

Prof. Brown

J&J-0061482

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

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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 monochromator and sample spinner was used in this work. Other pertinent experimental conditions were as follows:  $\text{CuK}\alpha$  radiation (40KV, 20mA); scan speed =  $1/4^\circ 2\theta/\text{min.}$ ; scale factor = 4; time constant = 4;  $1^\circ$  slits; scan range =  $5-80^\circ 2\theta$ ; 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 (006,115) and cannot be a tremolite peak. Peaks due to  $\text{CuK}\beta$  were also searched for but were not found.

It is concluded that retain 108T (4-17-72) from Johnson & Johnson's Vermont 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.

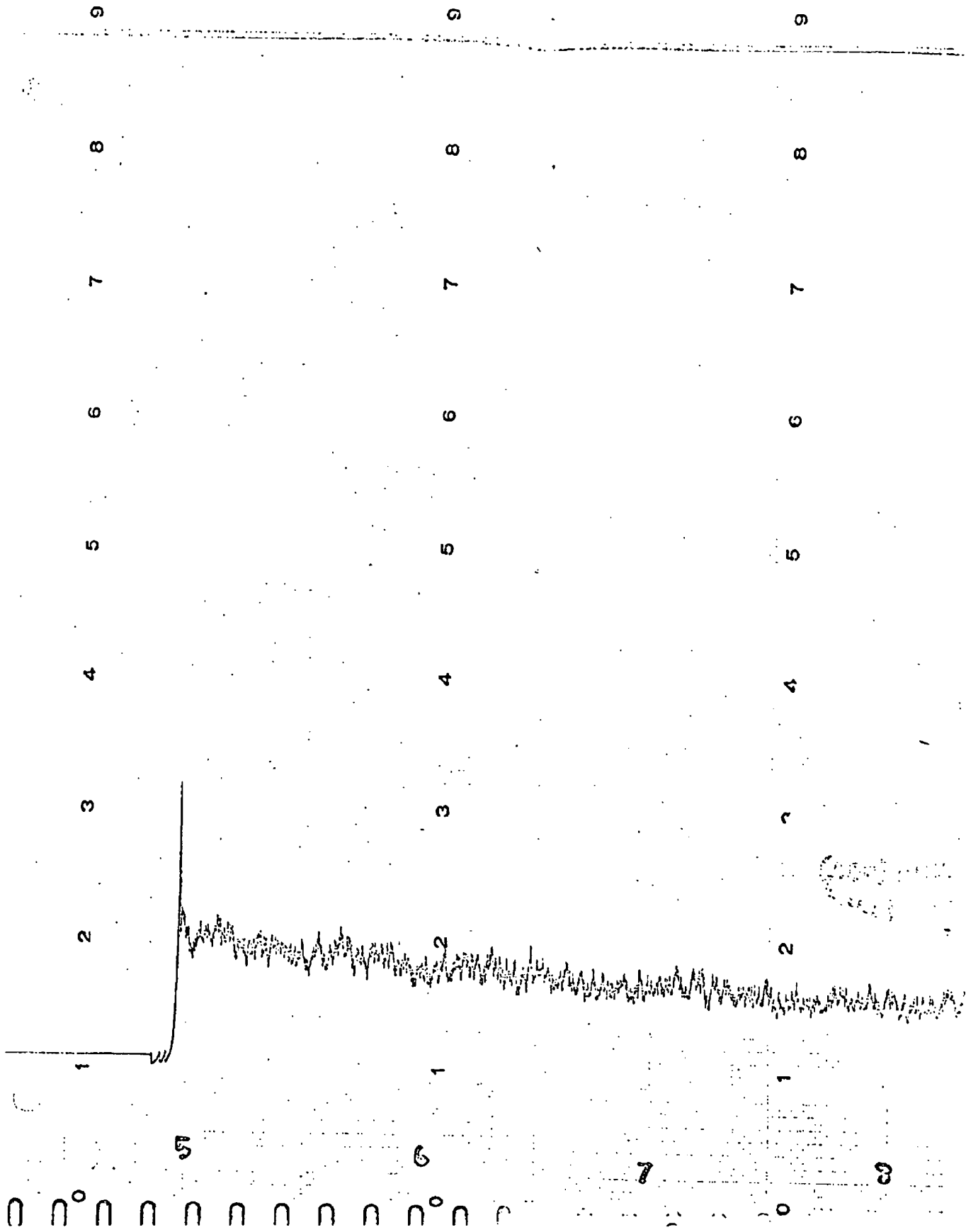
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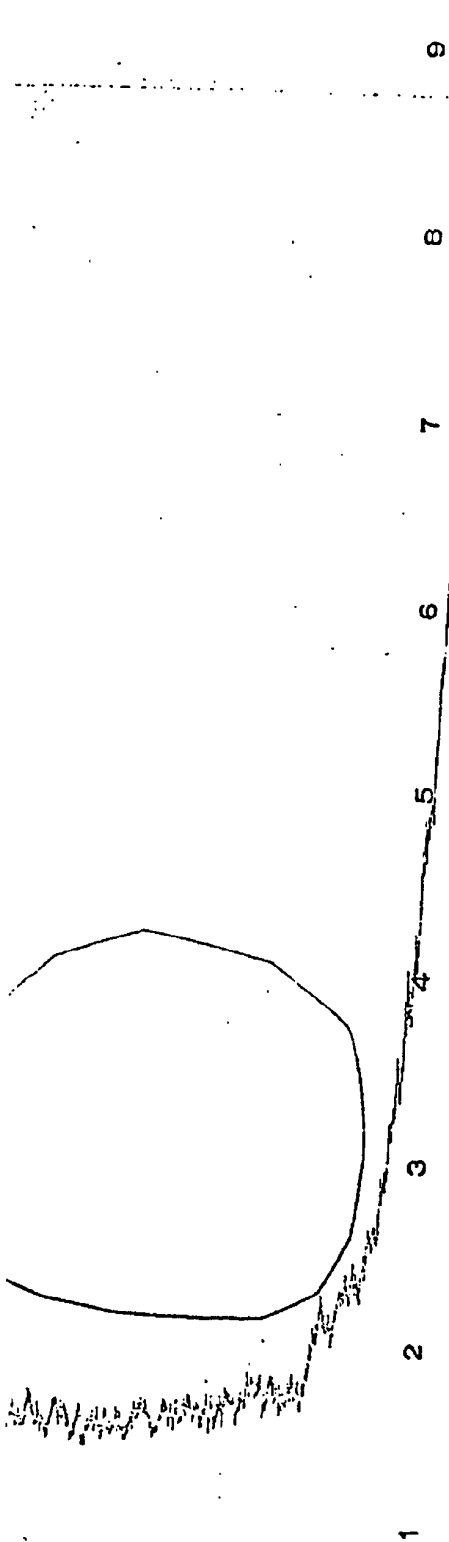
Table 1: X-ray Data for Johnson and Johnson's Baby Powder  
Retain 108T (4-17-72)

<u>Mineral</u>	<u>hkl</u>	<u>d(Å)</u>	<u>2θ(deg.)</u>
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 $\alpha_1$ )
Talc	0.0.14	1.34	70.38
Talc	26 $\bar{4}$	1.30	72.90

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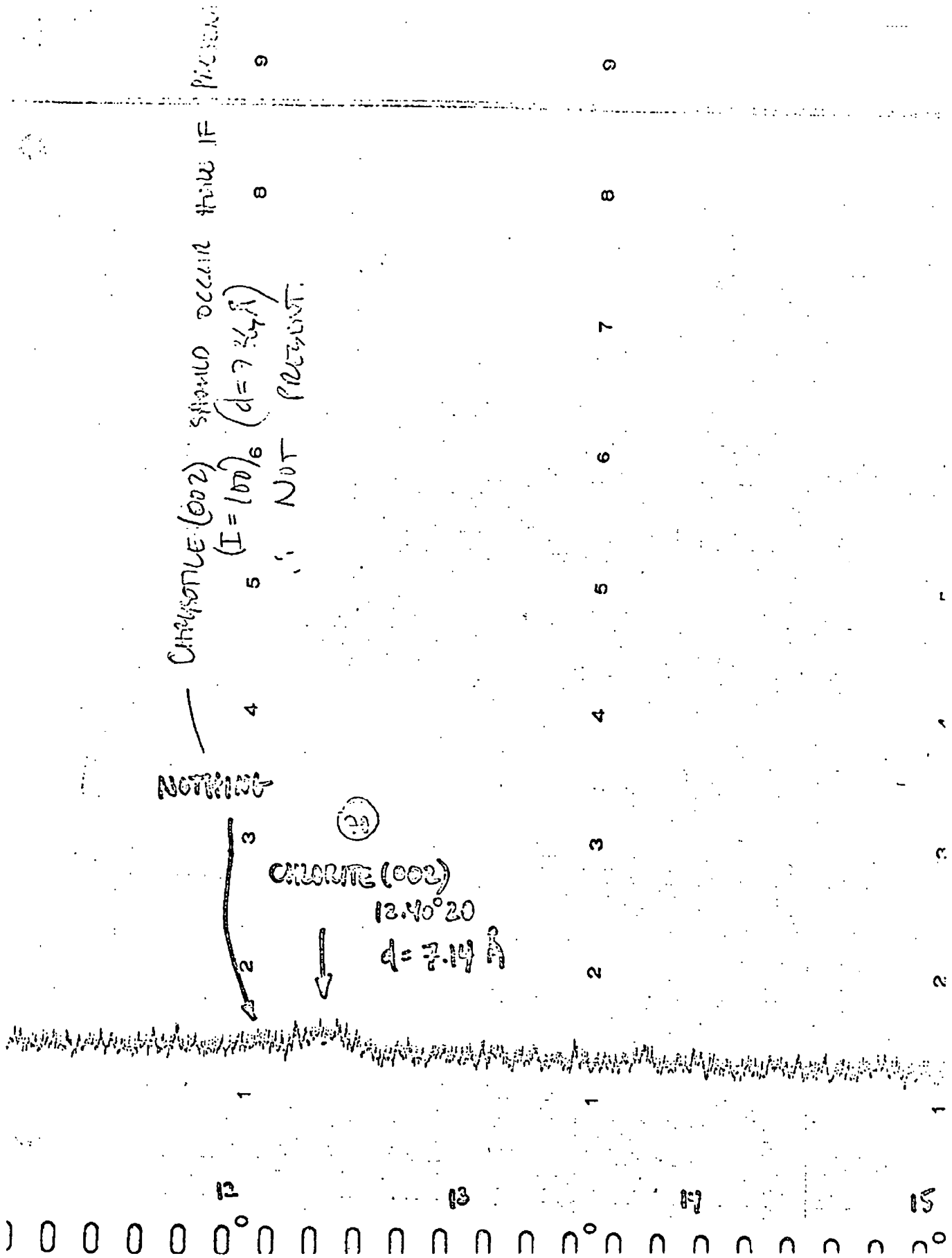
← TAIL (002) 9

$9.36^\circ 2\theta$   
 $d = 9.45 \text{ \AA}$

IF TRIMOLTS WERE PRESENT  
 THERE <sup>4</sup> SHOULD BE A PEAK (I=100) 7  
 AT  $\sim 10.55^\circ 2\theta$  ( $d = 8.38 \text{ \AA}$ )

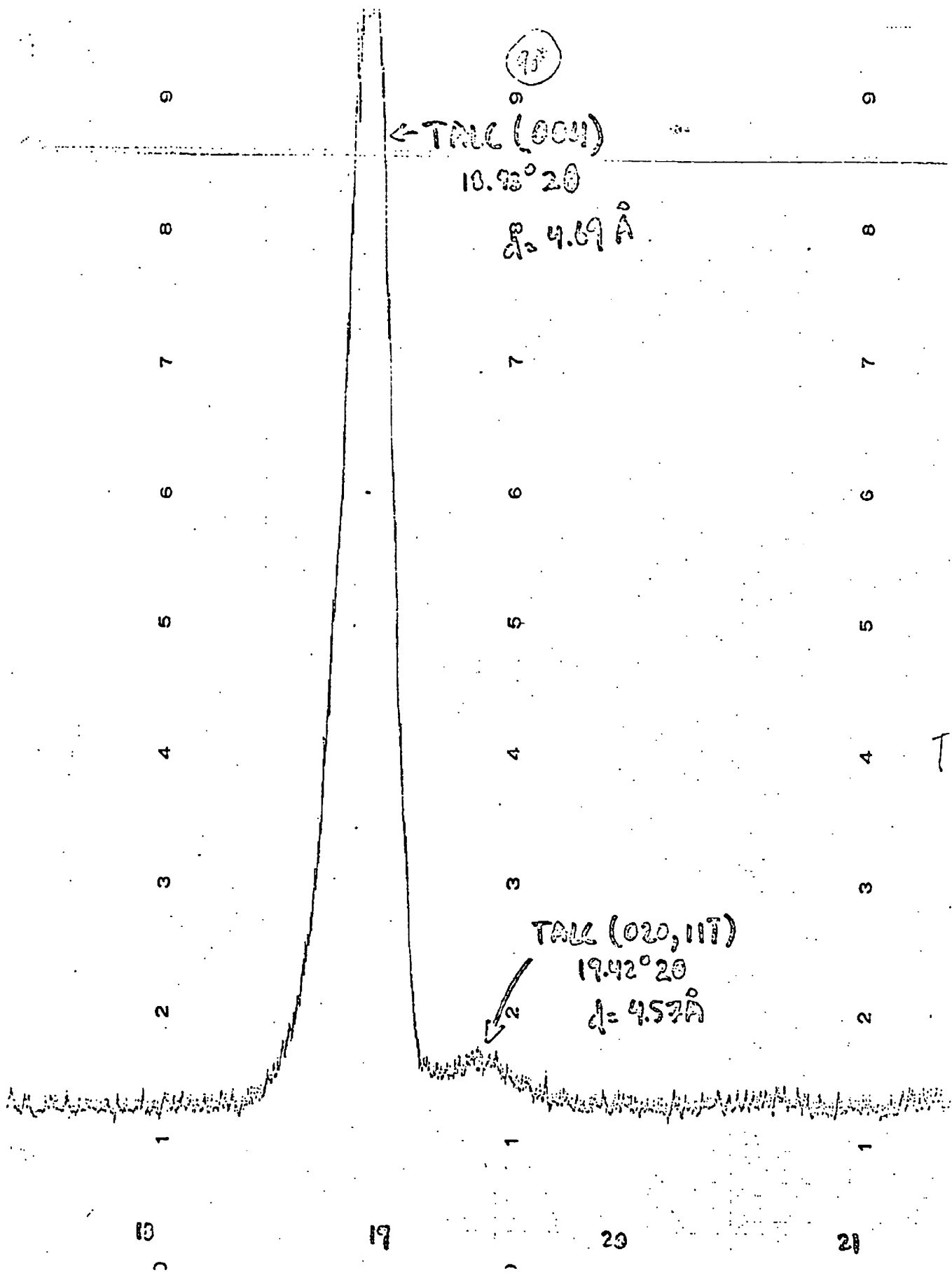
NOT

— CRYSTAL (002) SHOULD BE THERE IF PRESENT



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J&J-0061489



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TALL (?) (022) (6)



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3

$d = 4.13 \text{ \AA}$

$215^\circ 20'$

2

2

2



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1

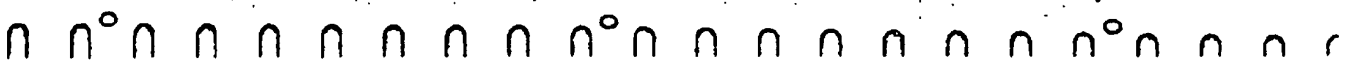
1

21

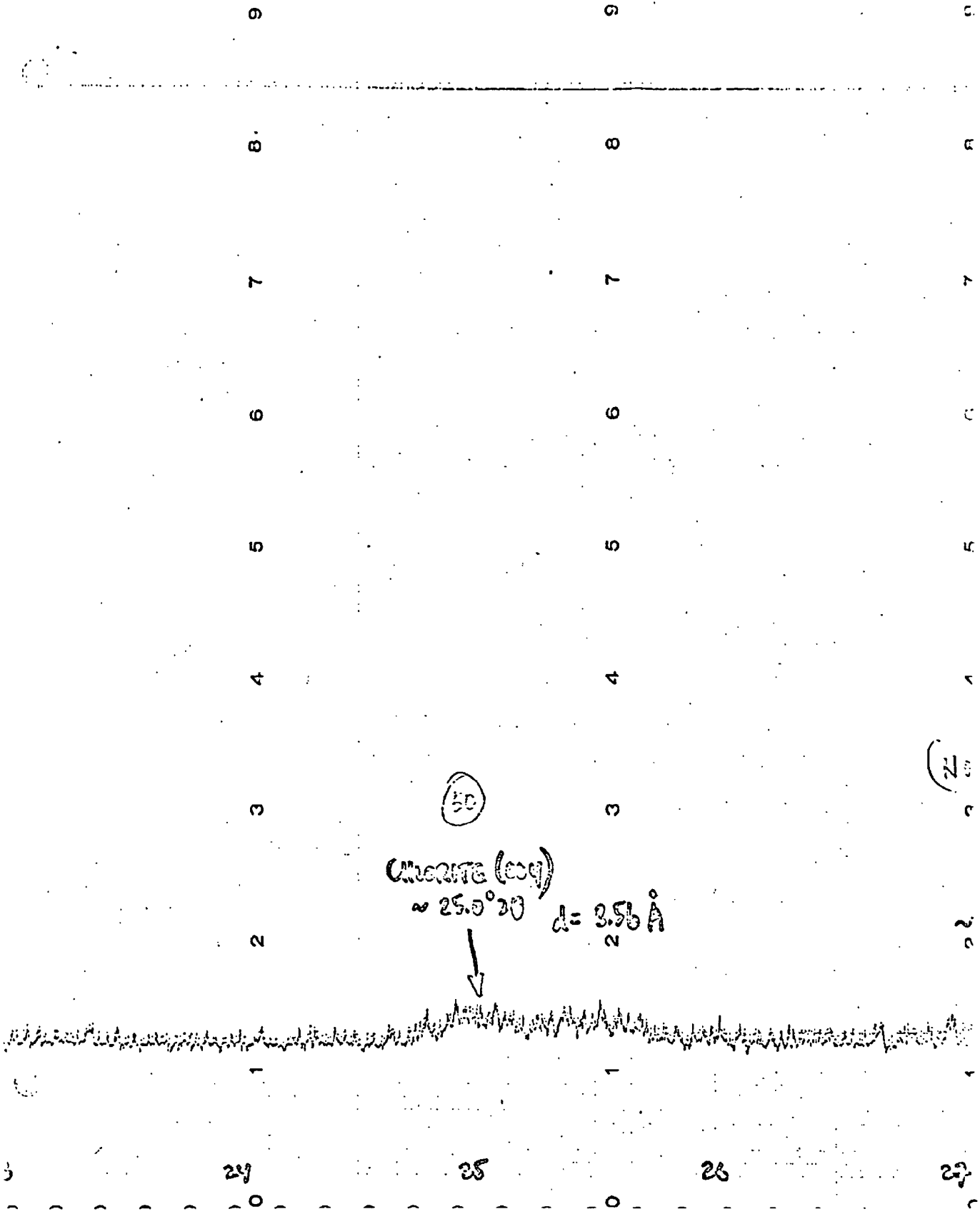
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23

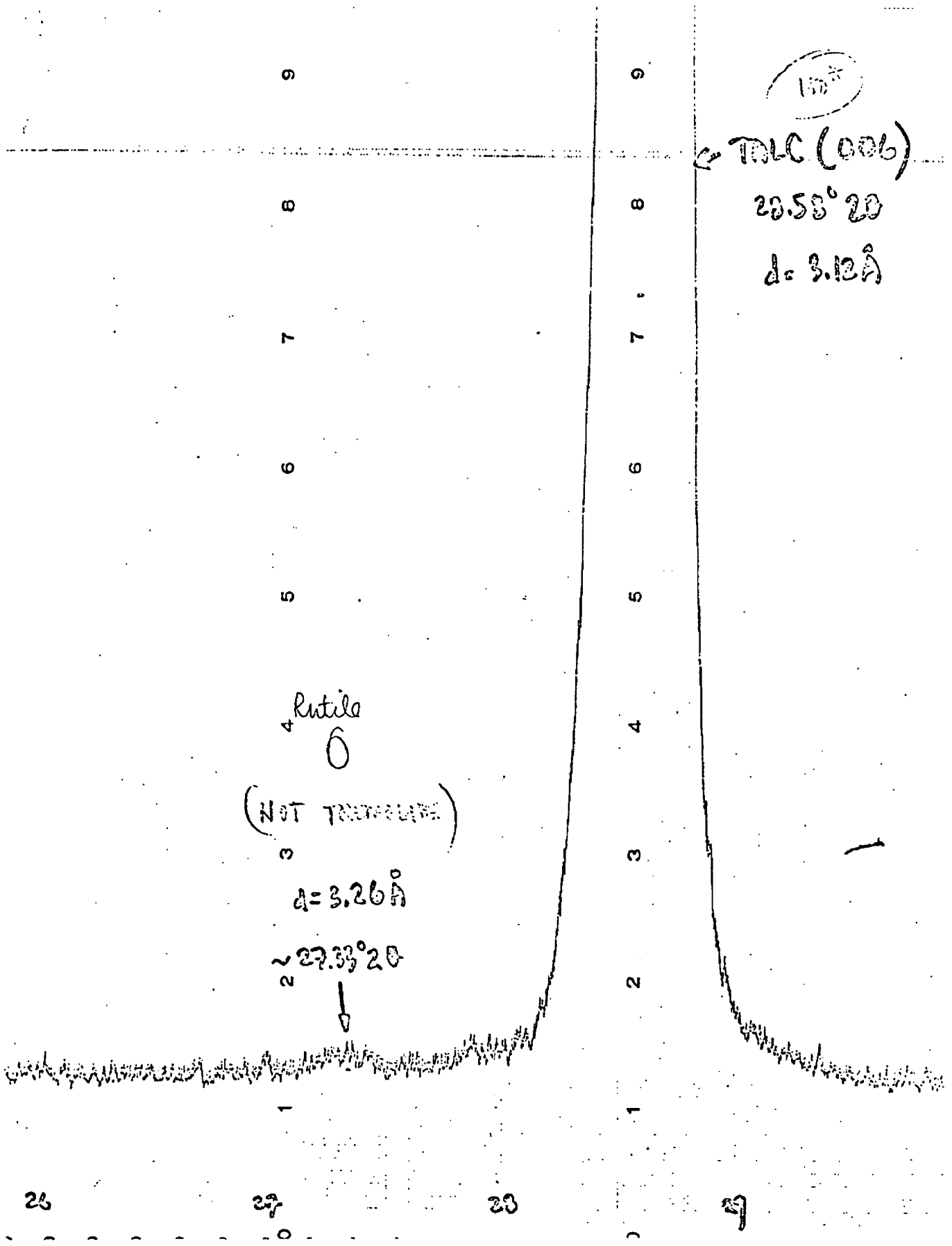
24



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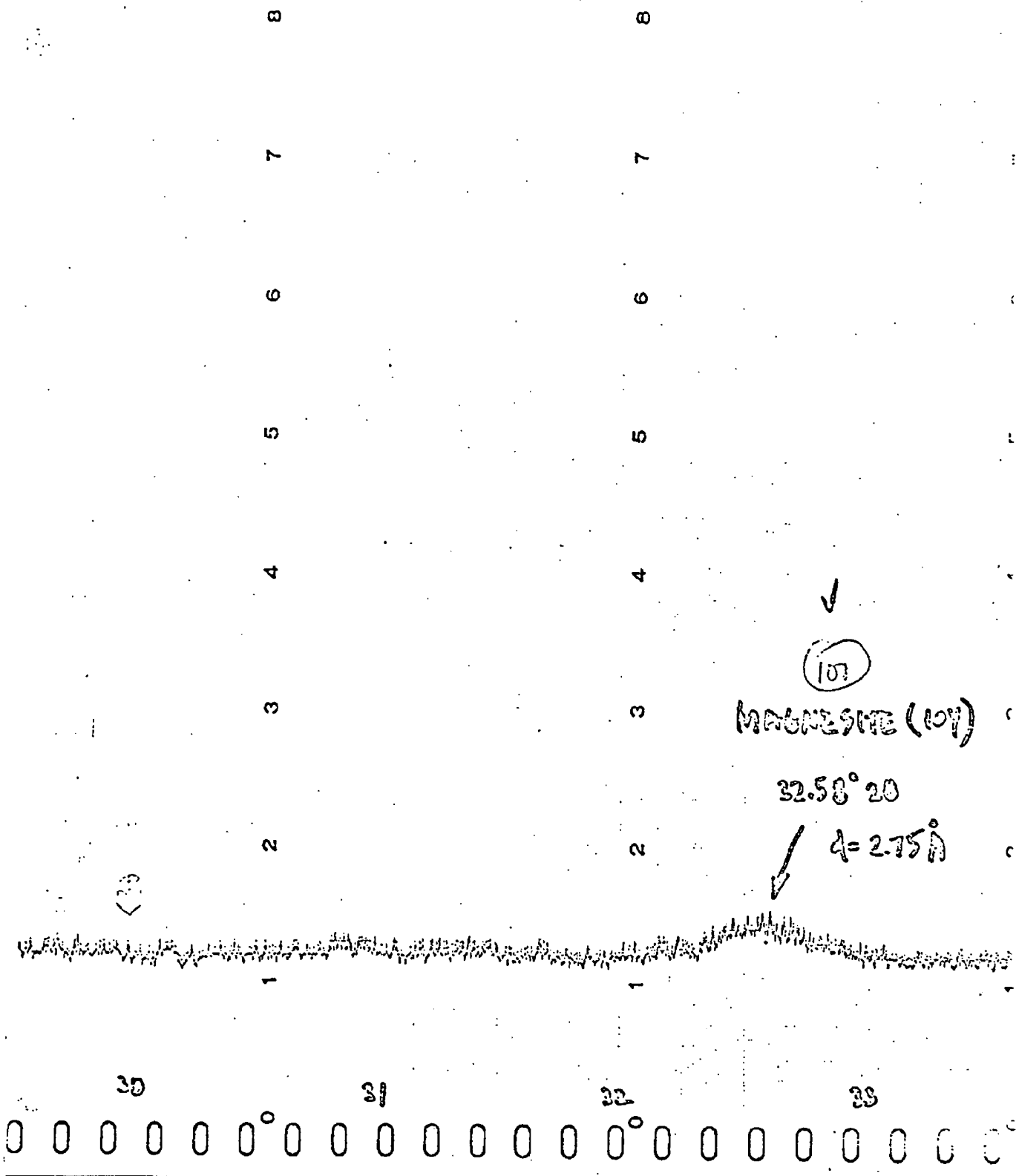


J&J-0061491

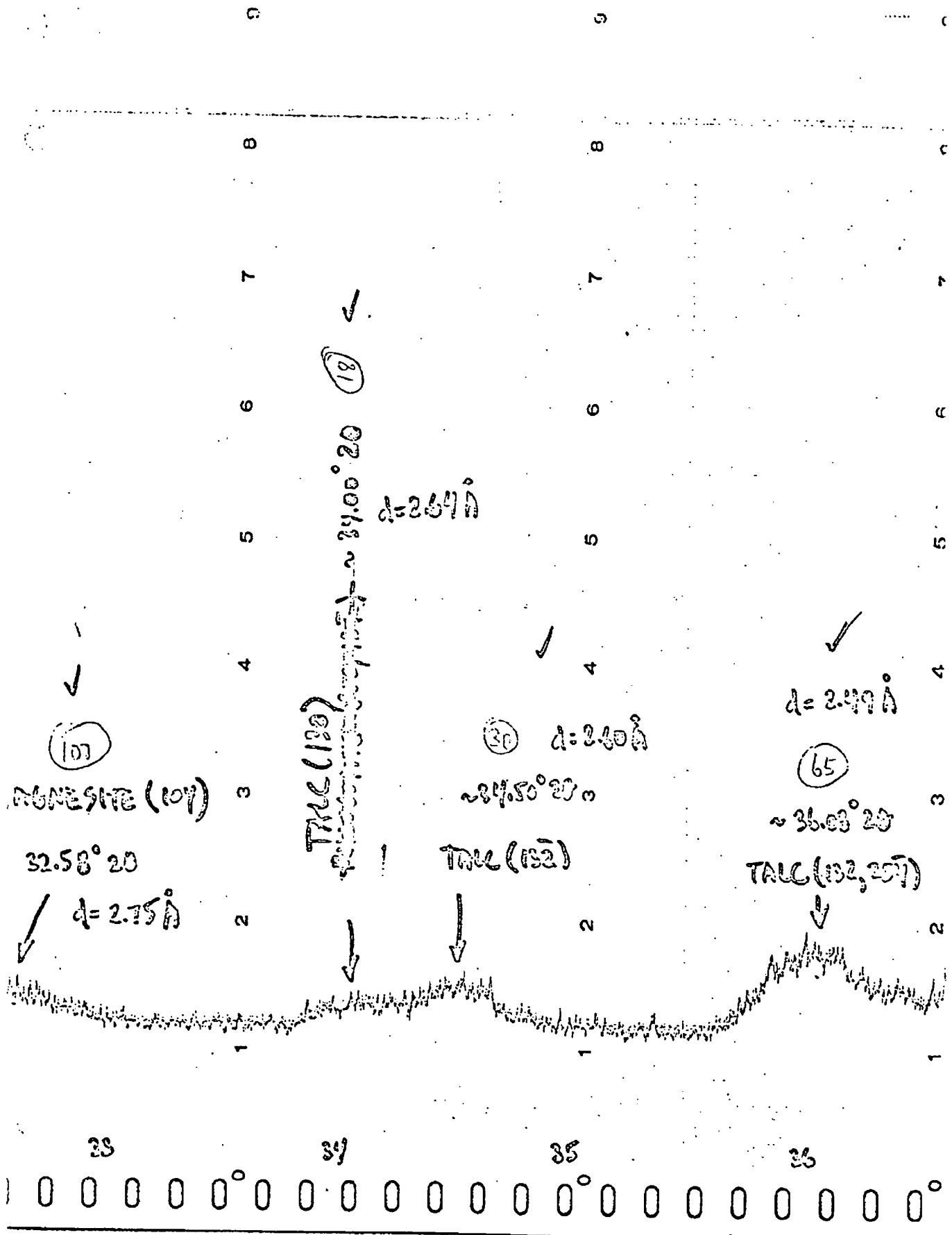


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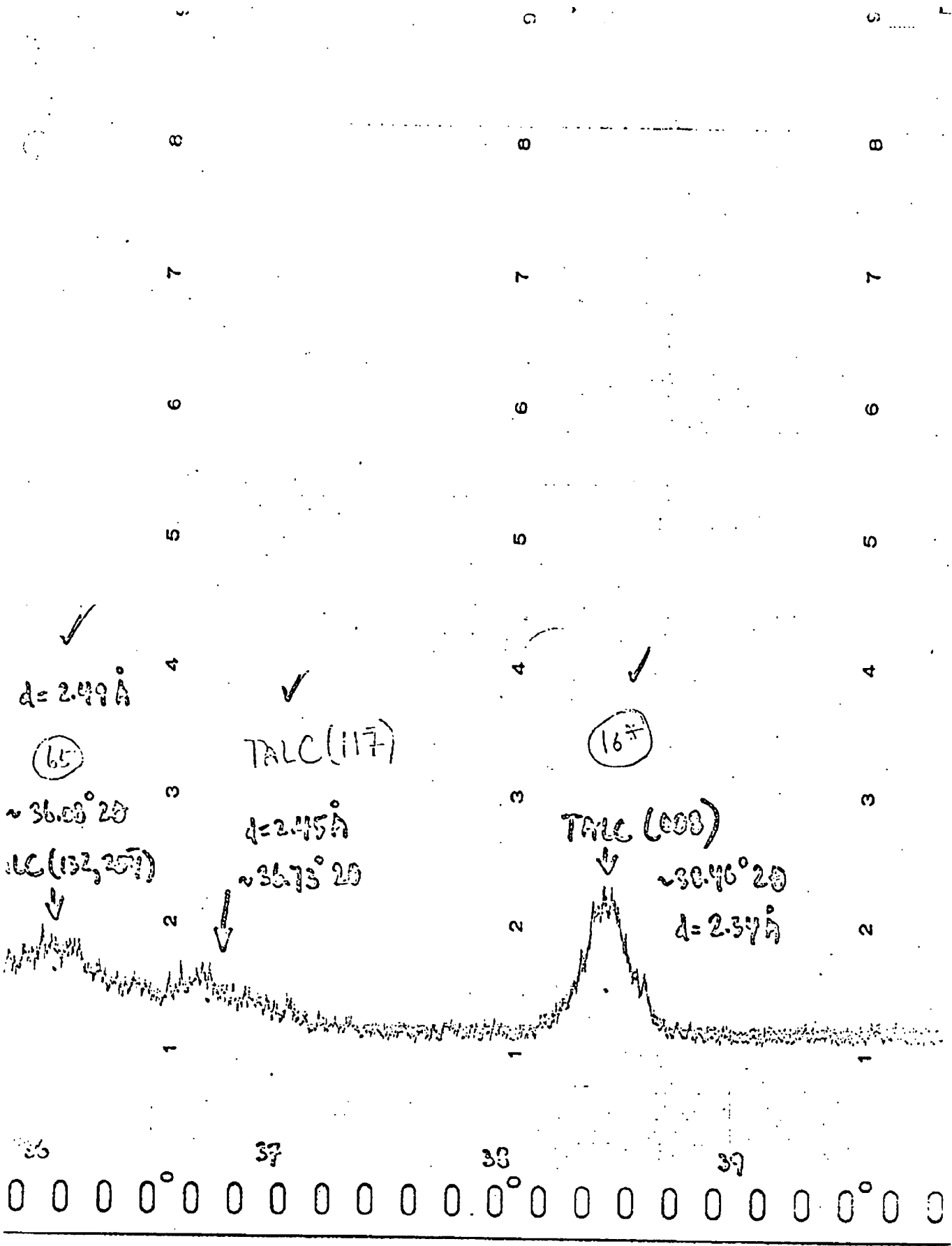




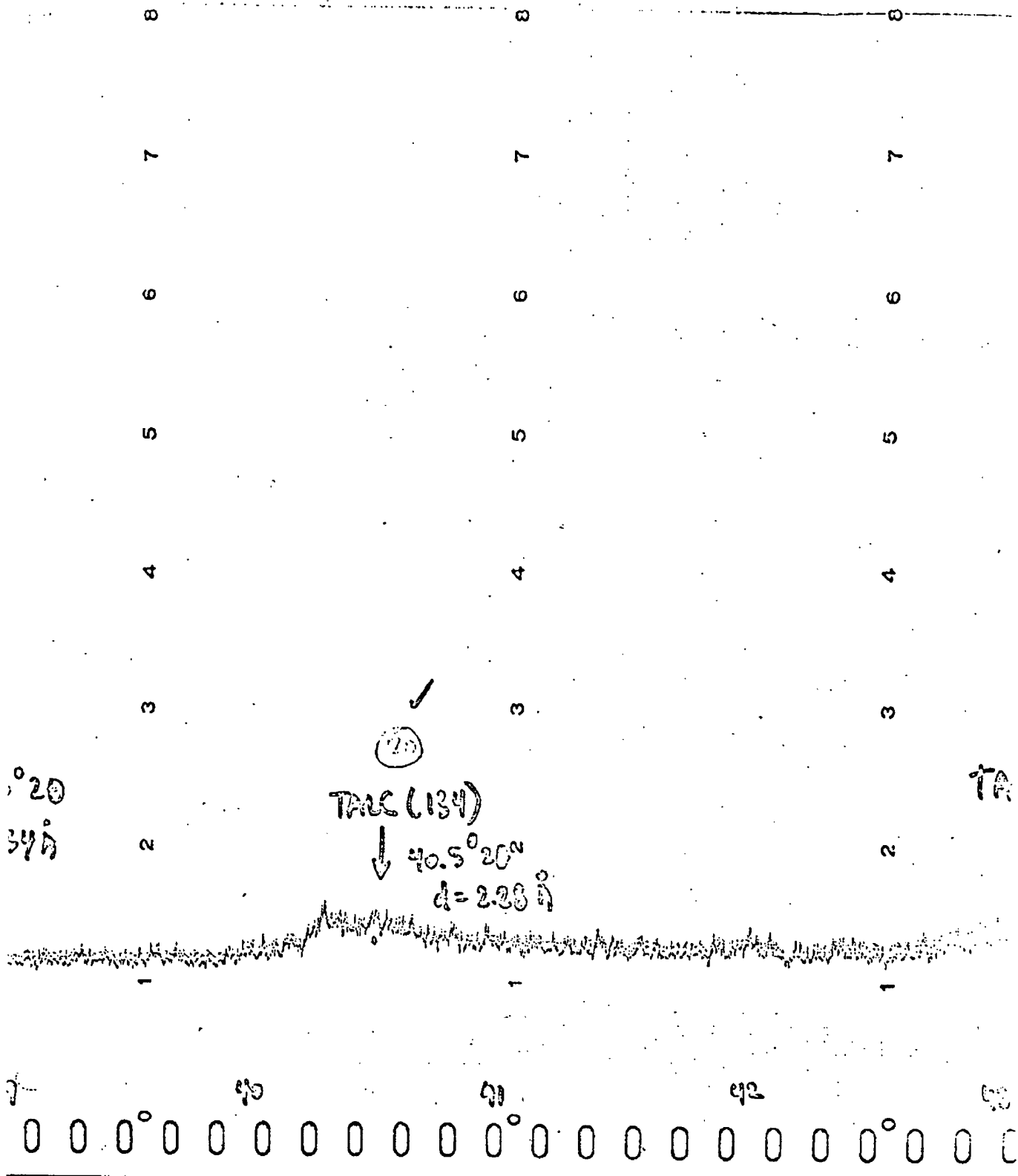
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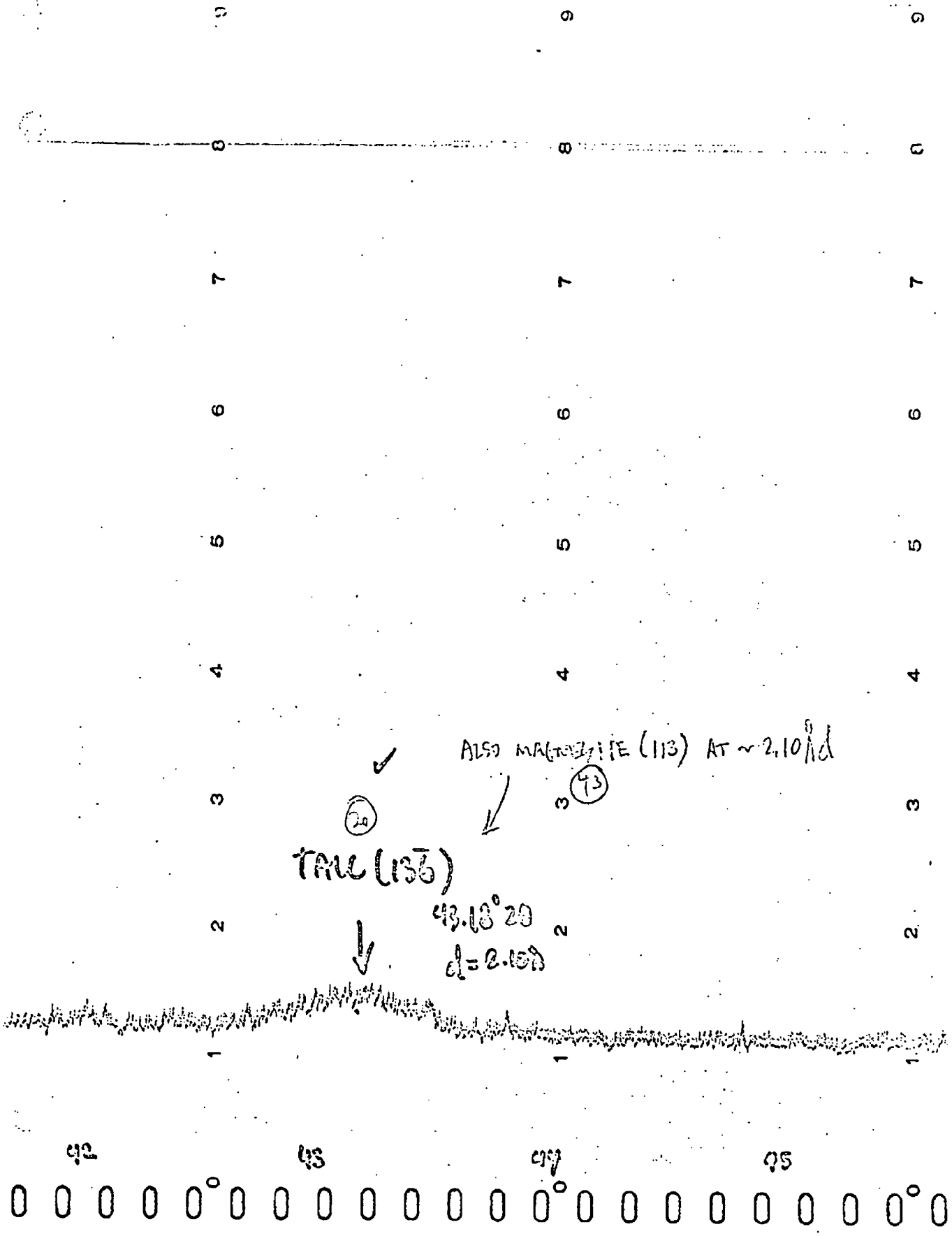


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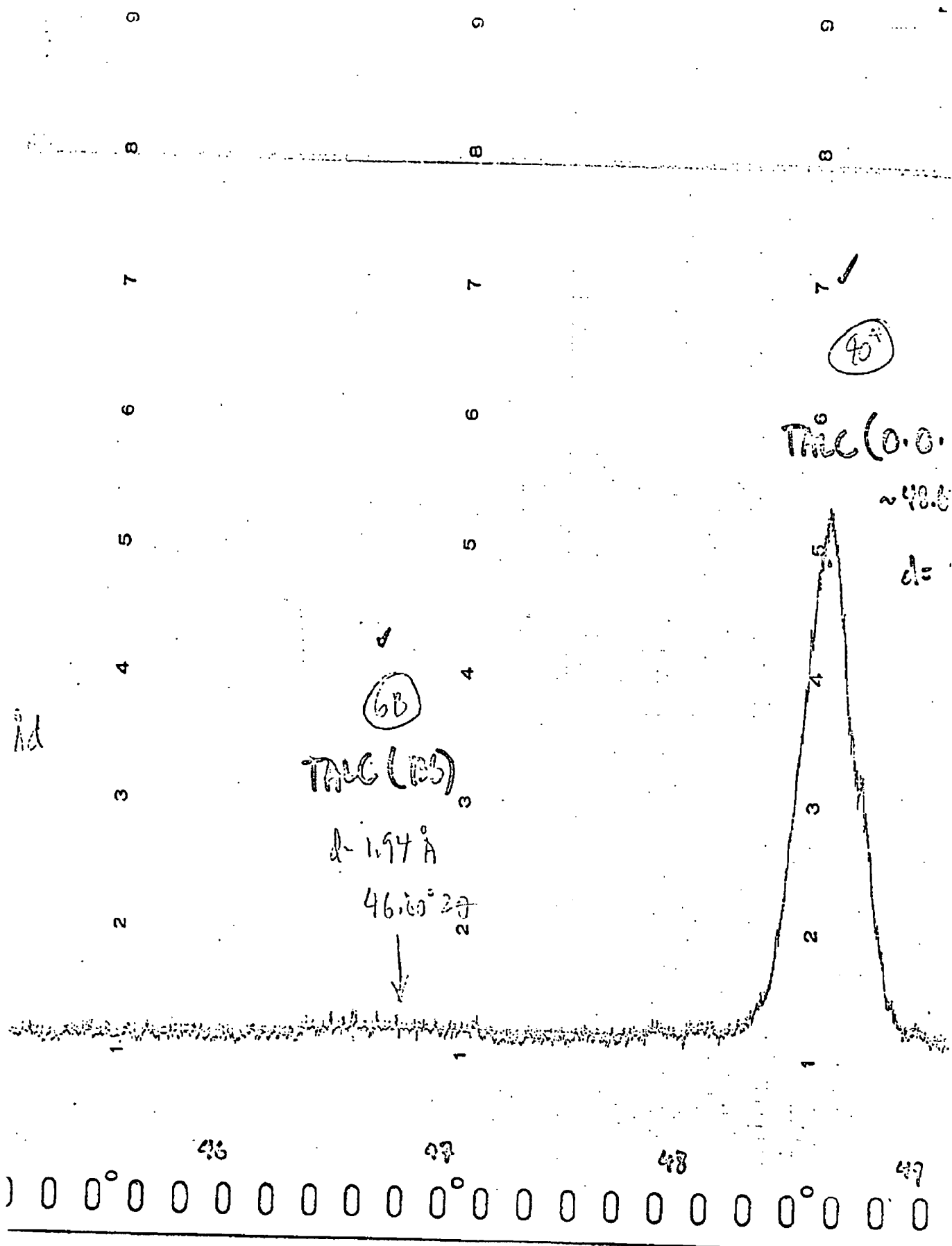


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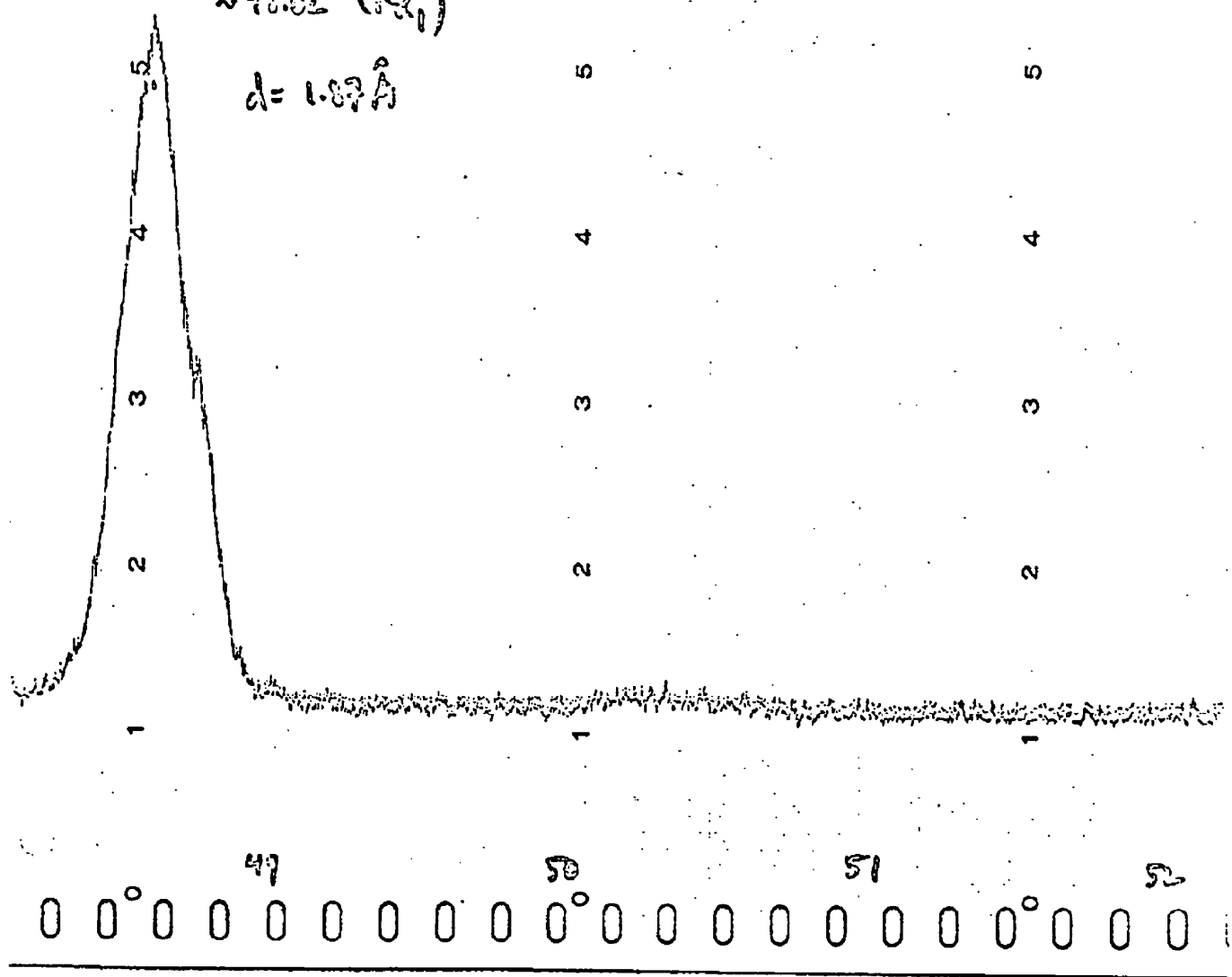
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7 ✓  
70°

TiCl<sub>3</sub> (0.0.10)  
~48.62 (K<sub>α1</sub>)  
d = 1.87 Å



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USMC (116) AT ~1.70 Å

d = 1.69 Å

130°  
134° 20'

TALC

d = 1.65 Å

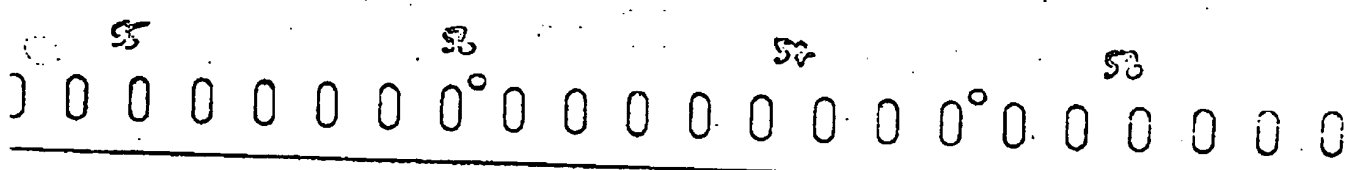
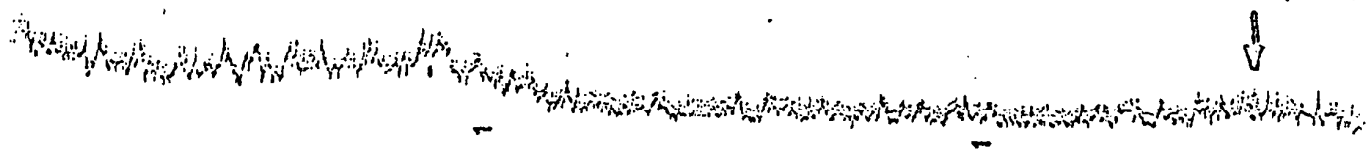
~55.90° 20'

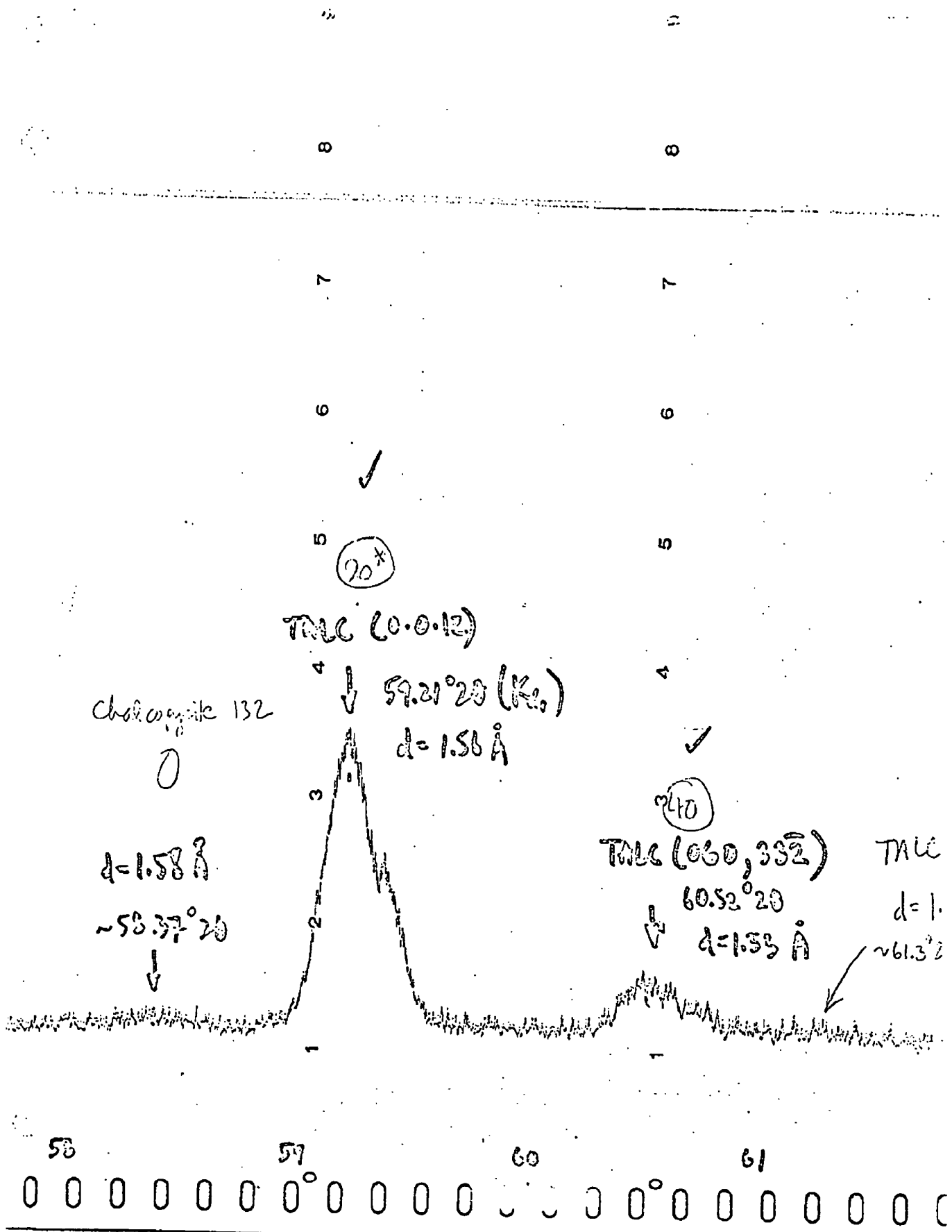
Chalcopyrite

0

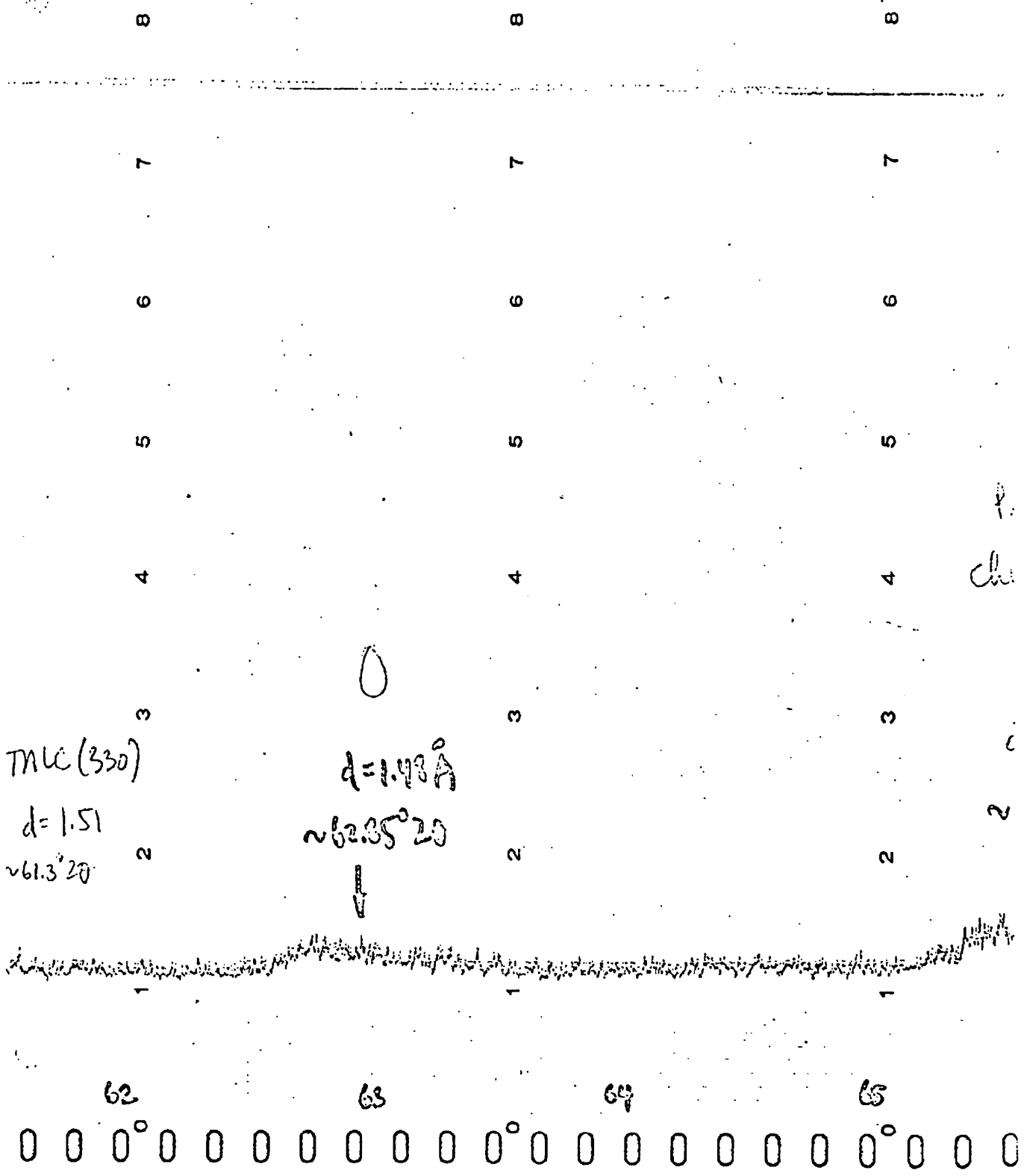
d = 1.58 Å

~58.37° 20'

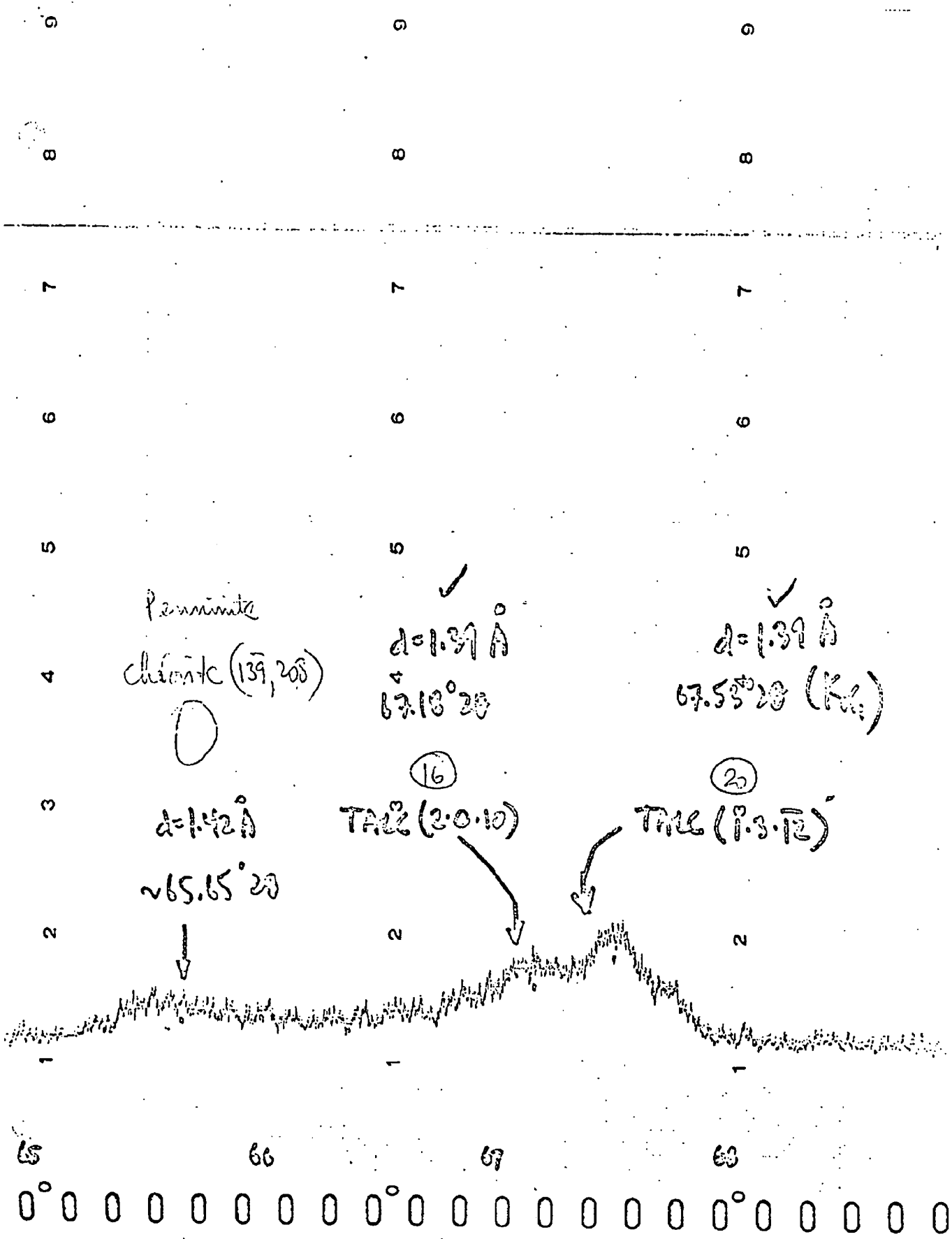




J&J-0061503

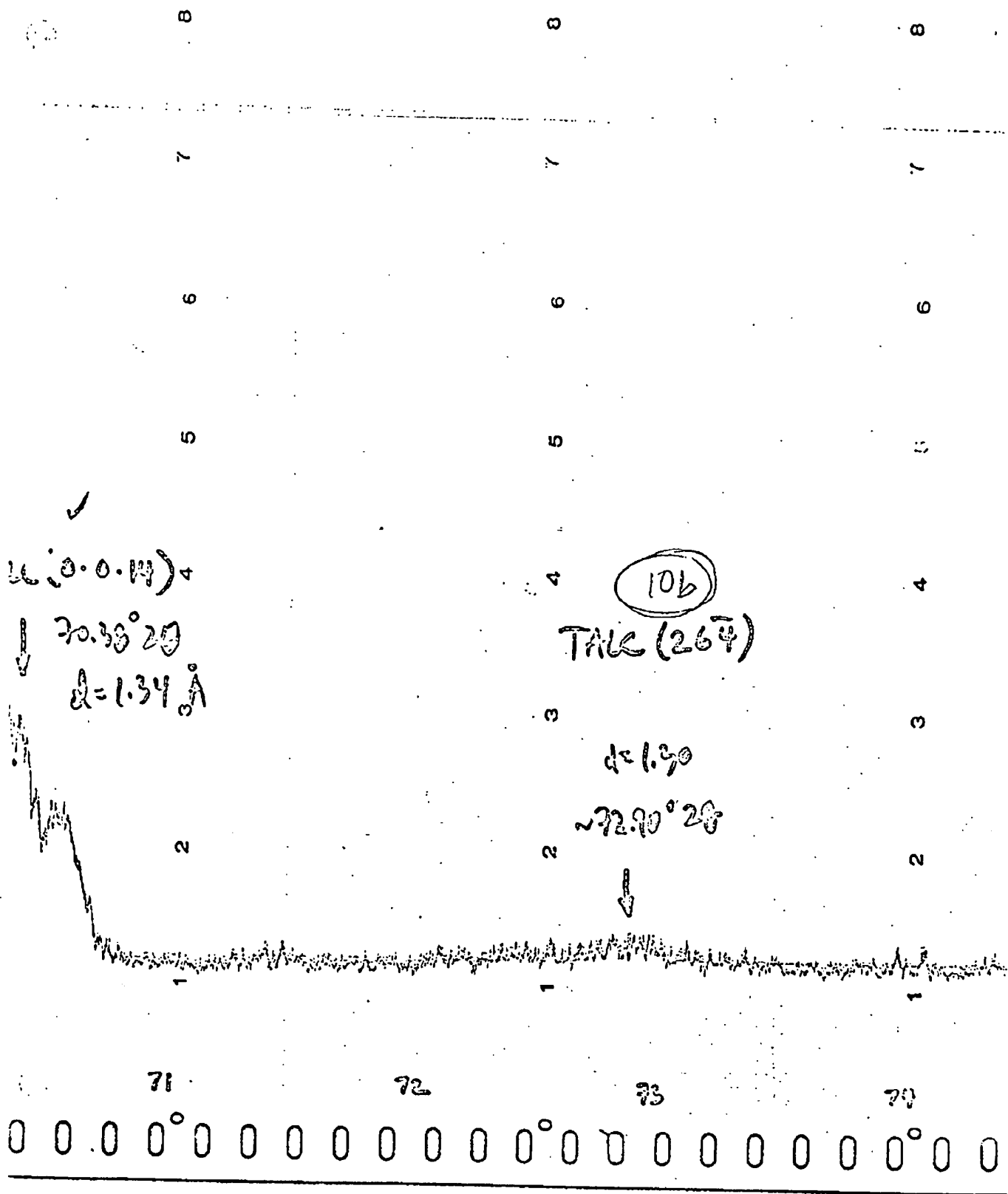


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J&J-0061505





J&J-0061506

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0° 0 0 0 0 0 0 0 0° 0 0 0 0 0 0 0 0 0 0 0 0

J&J-0061507

Prof. G. Brown

J&J-0130698

JNJAZ55\_000017718

CURRICULUM VITA: GORDON E. BROWN

Personal Data: Born Redacted - Personal Information 1943, San Diego, California;  
Married. One child.

Education: B.S. Geology and Chemistry, Millsaps College, Jackson, Miss., 1965.  
M.S. Mineralogy, V.P.I., 1968.  
Ph.D. Mineralogy, V.P.I., 1970.

Employment Experience:

Assistant Professor, Dept. of Geological and Geophysical Sciences. Princeton University, September, 1971- present.  
Post-Doctoral Research Associate, State University of New York at Stony Brook, May 1970- August 1971.  
Research and Teaching Assistant, Virginia Polytechnic Institute and State University, September 1966- April 1970.  
Research Assistant, Sun Oil Company, Jackson, Mississippi, June 1963- September 1965.

Professional Societies:

Mineralogical Society of America  
American Geophysical Union  
American Association for the Advancement of Science

Research Experience:

G.E. Brown investigated the crystal chemistry of the olivines as part of his doctoral research. The crystal structures of Ni-, Co-, (Fe,Mn)- (Mn,Mg)- (Fe,Ca)- and (Mn,Ca)- olivines were refined from single crystal X-ray data and cation ordering was studied in the (Fe,Mn)- and (Mn,Mg)- olivines. Earlier efforts were devoted to a study of bonding in the tetrahedral portions of silicates in collaboration with G.V. Gibbs. During post-doctoral work at Stony Brook, he collaborated with C.T. Prewitt and J.J. Papike on X-ray studies of the Apollo 12 clinopyroxenes and olivines. Included in this work are high-temperature X-ray studies of the  $P2_1/c \leftrightarrow C2/c$  transition in intergrown lunar pigeonite/angites, design of several high temperature furnaces for single crystal X-ray work (also in collaboration with S. Sueno), high temperature structural studies of pigeonite and cummingtonite, order-disorder studies of lunar olivines over a range of temperatures, and a neutron diffraction study of Al/Si ordering in sanidine.

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X-ray Diffraction Study of Johnson & Johnson's Shower-to-Shower  
Talcum Powder

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Abstract

An x-ray diffraction study of Lewin's sample of Johnson & Johnson's Shower-to-Shower talcum powder using slow, continuous scanning, diffractometer techniques with monochromatized Cu radiation showed that two forms of chlorite, phlogopite, magnesite, dolomite and calcite are minor contaminants (< 10% total) of the talc. No evidence of chrysotile or tremolite in Lewin's sample was found. Examination of the  $2\theta$  regions around  $12^\circ$  and  $24^\circ$  alone is not necessarily sufficient to establish the presence of chrysotile asbestos because of the similar peak positions of two other non-fibrous serpentine polymorphs, lizardite and antigorite. However, detailed examination of the range  $5-80^\circ$   $2\theta$  does permit discrimination between chlorite and serpentine peaks, assuming that these minerals are present in detectable amounts.

The x-ray data on J&J's Shower-to-Shower powder presented by S.Z. Lewin in a written report (May 16, 1972) to the Cosmetic, Toiletry and Fragrance Association and during a hearing (Sept. 21, 1972) before officials of the Food and Drug Administration show no evidence of chrysotile or tremolite when examined carefully. Moreover, the fibrous-looking particles in the photomicrographs also presented by Lewin at the Sept. 21 hearing can best be interpreted as rolled talc particles, which are sometimes produced during grinding. Lewin's claim that chrysotile is present in J&J's Shower-to-Shower powder is not supported by his data or by any of the results (from x-ray and electron diffraction, optical dispersion staining, transmission and scanning electron microscopy, electron microprobe analysis, differential thermal analysis, petrographic examination of the uncrushed Italian talc and detailed field study of the Italian mine) amassed by Johnson and Johnson's team of consultants.

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### Introduction

Talc deposits can result from a variety of processes including hydrothermal alteration of ultrabasic rocks (steatitization) and thermal metamorphism of siliceous dolomites (Turner, 1948). In the former process serpentinization may or may not occur before the development of talc. In either case the resulting talc may contain minor contaminants which can occur from incomplete steatitization, retrograde metamorphic processes or more commonly simultaneous crystallization of additional phases necessary to maintain mass balance. Contaminants such as magnesite, dolomite, calcite, chlorite, serpentine, tremolite, actinolite, phlogopite and quartz may result.

It is the purpose of this study to identify the contaminants present in Johnson & Johnson's Shower-to-Shower talcum powder using x-ray diffraction techniques. Because of S.Z. Lewin's claim that this powder contains ~ 5% chrysotile by weight, particular care was taken to verify or reject its presence using a continuous scanning x-ray technique similar to that employed by Lewin. The sensitivity of the continuous scanning technique to small concentrations of chrysotile in talc is estimated to be detection of ~2-3% chrysotile by weight. Lewin in a report to CTFA (5-16-72), claims a sensitivity of 1-2% chrysotile using a scanning rate of  $1^\circ 2\theta/\text{min}$ . Therefore, if chrysotile is present in the Shower-to-Shower powder in amounts of ~5% by weight, as claimed by Lewin, its presence should be easily detectable.

### Experimental

A powder mount of Lewin's sample of J & J's Shower-to-Shower powder was prepared by mixing 0.1 gr. of powder (< 325 mesh) with a mixture of acetone and duco cement on a glass slide. A very small quantity (~0.01 gr.) of powdered Si metal (325 mesh), for use as an internal standard, was sprinkled onto this mixture and stirred in. Powder mounts of three chemically diverse chrysotiles were also prepared in the same manner.

All x-ray data were collected using a Norelco vertical diffractometer mounted on a Norelco generator with a Cu standard focus x-ray tube. The diffractometer is equipped with a sample spinner, which is used to reduce the effects of preferred orientation, and a LiF crystal monochromator, which is set to produce a beam of Cu  $K\alpha$  x-rays. The PHA was tuned using the 111 peak of Si-metal and 2 V baseline and window settings were found satisfactory. Diffractometer alignment was then checked using Si-metal peaks which were located within  $0.01^\circ 2\theta$  of their correct values.  $1^\circ$  source and receiving slits were used in all runs. Smaller receiving slits, which are used to increase resolution, were found



to reduce intensity significantly. Enhancement of intensity was considered more important than increasing resolution because of the small amounts of impurities being searched for. All tracings were run at 40 KV and 20 mA using a scan speed of  $1/4^\circ 2\theta/\text{min}$ . and a time constant =2. Scans from  $5-80^\circ 2\theta$  were run for the Shower-to-Shower sample and the three crysotiles. Table 1 contains the  $2\theta$  values and d-spacings for all peaks in this interval for the Shower-to-Shower sample and Table 2 lists the chemical compositions and positions of the three strongest peaks for the three crysotile specimens studied. Figure 1 is a copy of the diffractogram for the Shower-to-Shower sample.

### Interpretation

The interpretation of a diffraction pattern from a complex mixture of phases is always difficult and necessitates great care. The standard philosophy employed in such an interpretation is outlined in Klug and Alexander (1954) and simply requires that all peaks be explained using the ASTM Powder Diffraction File or other standard patterns. The patterns in the ASTM file are for minerals of specific compositions, so the effects of solid solution on d-spacings must be taken into account. Because of the paragenesis of talc, a number of specific contaminants are expected and were searched for. In addition to peaks due to talc and the Si internal standard, 22 extra peaks were recorded in the diffraction pattern of J & J's Shower-to-Shower powder (See Table 1 and Figure 1). These additional peaks are due to two varieties of chlorite ( $\sim 5\%$  by weight), phlogopite ( $\sim 2\%$ ), magnesite ( $\sim 2\%$ ), dolomite ( $\sim 1\%$ ) and calcite (trace). Chlorite has the hypothetical composition  $\text{Mg}_{12}\text{Si}_8\text{O}_{20}(\text{OH})_{16}$ , however, Al can substitute for Mg and Si. When Al replaces about 3 Si per formula unit, the chlorite is called sheridanite; when about 1 Al substitutes for Si, it is termed a penninite. Because tetrahedral Al and Si ions differ by about  $0.14\text{\AA}$  in size, penninite and sheridanite have different d-spacings ( $\Delta \sim 0.1\text{\AA}$ ) and  $2\theta$  positions. The main peak at  $7.15\text{\AA}$  and its low angle shoulder are therefore interpreted to be due to sheridanite and penninite, respectively, with sheridanite and penninite constituting  $\sim 4\%$  and  $\sim 1\%$  by weight of the sample. For each of the contaminants listed above, identification was not accepted unless their peaks showed self-consistent intensity relationships. The presence of tremolite was not confirmed because its three strongest peaks ( $8.38$ ,  $3.12$  and  $2.70\text{\AA}$ ) are absent. Likewise, chrysotile can be rejected as a component because its two strongest peaks, the 002 and 004, predicted to occur at  $7.31$  and  $3.65\text{\AA}$ , respectively, are missing. Although the low angle shoulder ( $\sim 7.2\text{\AA}$ ) to the 002 chlorite peak ( $7.15\text{\AA}$ ) overlaps the  $7.3\text{\AA}$  region, it is due to the 002 of penninite, not the 002 of crysotile. If the 002 of crysotile were present,

the 004 of chrysotile is required to be present at 3.65Å and should have nearly the same relative intensity as the shoulder at 7.2Å. Examination of Figure 1 shows no peak in the 3.65Å region. Preferred orientation, which undoubtedly would have occurred for chrysotile if present, would enhance all of its basal reflections, not just the 002. One might argue that substitution of Al and Fe for Si and Mg in chrysotile would cause a shift in d-spacings such that the 002 and 004 of chrysotile are coincident with the chlorite peaks at 7.22-7.15Å and 3.56Å. To test this hypothesis, diffraction patterns of three chemically diverse chrysotiles were run and the results are reported in Table 2. The effects of these chemical substitutions on the basal spacing of chrysotile appear to be small. Therefore, it is doubtful that the 004 chlorite peak at 3.65Å obscures an 004 of chrysotile.

A word of caution is necessary even if peaks occur at 7.31 and 3.65Å. Besides chrysotile, there are two other serpentine polymorphs known as lizardite and antigorite which differ slightly in structure and totally in morphology (both are platy) from chrysotile. However, the two strongest peaks of each polymorph occur near 7.36 and 3.66Å (Whittaker and Zussman, 1956; Page and Coleman, 1967). Whittaker and Zussman (1956) emphasize that correct identification of a serpentine polymorph is possible only if a complete and accurate diffraction pattern is obtained. Obviously discrimination of this type would be impossible if small amounts of one or more of the serpentine polymorphs did occur in the Shower-to-Shower powder because of the weakness of the serpentine pattern and possible interference from other peaks.

#### Conclusions

The 22 extra peaks observed in the x-ray pattern of Lewin's sample of J&J's Shower-to-Shower talcum powder are interpreted as being due to sheridanite (4%), penninite (1%), phlogopite (2%), magnesite (2%), dolomite (1%) and calcite (trace). No evidence for chrysotile or tremolite was found. The diffraction patterns of three chemically different chrysotiles were run to check the effects of cation substitutions on basal spacings. These effects were found to be minor and it is concluded that the 004 peak of chlorite does not obscure an 004 peak of chrysotile. Although the x-ray diffraction pattern from a multi-component mixture such as Shower-to-Shower is complicated, it can be interpreted by careful use of the ASTM Powder Diffraction File.

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Table 1: X-ray Data for Lewin's Sample of Shower-to-Shower Powder

<u>hkl</u>	<u>Mineral</u>	<u>I/I<sub>0</sub>***</u>	<u>d(A)</u>	<u>2θ(deg.)</u>
001	Chlorite	60	14.5-14.3	6.10-6.15
001	Phlogopite	100	9.9-9.8	8.90-9.00
002	Talc	100	9.47	9.34
002	Chlorite (P)**	100	7.22	12.26
002	Chlorite (S)**	70	7.15	12.38
003	Chlorite (S+P)	50	4.75	18.70
004	Talc	90	4.70	18.88
020,111	Talc	30	4.59	19.35
020	Chlorite (S+P)	40B	4.58	19.39
004	Chlorite (S+P)	50(S) or 100(P)	3.56	25.00
022	Phlogopite	20	3.37	26.42
003	Phlogopite	100	3.35	26.60
111	Si Standard	100	3.14	28.46
006	Talc	100	3.13	28.65
220	Calcite	100	3.02	29.50
104	Dolomite	100	2.90	30.87
005	Chlorite (P)	60	2.88	31.02
005	Chlorite (S)	20	2.85	31.42
104	Magnesite	100	2.75	32.54
200,131	Phlogopite	30	2.63	33.98
132	Talc	30	2.61	34.42
131,202	Chlorite (S+P)	40	2.59	34.60
132,204	Talc	65	2.48	36.18
132,203	Chlorite (S+P)	50	2.44	36.8
008	Talc	16*	2.34	38.42
134	Talc	20	2.21	40.75
136	Talc	20	2.11	42.85
113	Magnesite	43	2.10	
135,204	Chlorite (S+P)	40	2.02	44.85
220	Si-Standard	60	1.92	47.18 (K $\alpha_1$ )
	Si-Standard		1.92	47.33 (K $\alpha_2$ )
0-0-10	Talc	40*	1.87	48.56
116	Magnesite	34	1.70	
244,138	Talc	20B	1.68	54.72°
311	Si-Standard	35	1.64	56.02 (K $\alpha_1$ )
0-0-12	Talc	20*	1.56	59.16
331,060	Chlorite (S+P)	20	1.54	60.32
060,332	Talc	40	1.53	60.55 (K $\alpha$ )
1-3-10	Talc	8B	1.46	63.2
139,208	Chlorite (P)	30	1.41	66.2
2-0-10	Talc	16	1.41	67.0
1-3-12	Talc	20	1.39	67.1
400	Si-Standard	8	1.36	69.20

620

<u>hkl</u>	<u>Mineral</u>	<u>I/Io</u>	<u>d(A)</u>	<u>2θ(deg.)</u>
0·0·14	Talc	16*	1.34	70.34
264	Talc	108	1.30	72.90
331	Si-Standard	13	1.25	76.30

\*Enhanced by preferred orientation

\*\* (S)= Sheridanite; (P)= Penninite

\*\*\* ASTM Intensity

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Table 2: Chemical and X-ray Data for Chrysotile

	<u>Jeffrey</u>	<u>Arizona</u>	<u>Coalinga</u>
Tetrahedral Cations	Si <sub>3.91</sub> Al <sub>0.07</sub>	Si <sub>3.98</sub> Al <sub>0.02</sub>	Si <sub>3.82</sub> Al <sub>0.18</sub>
Octahedral Cations	Mg <sub>5.78</sub> Fe <sub>0.19</sub>	Mg <sub>5.93</sub> Fe <sub>0.05</sub>	Mg <sub>5.62</sub> Al <sub>0.41</sub> Fe <sub>0.15</sub>
d <sub>002</sub> (Å)	7.36	7.35	7.31
d <sub>004</sub> (Å)	3.66	3.66	3.65
d <sub>060</sub> (Å)	1.54	1.54	1.54

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Comments on S.Z. Lewin's Presentation at the FDA- Johnson & Johnson Meeting (9/21/72)

That Professor Lewin is a polished speaker is not questioned after his presentation on the morning of Sept. 21; however, his interpretation and command of the theory of x-ray diffraction and optics is suspect. After a somewhat theatrical and generally non-relevant discourse on the structures of talc and chlorite, he claimed that Shower-to-Shower did indeed contain chrysotile. It appears difficult for Professor Lewin to decide on a fixed amount of chrysotile (his estimates vary from 2-5% on a given date) or whether tremolite is present. The diffraction patterns of sample numbers 136 and 137 handed out by Lewin are totally useless in deciding almost anything except the presence of talc and chlorite. On the other hand they do show, if marked correctly, that a  $\beta$ -filter was not used, a dangerous practice in general which only complicates an x-ray pattern. They do not show evidence of chrysotile or tremolite. His interpretation of the photomicrographs demonstrated a seeming lack of experience and little knowledge of the optical properties of talc. It is not uncommon for talc edges to be rolled or bent during mechanical grinding. This would explain Lewin's observation that some of the talc plates display two different refractive indices which he interpreted as indicating talc intergrown with chrysotile. According to Deer, Howie and Zussman (1962) the refractive indices of talc are  $n_x \cong 1.54$ ,  $n_y \cong 1.590$ ,  $n_z \cong 1.590$ . When a talc plate has rolled edges, one of the measured indices for the rolled edge will be near 1.54 with the unrolled plate exhibiting indices near 1.590. Lewin also claimed that parallel extinction of these edges and other fibrous-looking particles precludes their being talc. Talc always shows parallel extinction when viewed parallel to the plates or when rolled into fibrous-looking tubes. His claim that the "intergrown chrysotile" represents a new variety with different physical properties and structure from the normal fibrous variety is totally without sound scientific evidence. No such discovery has been claimed in the rather extensive literature on talc and chrysotile. Neither Lewin's x-ray data nor his optical data supports his claim that chrysotile is present in Shower-to-Shower.

Gordon S. Brown