.42
Talc	022	4.13	21.50
Chlorite	004	3.56	25.0
Rutile(?)		3.26	27.33
Talc	006,11$\bar{5}$	3.12	28.58
Magnesite	104	2.75	32.58
Talc	130	2.64	34.00
Talc	200,13$\bar{2}$,131	2.60	34.50
Talc	13$\bar{3}$,132,11$\bar{7}$	2.49	36.08
Talc	20$\bar{4}$	2.45	36.73
Talc	008	2.34	38.46
Talc	many including 134	2.28	40.50
Talc	13$\bar{6}$	2.10	43.18
Talc	136	1.94	46.80
Talc	0.0.10	1.87	48.62
Talc	24$\bar{2}$	1.73	52.93
Talc	24$\bar{4}$,138	1.69	54.34
Talc		1.65	55.90
Chalcopyrite(?)		1.58	58.37
Talc	0.0.12,31$\bar{7}$	1.56	59.21
Talc	060,33$\bar{2}$	1.53	60.52
Talc	330	1.51	61.30
Unknown		1.48	62.85
Chlorite	13$\bar{9}$,208	1.42	65.65
Talc	2.0.10	1.39	67.18
Talc	1.3.$\bar{12}$	1.39	67.53(K_{α_1})
Talc	0.0.14	1.34	70.38
Talc	26$\bar{4}$	1.30	72.90

5 6 7 8 9

5 6 7 8 9

5 6 7 8 9

4
3
2
1
0
4
3
2
1
0
4
3
2
1
0

TALC (CC)
9.36° 2θ
9.45Å
IF TREMOLITE WERE PRESENT
THERE SHOULD BE A PEAK (I=10)
AT ~ 10.55° 2θ (d = 8.38Å)
CHRYSOTILE (CC)
IF TREMOLITE WERE PRESENT
THERE SHOULD BE A PEAK (I=10)
AT ~ 10.55° 2θ (d = 8.38Å)

1
2
3
4
1
2
3
4
1
2
3
4

CHRYSOTILE (002) SHOULD OCCUR HERE IF PRESENT.
$(I = 100)_6$ $(d = 7.34 Å)$

5 8 9

NOT PRESENT.

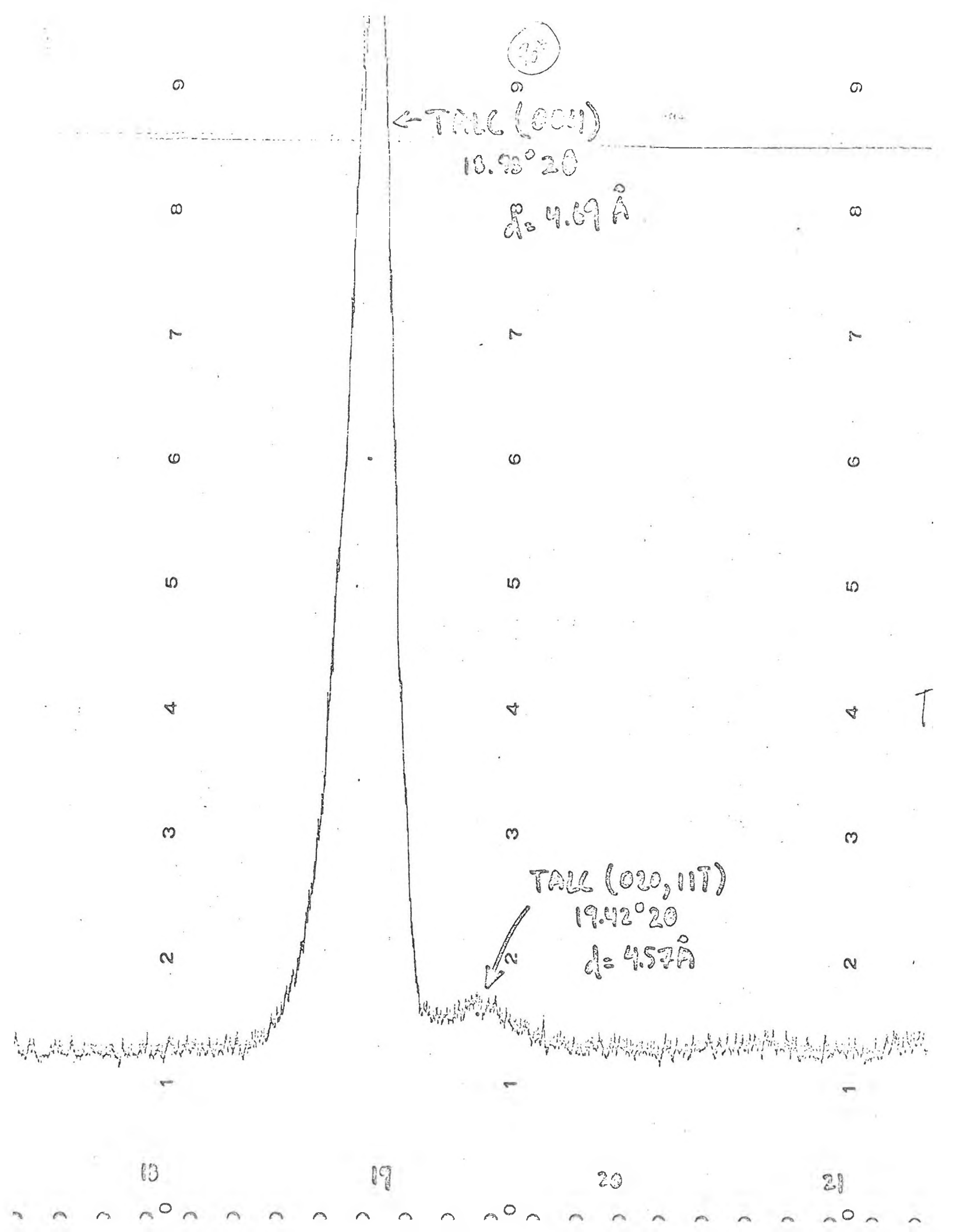
5 6 7 8 9

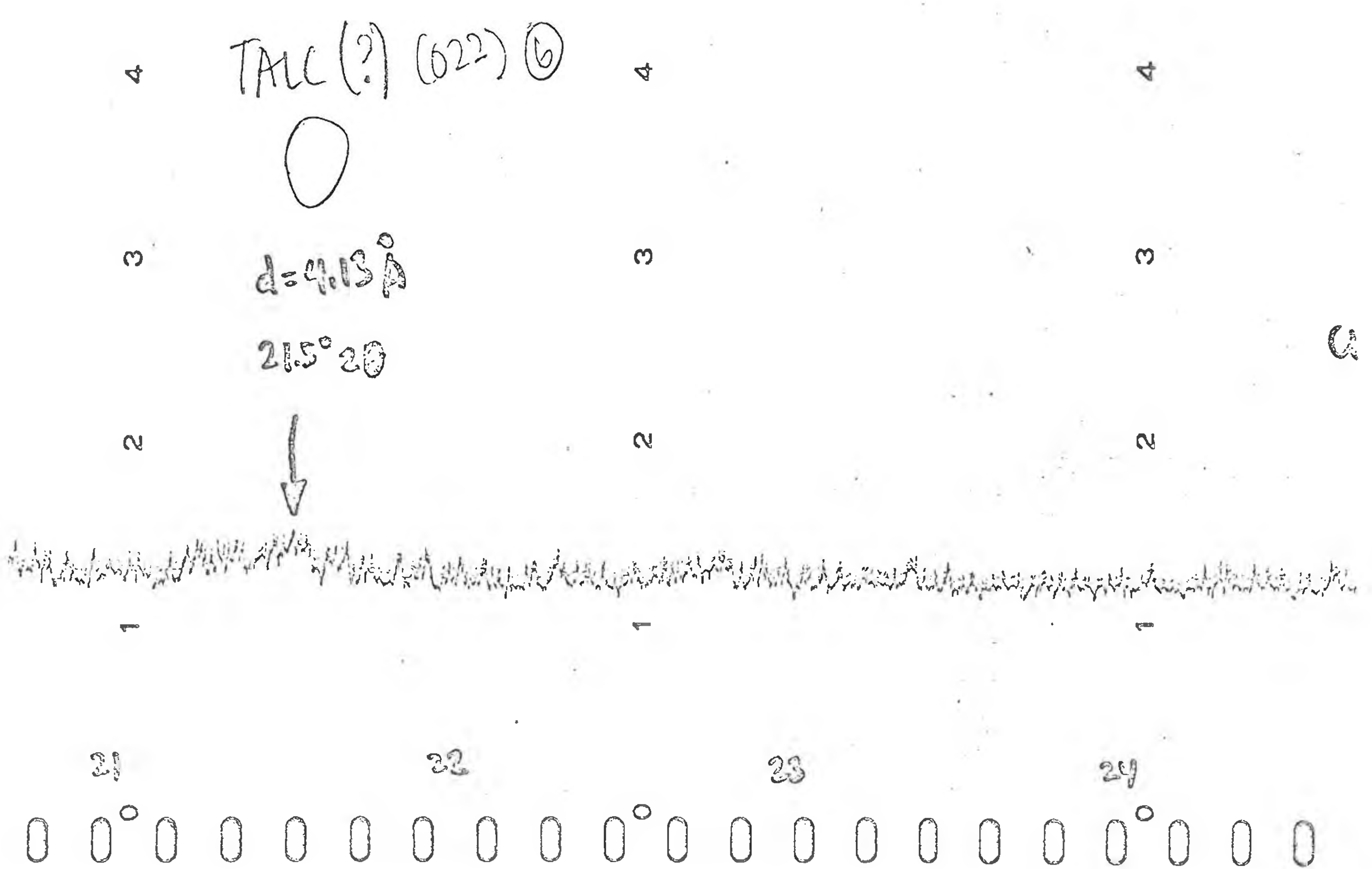
9 8 7 6 5 4 3 2 1

9 8 7 6 5 4 3 2 1

9 8 7 6 5 4 3 2 1

15 16 17 18


TALC (004)
18.98° 2θ
d: 4.69 Å
TALC (020, 111)
19.42° 2θ
d: 4.57 Å
18
19
20
21


TALC (?) (622) 6
d=4.13 Å
21.5° 2θ
21
22
23
24

5 6 7 8 9

5 6 7 8 9

5 6 7 8 9

50
Chromite (esq)
≈ 25.0°30
d = 2.56 Å
24
25
26
27

Rutile
(NOT TRIPLICATE)
d= 3.26 Å
~ 27.33° 2θ
TALC (001)
28.58° 2θ
d= 3.12 Å
26
27
28
29
°
°

5 6 7 8 9

5 6 7 8 9

5 6 7 8 9

4
3
2
1
0
4
3
2
1
0

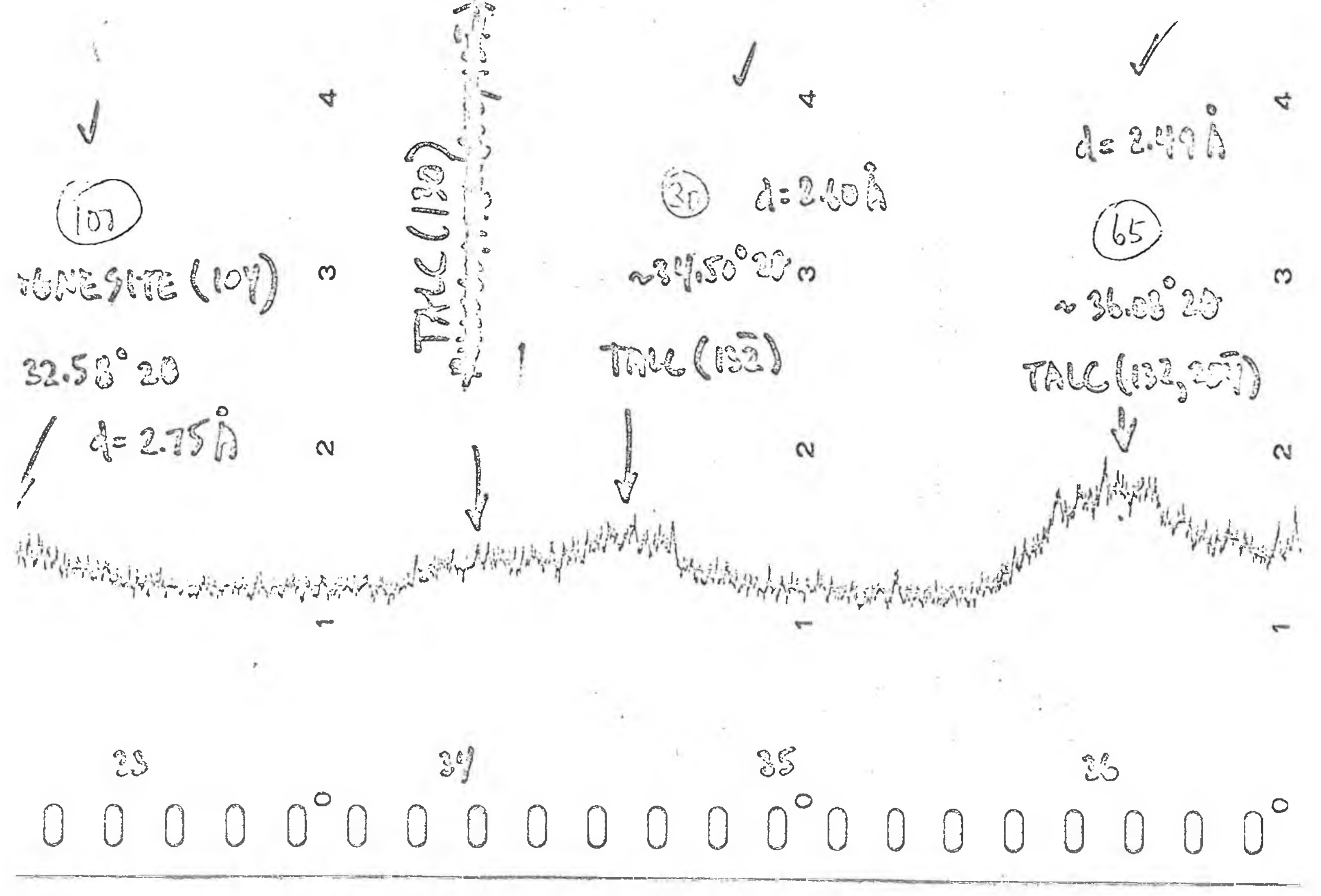
(101)
MAGNESITE (101)
32.58° 2θ
d= 2.75 Å
30
31
32
33

5 6 7 8 9

34.00° 20 (18)

5 6 7 8 9

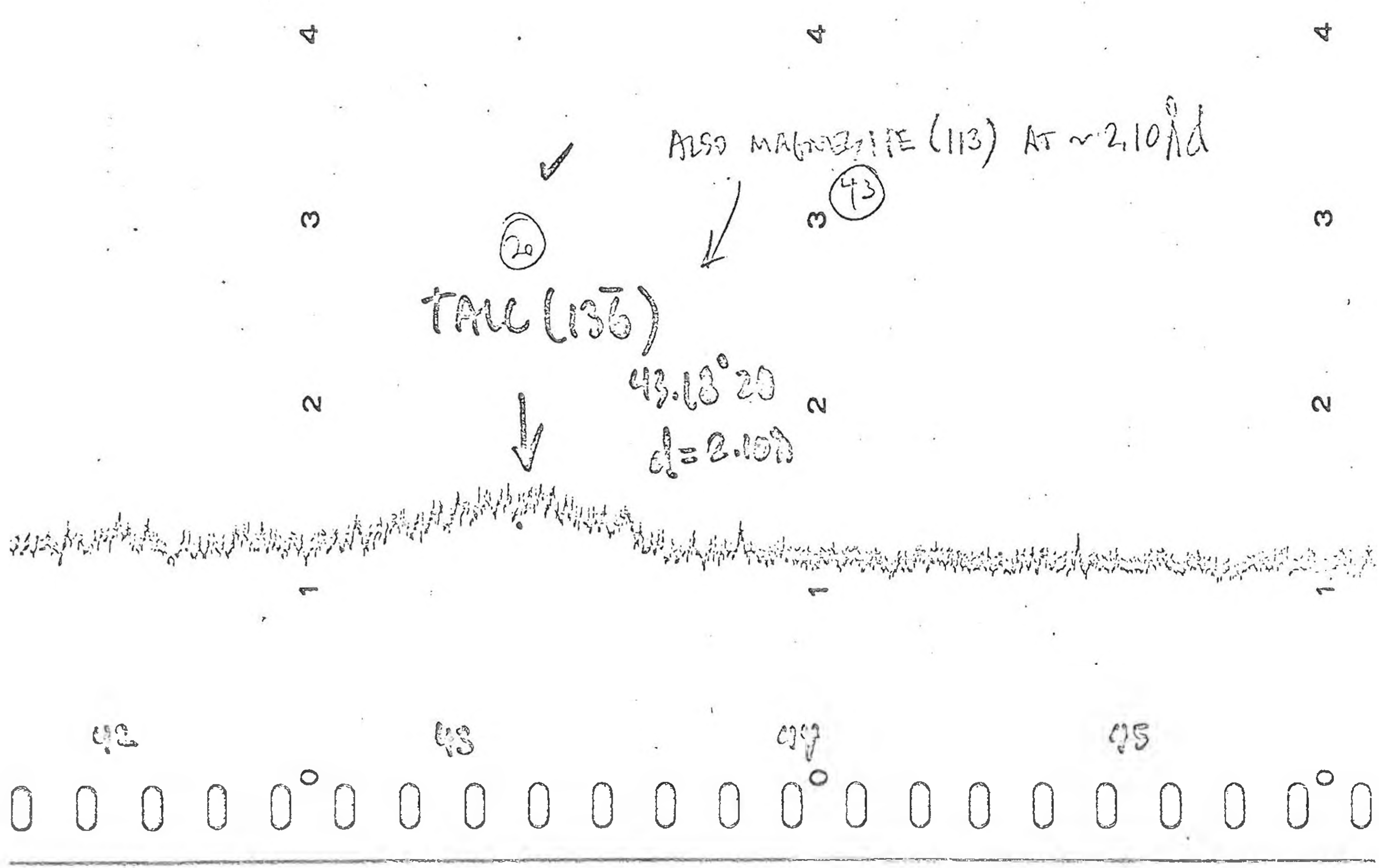
5 6 7 8 9



107
32.58° 2θ
d= 2.75Å
TALC (130)
30
~34.50° 2θ
d= 2.60Å
TALC (132)
65
~36.03° 2θ
d= 2.49Å
33
34
35
36

d= 2.49 Å
(65)
~ 36.08° 2θ
LC (132, 207)
TALC (117)
d=2.45 Å
~ 36.73° 2θ
(16*)
TALC (008)
~ 36.96° 2θ
d= 2.34 Å
35
37
38
39
°

TALC (134)
40.5°2θ
d = 2.23 Å


ALSO MAGNETITE (113) AT ~2.10Å d
43
20
TALC (13̄6)
43.18° 2θ
d = 2.10Å
42
43
44
45
4
3
2
1
0

5 6 7 8 9

5 6 7 8 9

5 6 7 8 9

(6B)
TALC (125)
d- 1.94 Å
46.60° 2θ
(45)
TALC (0.0.
~ 19.6
d: 1
96
97
98
97

40°

TMC (0.0.10)

2θ 48.62 (K_{α_1})

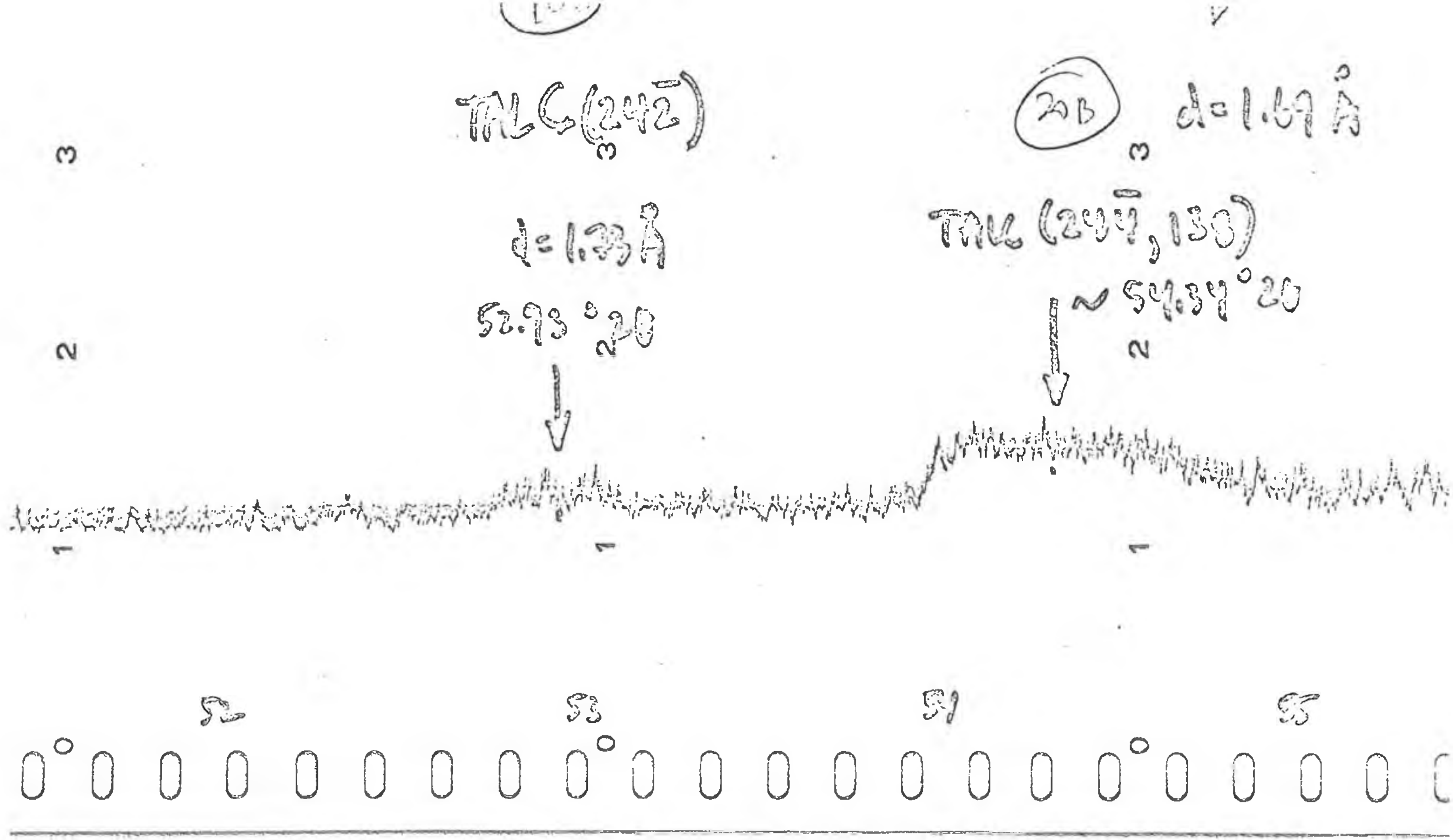
d = 1.87 Å

4 5 6 7 8

4 5 6 7 8

4 5 6 7 8

ALSO MARKED (UL) AT ~



TALC (24$\bar{2}$)
d=1.73 Å
52.93 °2θ
2B
d=1.69 Å
TALC (24$\bar{4}$, 130)
~54.34 °2θ
52
53
54
55

8
7
6
5
4
3
2
1

ASHIE(116) AT ~1.70Å

d=1.69 Å

130)
1.34°20

TALC

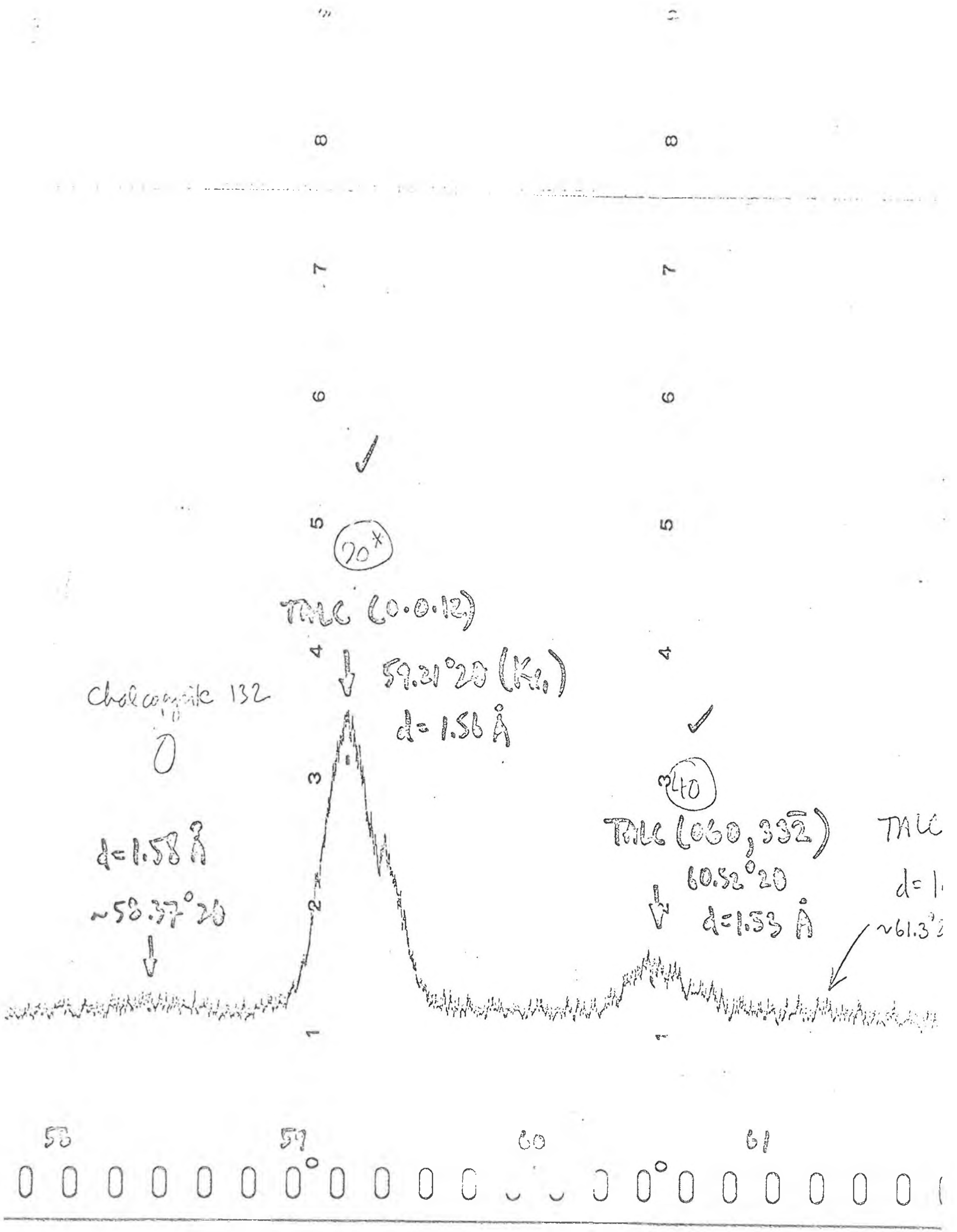
d=1.65 Å
~55.90°2θ

Chalcopyrite

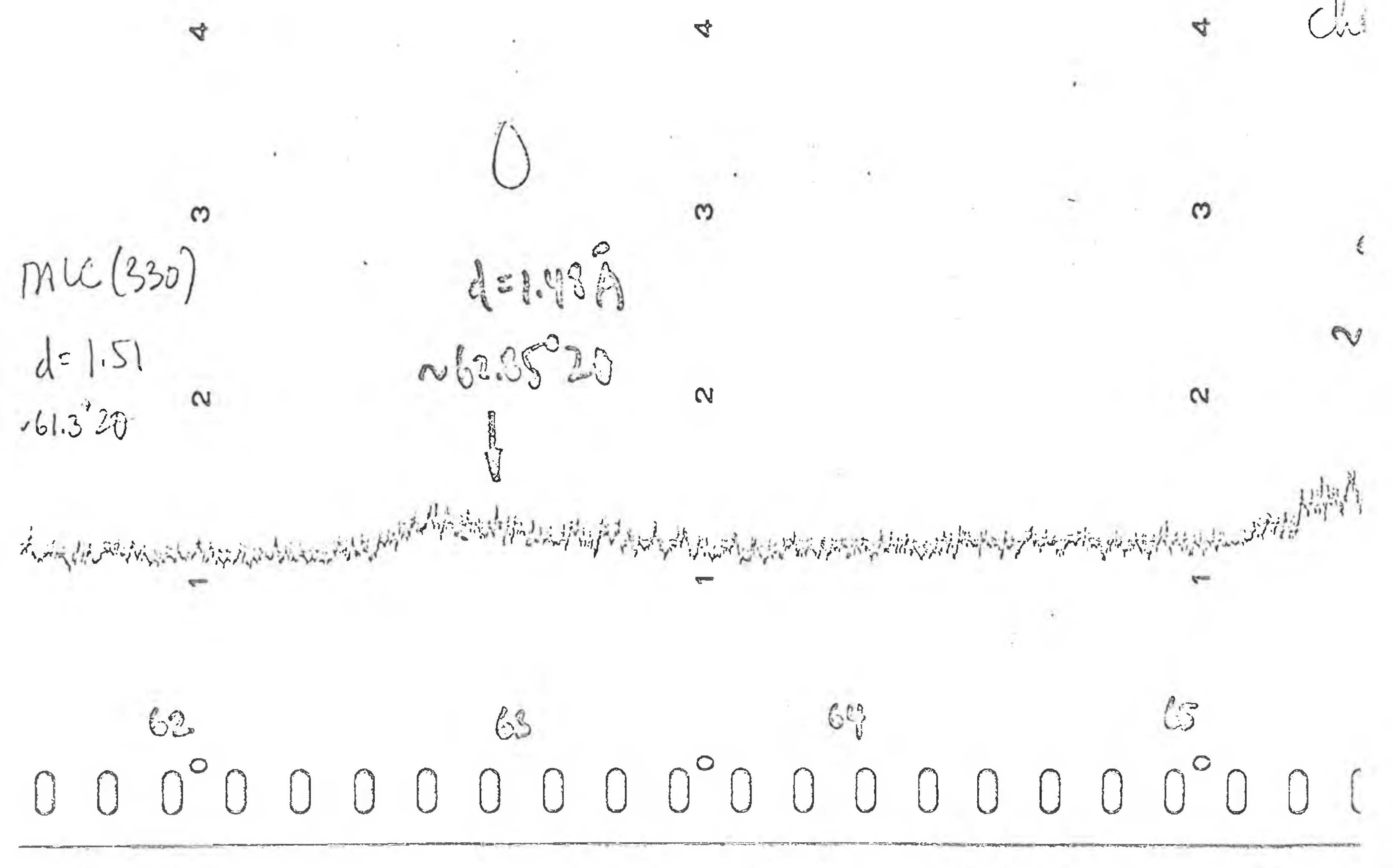
d=1.53 Å
~58.37°2θ

55 56 57 58

°



Chalcopyrite 132
d=1.58 Å
~58.37°2θ
TALC (0·0·12)
59.21°2θ (Kα1)
d= 1.56 Å
70*
340
TALC (060, 33$\bar{2}$)
60.52°2θ
d=1.53 Å
TALC
d= 1.
~61.3°2
58
59
60
61



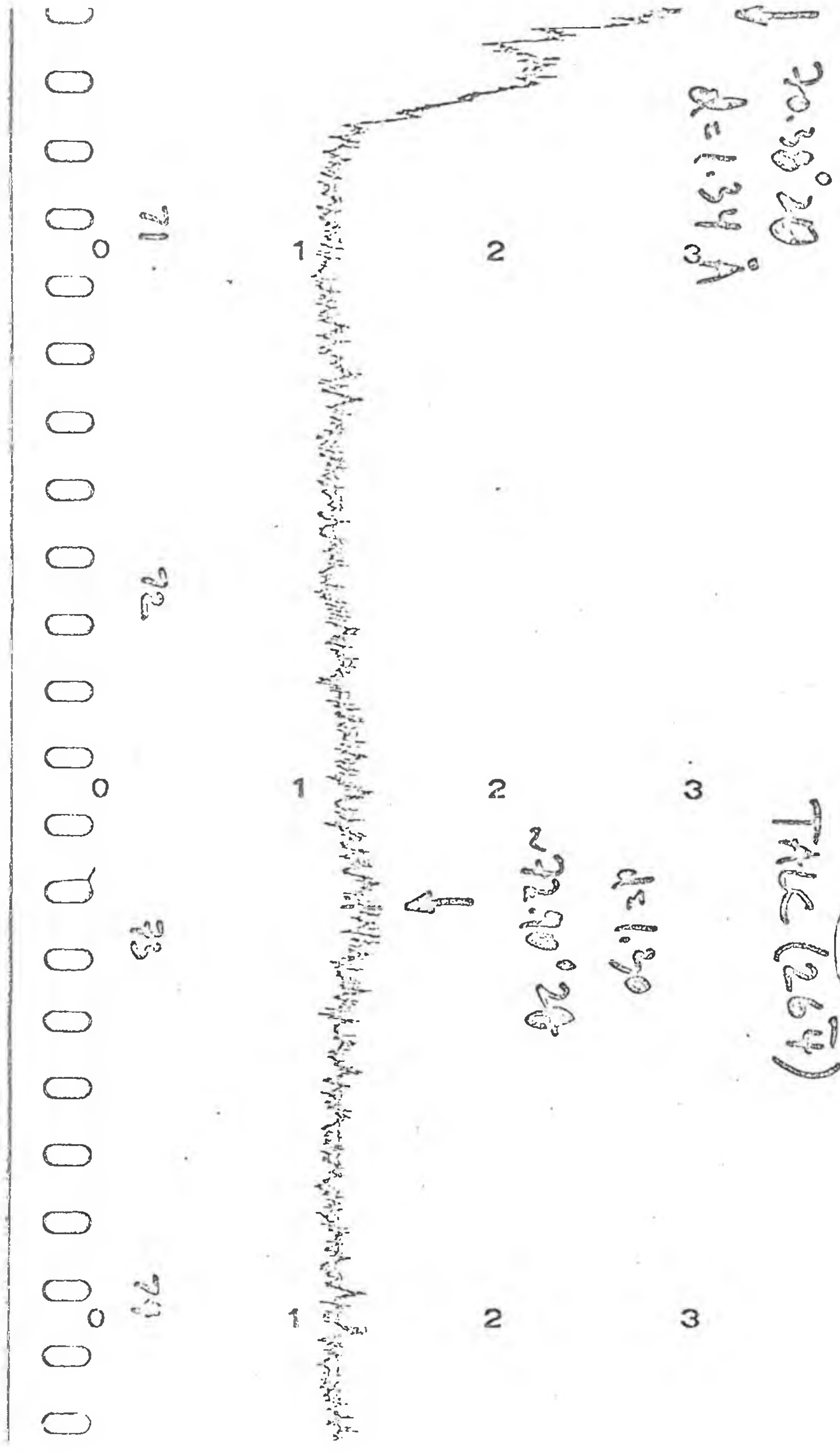
MLC (330)
d = 1.51
61.3° 2θ
d = 1.48 Å
~62.85° 2θ
62
63
64
65

5 6 7 8

5 6 7 8

5 6 7 8

Pennine
chlorite (139, 208)
d=1.42 Å
65.65°2θ
d=1.39 Å
67.18°2θ
(16)
TALC (2·0·10)
d=1.39 Å
67.53°2θ (Kα1)
(20)
TALC (1·3·12)
65
66
67
68


0
1
2
3

(M.O.O.)

4 5 6 7 8

4 5 6 7 8

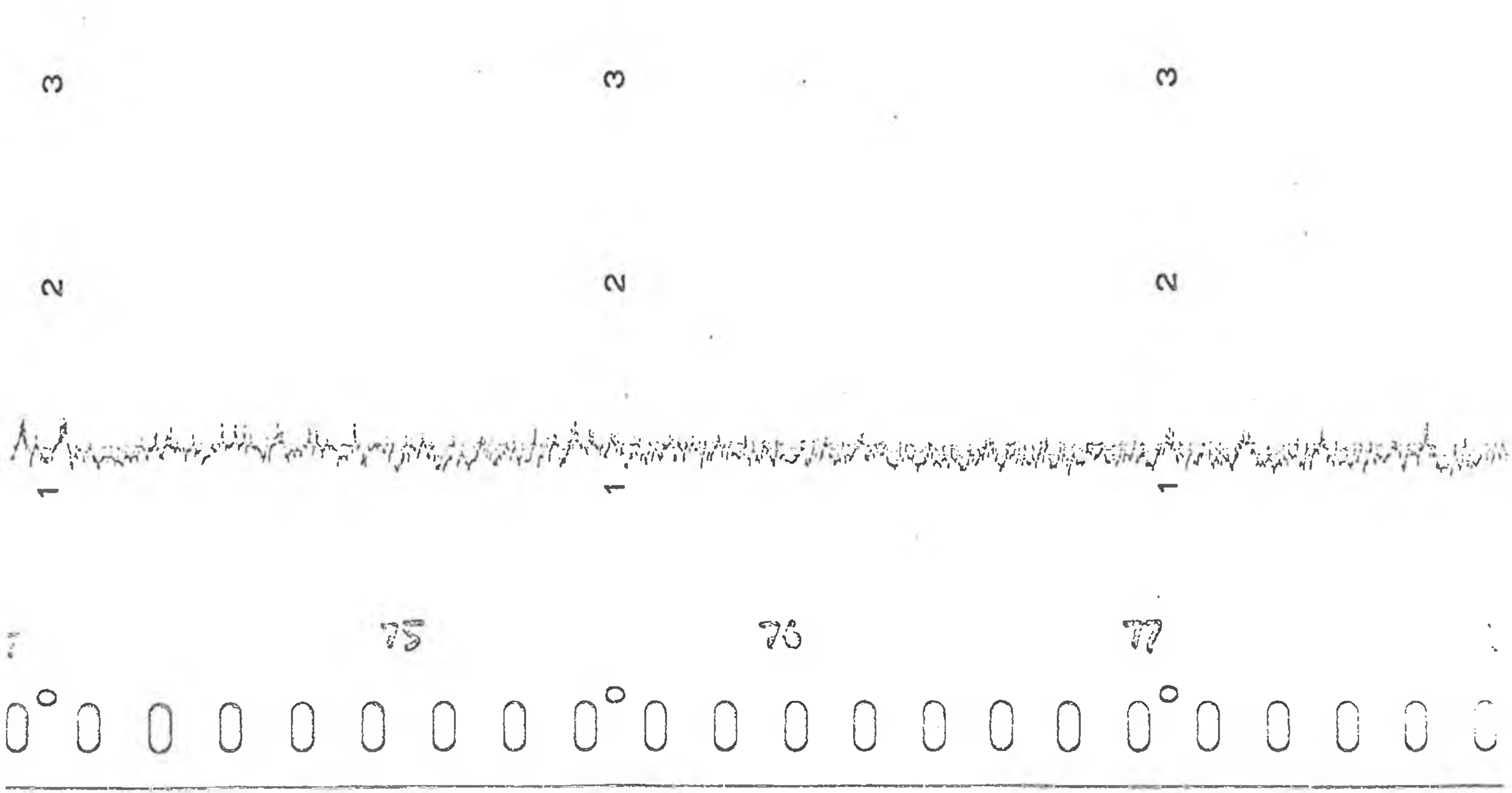
(901)

4 5 6 7 8

5 6 7 9

4 5 6 7 8

4 5 6 7 8



75
76
77

Johnson & Johnson

New Brunswick, N.J.
October 31, 1972

Subject: ANALYSIS OF JOHNSON'S* BABY POWDER FOR CHRYSOTILE ASBESTOS
PROJECT NO. 503

X-Ray Diffraction

Samples from the two lots of JOHNSON'S Baby Powder which had been examined by Dr. Lewin were subjected to qualitative analysis by means of continuous X-Ray diffractometer scans. The following mineral content was determined:

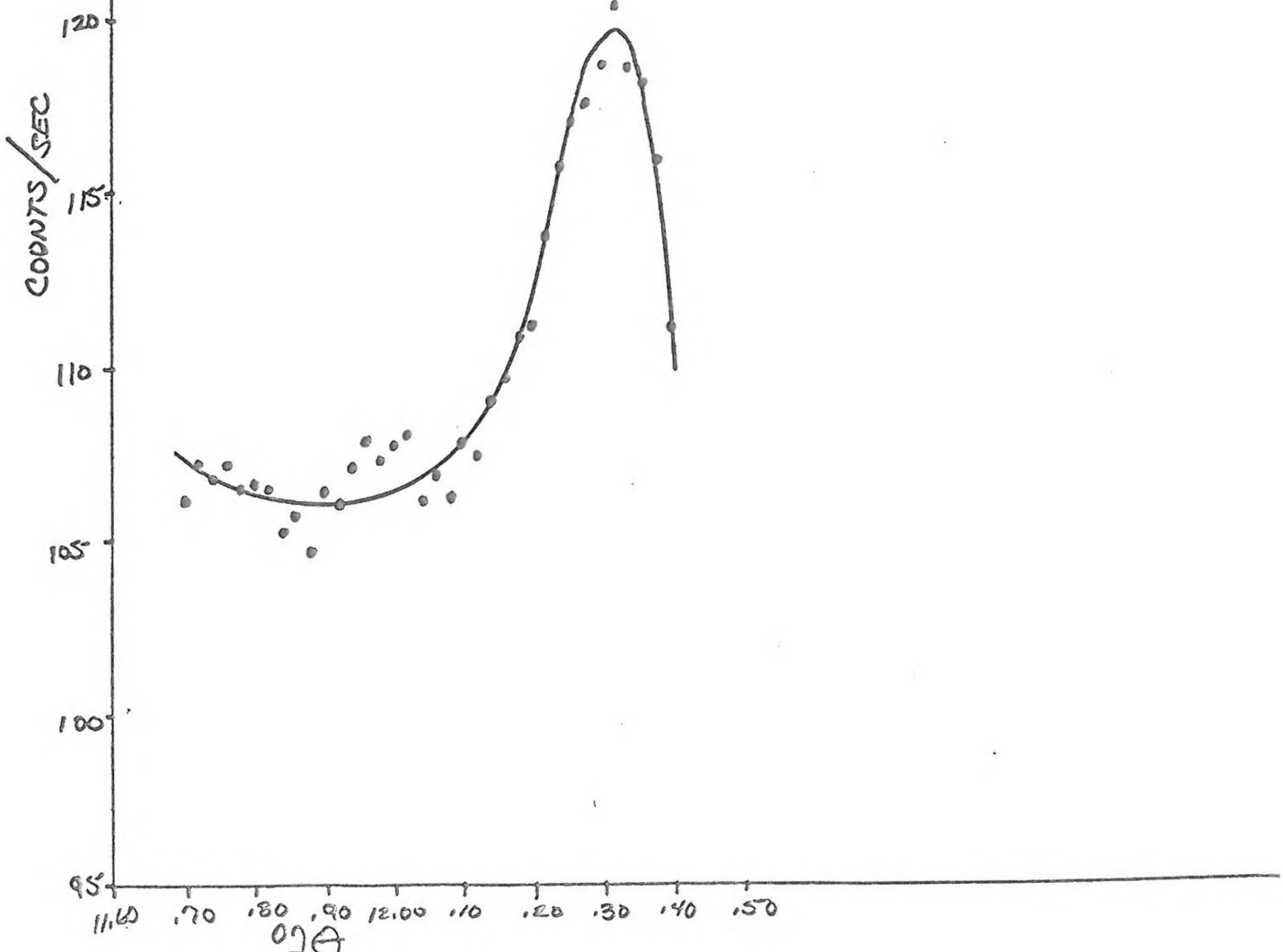
Lot 108T	Major:	Talc
	Trace:	Chlorite, Magnesite
Lot 109T	Major:	Talc
	Trace:	Chlorite, Magnesite, Dolomite

The step-scanning technique was then employed for increased sensitivity in the detection of chrysotile asbestos. It had been previously demonstrated that with this procedure it was possible to detect chrysotile in talc at a level of 2-3% by weight. The results, shown in Figures 1 and 2, indicate no diffraction peak in the region corresponding to an intense chrysotile reflection (7.31Å or 12.1° 2θ). Therefore, the samples of JOHNSON'S Baby Powder do not contain chrysotile at the minimum detectable level which is attained by X-Ray diffraction.

Differential Thermal Analysis

A semi-quantitative method based on the analysis of standard samples of chrysotile in talc had been developed using the technique of differential thermal analysis (DTA). The limit of detection of this method is 1% by weight of chrysotile. Thermograms of JOHNSON'S Baby Powder, Lots 108T and 109T, showed no thermal

*A Trademark of JOHNSON & JOHNSON.

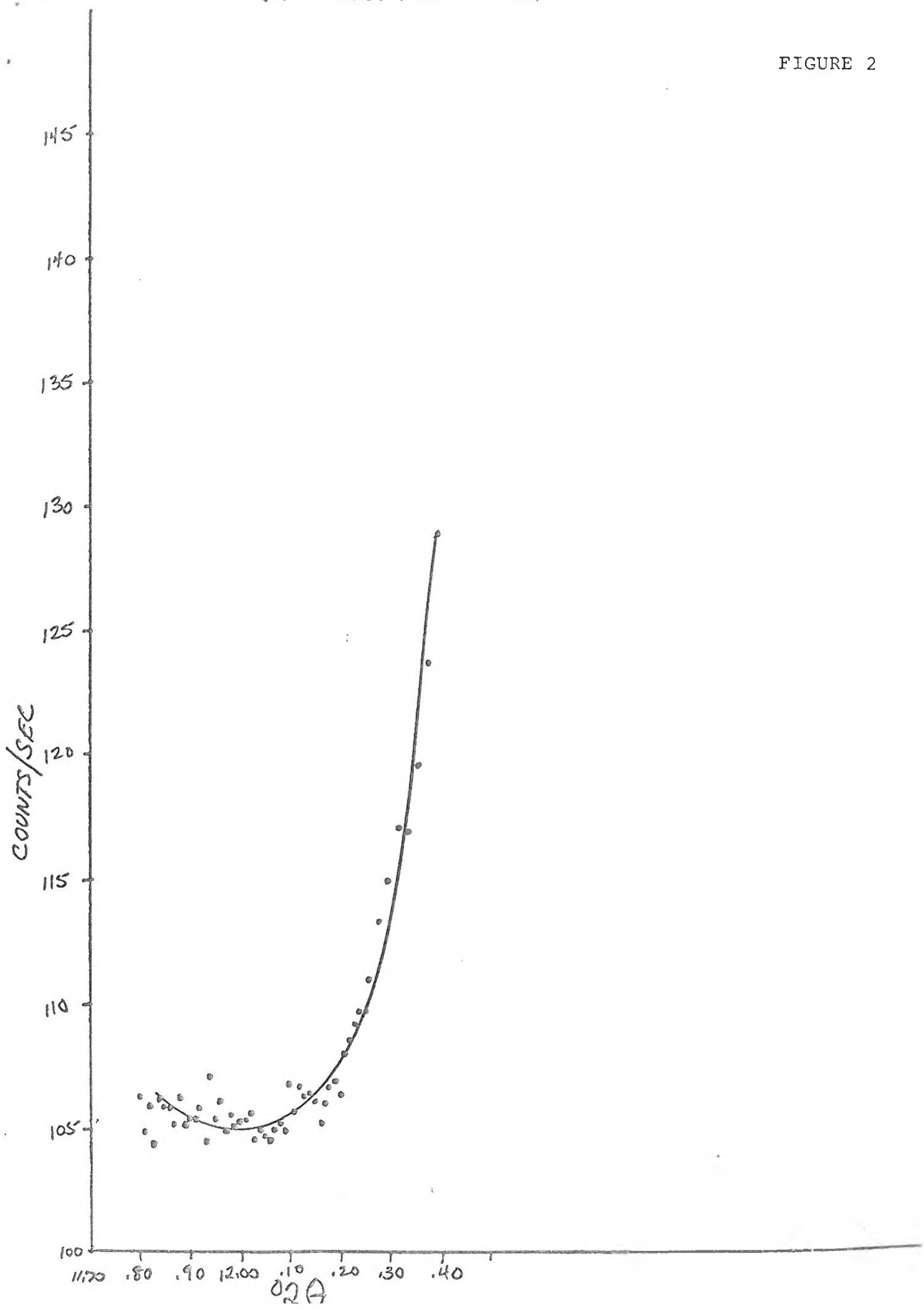

COUNTS/SEC
120
115
110
105
100
95
11.60
.70
.80
.90
12.00
.10
.20
.30
.40
.50
2θ

J & J BABY POWDER RETAINED LOT #108T

FIGURE I

J & J BABY POWDER LOT #109T

FIGURE 2



Johnson & Johnson

New Brunswick, N.J.
November 8, 1972

Subject: ANALYSIS OF JOHNSON'S* BABY POWDER
FOR TREMOLITE ASBESTOS
PROJECT NO. 503

The two lots of JOHNSON'S Baby Powder examined by Dr. Lewin had been previously analyzed qualitatively by X-Ray diffractometry (report of October 31, 1972). It was desired to further analyze these samples for the presence of trace quantities of amphibole asbestos minerals; such as, tremolite. An X-Ray step-scanning procedure had been developed for the detection of tremolite in Vermont talc. The limit of detection of this method is 0.1% by weight of tremolite. Figure 1 shows the peak obtained for an intense tremolite reflection (8.38Å or 10.5° 2θ) for a standard sample of 0.5% tremolite in Vermont talc. Plots of the step-scans through the same region for JOHNSON'S Baby Powder, Lots 108T and 109T, are shown in Figures 2 and 3, respectively. The results do not indicate the presence of amphibole minerals. Therefore, the samples of JOHNSON'S Baby Powder do not contain tremolite within the limit of detection of the X-Ray diffraction step-scanning technique.

Since tremolite does not exhibit intense thermal transitions distinguishable from talc, differential thermal analysis is not applicable for the detection of this mineral in talc at low levels.

John P. Scheiz

ab

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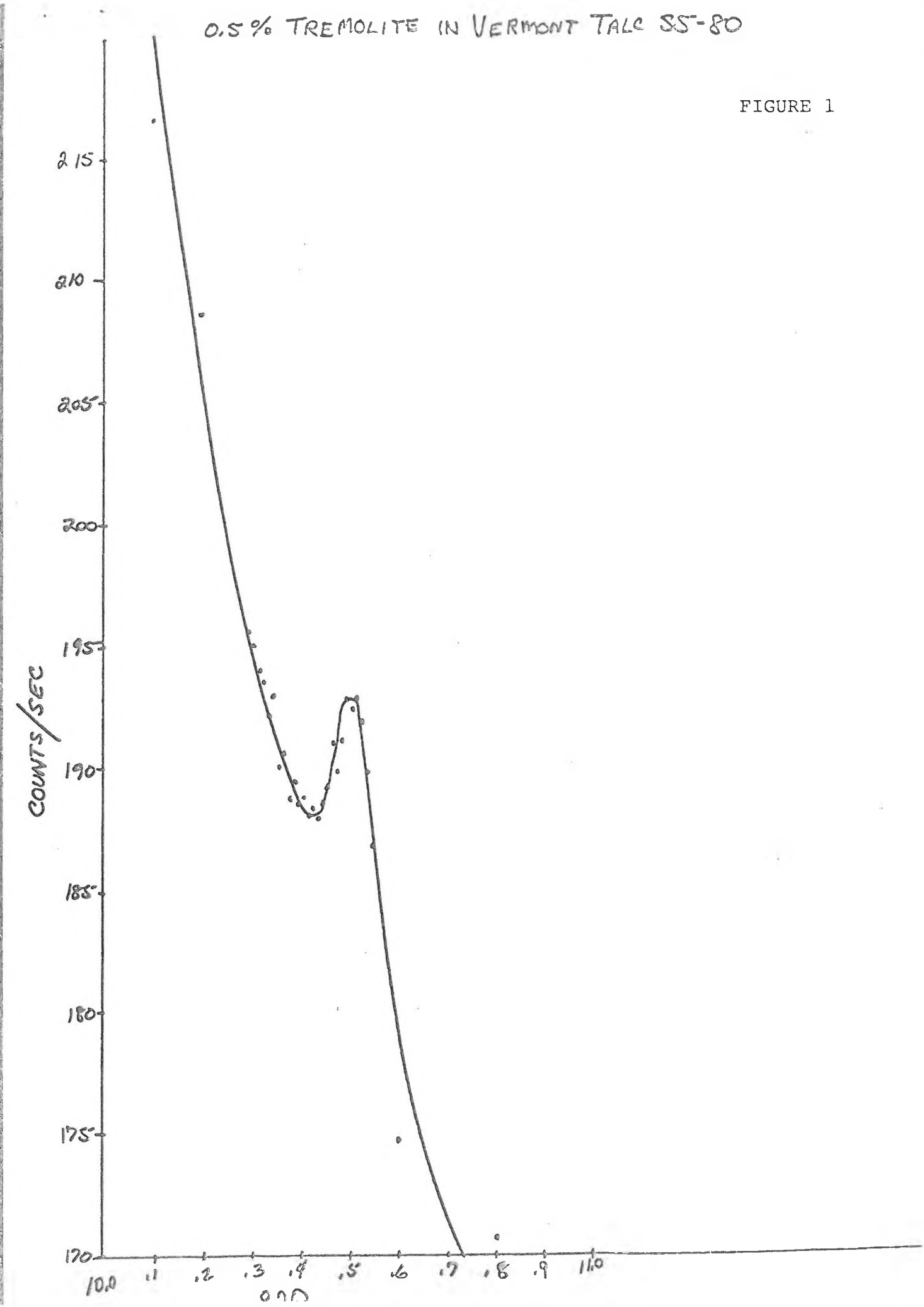
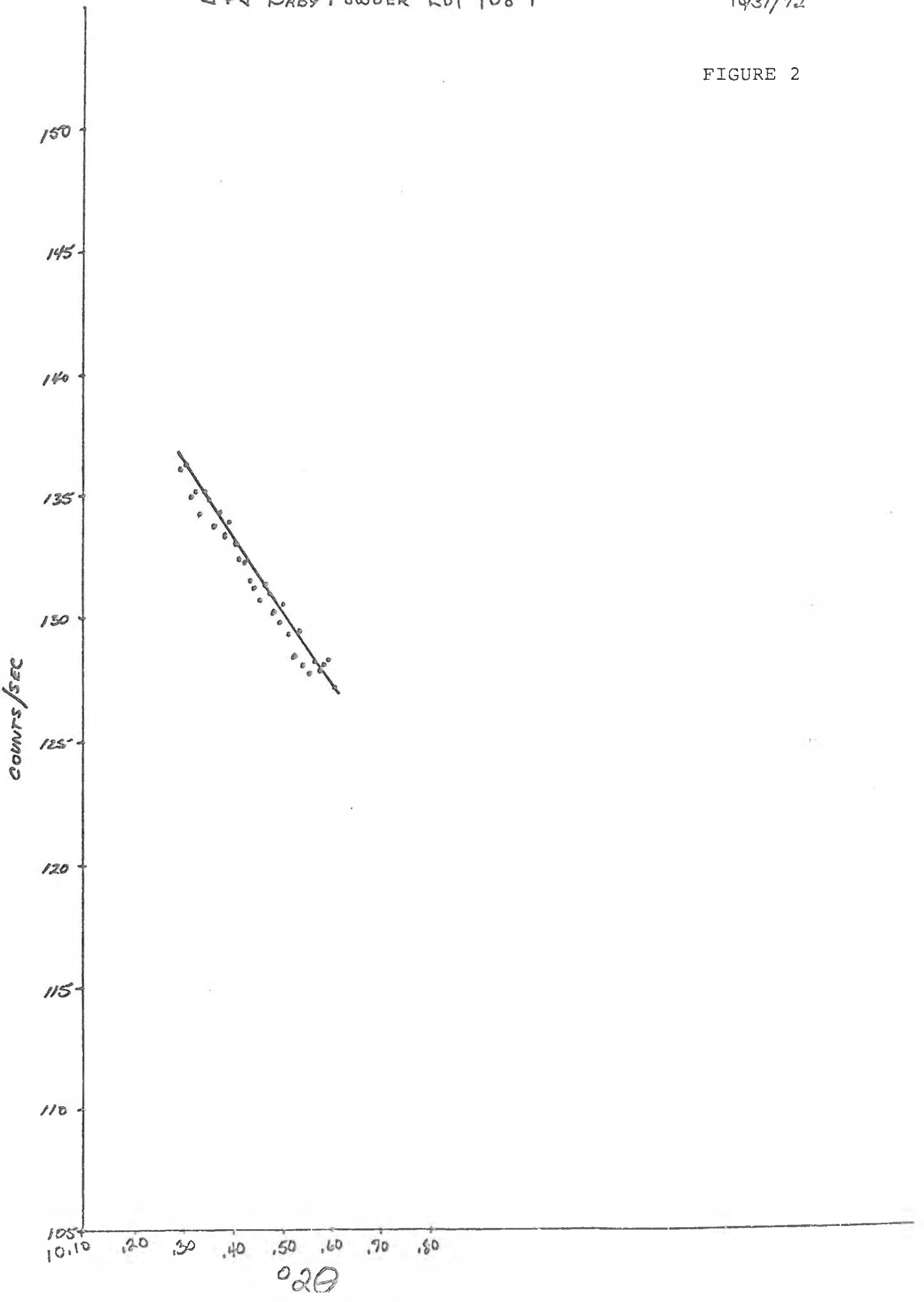

0.5% TREMOLITE IN VERMONT TALC SS-80
COUNTS/SEC
215
210
205
200
195
190
185
180
175
170
10.0
.1
.2
.3
.4
.5
.6
.7
.8
.9
1.0

FIGURE 1

J+J Baby Powder Lot 108 T

10/31/72

FIGURE 2



J&J BABY POWDER LOT 109 T 11/1/72

FIGURE 3