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Report on

GEIGER COUNTER TECHNIQUE FOR X-RAY DIFFRACTION
PART I. - SPECTROMETER FOR MEASURING POWDER DIFFRACTION PATTERNS

Naval Research Laboratory
Anacostia Station
Washington 20, D. C.

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Prepared by:

Herbert Friedman, Senior Physicist

LaVerne S. Birks, Associate Physicist

Reviewed by:

E. O. Hulburt, Superintendent
Physical Optics Division

Approved by:

A. H. Van Keuren, Rear Admiral, USN
Director

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GEIGER COUNTER TECHNIQUE FOR X-RAY DIFFRACTION
PART I. - SPECTROMETER FOR MEASURING POWDER DIFFRACTION PATTERNS

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Plate

REFERENCES

1. Memorandum to the Director, Naval Research Laboratory, 17 October 1942.
2. A. Hull, Phys. Rev., Vol. 10, p. 661, 1917.

I. ABSTRACT

A Geiger counter X-ray spectrometer for powder diffraction is described in this report. The counters are almost 100 percent efficient when used with target radiations from molybdenum to chromium. The spectrometer employs a focusing geometry which yields intense diffraction patterns. For the purpose of chemical identification, the counter spectrometer is many times as fast as cameras of equivalent resolution. It simplifies the technique and extends the range of X-ray diffraction in quantitative chemical analysis. Spacings are measurable from 1.0 Å to 150 Å with copper radiation.

II. INTRODUCTION

The problem in X-ray diffraction measurement is to locate an array of X-ray interference maxima on a given surface and to determine the intensity distribution in the pattern. Photographic registration is generally satisfactory for finding the positions of X-ray interferences, but quantitative intensity measurement is difficult. The photographic method leaves much to be desired in crystal structure determinations, quantitative chemical X-ray analysis, the study of alloy phase diagrams, measurement of preferred orientation and many other diffraction problems where accurate intensities are required.

The Bragg type ionization spectrometer is still used for intensity determinations with single crystals. The sensitivity of the ionization chamber, however, is not adequate for powder patterns, where currents of the order of 10^{-16} amperes must be measured. For weak intensities, the Geiger counter is the most efficient detector. Counters of close to 100 percent quantum efficiency, developed at this Laboratory, are now incorporated in a variety of X-ray spectrometers. These instruments produce diffraction data with quantitative accuracy at greater speed than is possible photographically. One such instrument, the quartz crystal analysis unit which is used in the manufacture of quartz crystal oscillator plates, has been briefly described, (1). The present report covers the technique of preparing sensitive X-ray counters and the design of a powder diffraction spectrometer. Illustrations of the use of the instrument for chemical identification and quantitative analysis are included. Subsequent reports will cover counter technique (1) for obtaining fiber patterns, (2) for particle size measurement, (3) for metallurgical diffraction problems, (4) for measurement of plating and film thickness, and (5) for stress analysis.

III. GEIGER COUNTERS FOR X-RAY DIFFRACTION

The photoelectric absorption of an X-ray quantum (about 1.0 A.U.) in the gas atmosphere of a counter produces of the order of 1000 ion pairs. Since a single photoelectron produced anywhere within the volume of a Geiger counter has a high probability of triggering a discharge, each quantum absorbed is recorded as a count, unless the absorption act follows a previous count by an interval less than the dead time of the counter. A counter, in which the X-ray beam is completely absorbed in the gaseous filling, is therefore 100 percent efficient except for counting losses due to finite resolving power. Such an instrument measures absolute intensity directly in units of quanta.

The development of high pressure self quenching Geiger Muller counters has made it possible to achieve close to 100 percent gaseous absorption, or 100 percent quantum efficiency. For every target radiation commonly employed in X-ray diffraction problems, a gas filling can be chosen, which absorbs almost the entire beam in a ten centimeter path. Referring to Plate (1), krypton is the most effective absorber of molybdenum K_α radiation, while copper, nickel, iron and chromium characteristic radiations are strongly absorbed in argon. Both these noble gases can be successfully used in Geiger counters, at pressures from 20 to 70 centimeters of Hg, with addition of alcohol vapor to make the counters fast and self quenching. The preparation of such counters is treated in NRL Report M-1800.

The geometry of the counter must permit a pencil of X-rays to travel a ten centimeter path without obstruction. The X-ray counter is constructed as shown in Plate (2) with a stiff 20 mil tungsten wire anode supported from the stem end of the tube. A diffracted beam entering the window of the tube, off the axis, can be absorbed in the gas without striking either of the electrodes. A pyrex bubble window, 10 microns thick absorbs relatively little of the radiation from targets of iron and higher atomic number elements. Lindemann glass 1/2 mm thick has about the same transmission as a pyrex bubble window. Beryllium windows 1/16 inch thick are satisfactory for all the common targets including chromium. Transmission characteristics of various window materials are plotted in Plate (3).

IV. THE GEIGER COUNTER SPECTROMETER FOR POWDER DIFFRACTION

An X-ray beam is diffracted from a crystal if the geometrical conditions stated by Braggs' Law are fulfilled. The Bragg law relates the angle, θ, which the diffracted beam makes with a set of reflecting planes, to the wavelength, λ, of the X-rays, the planar spacing, d, giving rise to that particular reflection, and the order, n, of the interference. The relationship is expressed as,

$$n\lambda = 2d \sin \theta \qquad (1)$$

In the powder method, the specimen which is composed of a great many crystallites oriented at random, is irradiated with monochromatic X-rays. The usual photographic arrangement is illustrated in Plate (4), Fig. 1. For every possible set of reflecting planes, some crystalline fragments are aligned so as to yield reflections in accord with (1). Planar spacings, d, are defined in terms of the conventional planar indices, h, k, l, and the lattice parameters. Rotating any reflecting crystal around the direction of the primary beam causes the diffracted beam to sweep out a cone of half apex angle $2\theta_{hkl}$ corresponding to the d_{hkl} of the reflecting planes. The cylindrical film strip, in the camera of Plate (4), intersects these cones in short arcs or diffraction lines, each line corresponding to a particular d_{hkl}. Plate 4, Fig. 2)

Geiger counter spectrometers for powder diffraction at first involved only simple substitutions of counters for film. The camera geometry was retained with the counter carried by an arm pivoted at the specimen axis. A spectrometer of this type, built upon a quadrant cassette formerly used with an early model multiple diffraction unit, is shown in Plate (5). It is essentially a Bragg ionization spectrometer, Plate (6), with a capillary specimen replacing the single crystal.

With slight modification, the above system is converted to a focusing spectrometer giving more intense diffractions without loss of resolution. Referring to Plate (6), this is accomplished by removal of slit S_2 and replacement of the capillary specimen by a powder spread on a flat sheet with a suitable binder. This sheet specimen is mounted like the single crystal of Fig. 1. The beam from the target focal spot diverges beyond S_1, toward the specimen, and is "focused" after reflection, upon slit S_3. The "focusing" action is illustrated in Plate (6), Fig. 2. A hypothetical circle is drawn through three points locating the primary and receiving slits and the specimen axis. All angles inscribed in the chord joining S_1 and S_4 are equal. In practice, the specimen is planar and not curved to conform to the focusing circle. If the length of the specimen is one centimeter on a spectrometer of ten centimeter radius, the focusing error is insignificant. A one centimeter specimen contributes ten to twenty times as much intensity to the diffraction pattern as the capillary specimen of Plate (4).

The main features of the Geiger counter spectrometer for X-ray powder diffraction, illustrated in Plates (7, 8) include: a zero to 90° scale graduated in one degree steps and a micrometer dial calibrated in minutes, with negligible backlash; parallelogram slits for S_1, and S_4, with wedge type slit height adjustments; a fixed slit mounted in front of S_4 to prevent scattered radiation from reaching the counter; provision for insertion of a secondary slit in the primary beam, for low angle patterns. The spectrometer radius is 10 centimeters. With slits, S_1 and S_4, set at 0.5 millimeter width and 4 millimeter height, the 011 reflection from a powdered rock salt specimen has a half maximum breadth of about 20 minutes in 2θ for copper radiation. With half millimeter apertures in the photographic technique of Plate (4), the line width is close to 50 minutes. Without inserting the low angle slit, measurement is possible out to 20 Angstrom unit spacings. By insertion of the additional slit for low angle work, the limit is extended to 0.5 degree in 2θ, or about 150 A.U. The peaks of lines can be located within one minute of arc, anywhere on the scale, and repeated within this limit from sample to sample.

The Geiger counter spectrometer is equipped with two means of intensity indication: (1) a frequency meter circuit which integrates counter pulses to give continuous meter indication of counting rate; (2) a scale-of-64 counting circuit with electronically timed counting intervals of ten seconds, twenty seconds, and one minute. The frequency meter is adequate for rapid scanning of patterns to determine positions and approximate relative intensities of lines. The counting system is used for measurements of weak intensities. Either form of measurement lends itself readily to automatic recording.

The circuits are diagrammed in Plate (9). They are designed for greater resolving power than the Geiger counter, so that the limiting counting rate is attributable directly to the Geiger Counter. With D.C. voltage on the X-ray tube the resolving power is roughly 2200 counts per second. When a self-rectified tube is used the average resolving power drops to 700 counts per second

V. CHEMICAL IDENTIFICATION WITH THE GEIGER COUNTER X-RAY SPECTROMETER

The foundation for present methods of X-ray chemical analysis was laid by Hull (2) in 1919. He found that crystalline substances all gave X-ray patterns, that each substance always gave the same pattern, and that mixtures of substances gave the superposed individual patterns. Hanawalt is largely responsible for the development of a practical system of identification from X-ray powder patterns. Identification by the Hanawalt method differs from chemical analysis in that it determines a crystalline structure rather than the component atoms. The powder pattern is used as a fingerprint and the identification is made by comparison with patterns of known materials.

The "d" values (planar spacings) and relative intensities of the lines completely specify a powder pattern. For identification purposes, standard spectra are classified according to the spacings and intensities of the three strongest lines. In the Hanawalt card index system, three cards are assigned to each substance. One card lists the spacing of the strongest line first, another lists the second strongest line first and the remaining card lists the third strongest line first. All cards are arranged in a file according to first spacing listed.

In his first collection of the spectra of 1000 inorganic substances, Hanawalt found that fifty gave amorphous or liquid patterns which could not be employed for X-ray identification. The remaining specimens gave patterns of varied strength. The best patterns permitted identifications in concentrations under one percent. The poorest patterns gave difficulty even when present in concentrations of fifty percent.

X-ray identification has a definite future in conjunction with spectroscopic analysis. It has the advantage of requiring relatively minute samples. Amounts as small as 0.1 milligram can be conclusively identified and without

destruction of the sample. Identification by the X-ray method is always certain; if a pattern is obtained the material is definitely present in the form indicated by the characteristic pattern. Appendix I contains a series of abstracts from publications on applications of X-ray powder diffraction to chemical identification.

The Geiger counter technique described here for X-ray powder patterns removes many of the limitations that have retarded the general adoption of the X-ray method of analysis. The entire process of obtaining data is speeded up by a considerable factor, quantitative intensity measurement is obtained directly, and operation of the instrument and specimen preparation require no especial skill. An appreciation of the method can best be obtained by examination of the data presented on plates (10-18). Consider the specific problem of obtaining the Cu_2O patterns shown in Plates (10,11) by the use of a medium range powder camera and by the Geiger counter spectrometer method.

(1) Exposure time against scanning.

The photographic pattern was obtained in five hours on Type F film; the counter record required thirty minutes. (Both patterns were run with five milliamperes at 45 KV.) The irregularities in the microphotometer curve are characteristic of the grain size of the film. If Type A film is used for fine grain size and low background, the exposure is increased by a factor of four.

(2) Resolution.

The specimen which yielded the curves of Plates (11, 12) was a mixture of CuO and Cu_2O. The higher CuO spacing is not resolved on the film. At half maximum intensity, the breadth of the Cu_2O line on the counter curve is only one third that of the microphotometer trace, which was taken with 0.7 millimeter slits on the camera. To match the sharpness of the lines measured with the Geiger counter spectrometer, the slit width must be decreased considerably, with a resulting increase of five to ten times in exposure.

(3) Specimen preparation and setting up time.

For photographic exposure, one-half hour must generally be allowed to prepare and mount the sample, to load the camera with specimen and film, and to adjust the camera on the X-ray tube. After exposure, still more time is consumed in photographic processing and microphotometering. In contrast, a spectrometer specimen can be prepared in a few minutes. A few drops of collodion are spread on a glass slide and pigment is added to the collodion. The pigment is first worked into the binder with a spatula, then spread thin and allowed to dry. Mounting specimens on the spectrometer involves no difficulty. The data may be obtained directly in "d" values and absolute intensities.

(4) Other considerations.

For identification, where positions and relative intensities of only the strongest lines are needed, the counter system is capable of locating and measuring these lines in a few minutes. Where quantitative analysis is required and the line positions are known, counts are needed at only a few points for peak, background, and half maximum intensities. In a recent problem 85 copper oxide pigments were analyzed for cuprous and cupric contents within one percent. The entire job occupied two days. The same problem would require months to complete by photographic methods.

VI. ILLUSTRATIONS

Plates (10) and (11) offer a comparison between the Geiger counter spectrometer measurements of $Cu_2O + CuO$ mixtures and microphotometer traces of photographic patterns of the same samples. The counter records show markedly less background variation, greater resolution, and superior sensitivity. For quantitative analyses of the CuO content of such samples with the counter, it is only necessary to measure the peak intensity of the strongest CuO line (d = 2.31) and compare it with the intensity from a standard preparation. These peak intensity measurements can be carried out with an accuracy better than one percent, by counting for one minute.

Titanium dioxide pigment is widely used in the paint industry. The chemical compound TiO_2, may be prepared in two crystalline modifications, the rutile and anatase forms. Of the two, rutile titanium dioxide has much the greater covering power and brightness. The X-ray pattern offers a quick means of analyzing any mixed sample for relative amounts of the two forms. Plate (12) is the pattern of a sample containing about 30 percent of the rutile form. Here again quantitative analysis by the counter method involves only a measurement of peak intensity of the strongest line of either component.

To demonstrate the sensitivity of the instrument, three strontium compounds, Plate (13), were chosen from the weakest patterns listed in the A.S.T.M. card index. In this index system, the strongest line in the NaCl pattern is rated 100 and other line intensities are compared with this line as the absolute standard. The strongest lines in the three strontium compounds are rated at 12 for $SrCo_3$, 7 for $Sr(NO_3)_2$, and 5 for $SrSO_4$. The counter spectrometer registers all the lines of these patterns in the individual compounds and in a mixture of all three, without difficulty. (The intensity scale for the mixture pattern is double that for the three individual compounds on Plate (13)).

Chemical reactions are followed with quantitative accuracy by the X-ray spectrometer. For example, magnesium carbonate is converted to magnesium oxide by baking. The course of the reaction is followed (Plate 14) by studying a portion of the $MgCO_3$ pattern. At 200° C, roughly 50 percent of the carbonate is converted to oxide at the end of two hours. At 400° C the conversion is complete at two hours. Such processes may be studied in detail by the X-ray technique.

X-ray analysis is capable of identifying many natural and synthesized organic compounds. The pattern for glycine (Plate 15) is typical of the strong powder patterns obtained for a majority of crystalline organic compounds. By means of X-ray diffraction it is possible to determine molecular weight and molecular symmetry; with refined technique and correlation with chemical data the general size and shape of the molecule, the positions of sub groups, and in some cases the positions of all the atoms in the molecule are obtainable.

The paraffin hydrocarbons, acids, esters, soaps, etc., are all characterized by unit cells which have one dimension much longer than the others. This dimension grows in constant increments with increasing number of carbon atoms and is a measure of the length of the molecular chain. The side spacings remain nearly constant and correspond to cross-sectional dimensions of the chains. The pattern of plate (16) for arachidic acid was obtained from a specimen prepared by melting the material on a glass slide. The pattern shows a single strong line repeated through a number of higher orders of diffraction. The solidified film acts like a single crystal with many crystalline grains oriented exactly alike with respect to one axis, but at random around this axis. Bragg has drawn the analogy of a carpet as a layer of the crystal, the pile of the carpet as the molecules in a layer, and a stack of carpets as the crystal. The X-rays measure the perpendicular distance between successive layers and this spacing increases a constant amount for each addition of a CH_2 group. In the aliphatic acid series, the reflecting layers in any one acid are spaced twice as far apart as in the paraffin with the equivalent number of carbon atoms. The layers are therefore two molecules thick with COOH groups meeting at the ends of two chains turned in opposition to each other. The type of pattern shown for arachidic acid (20 carbons) in plate (16), is repeated for all the other members of the homologous series. The positions of corresponding reflections for stearic acid, containing 18 carbons, and other acids down to 10 carbons, are indicated by vertical lines alongside the first, second, and third order spacings shown for arachidic acid.

The technique of measurement at low angles in the above patterns requires no change in the spectrometer other than insertion of the "low angle" slit. These patterns cannot be registered with orthodox camera technique. Other illustrations of low angle measurements are given in Plates (17) and (18) showing long spacings in cholesterol and in some commercial soaps.

VII. APPENDIX I

SOME ILLUSTRATIONS OF THE USE OF X-RAY POWDER PATTERNS IN CHEMICAL ANALYSIS

The following examples represent a few of the cases reported in technical papers in which X-ray powder diffraction patterns provided solutions to the problems.

Pigments and Luminescent Materials

When solutions of ferrous salts are treated with milk of lime or calcium carbonate and then oxidized by blowing in air, they yield precipitates of various colors, which are used as pigments. Under certain conditions, a black pigment is also obtainable. By heat treatment, these pigments are converted into pigments of other colors. The crystals are too small for identification by microscope technique. X-ray diffraction identifies the yellow pigments as the mineral goethite (α - $FeO(OH)$), the brown pigments as lepidocrocite (γ - $FeO(OH)$). Orange precipitates give patterns not corresponding to that of any known mineral, indicating a third crystalline form of $FeO(OH)$. Three colors therefore result from three different arrangements of the same ions. The black precipitate was Fe_3O_4. Heating to 500° C converts all pigments to red, Fe_2O_3, the rhombohedral form. More moderate heating produces all shades of purple to brown in the cubic form of Fe_2O_3 where the atomic arrangement is that of Fe_3O_4 with some iron atoms missing.

Fluorescent and phosphorescent materials are used in discharge tube lighting and cathode ray tubes screens. New compounds for these purposes have been discovered through the use of X-ray diffraction. Many phosphors fluoresce only when a small amount of an impurity is incorporated during preparation. X-ray provided the first information as to the position of the activator in the matrix material. For many phosphors it was proven that a manganese activator does substitute for atoms of the matrix lattice. Addition of Mn to zinc orthoscilicate (green fluorescence) expands the lattice by measurable amounts. The phosphor is a solid solution of manganese orthosilicate in zinc orthosilicate. The displacement of the X-ray lines is measurable for as little as 0.5% Mn by weight. In red fluorescent cadmium silicate, the lattice parameter contracts as a result of random displacement of cadmium by manganese atoms.

Replacement of Zn by beryllium causes marked contraction of the lattice. Solid solution of beryllium orthosilicate takes place up to ten or fifteen molecular percent and is accompanied by considerable change in the fluorescent color.

The effect of solution of cadmium sulphide in zinc sulphide is to displace the green fluorescent band of ZnS towards the red. X-ray diffraction as a means of determining composition of a zinc-cadmium sulphide solid solution is accurate to one or two percent.

Clays, Ceramics, Refractories

In the heat treatment of kaolin, it was formerly believed that the original kaolin structure broke down at low temperatures (400° - 500° C) into an anhydrous silicate. X-ray diffraction showed the decomposition product was an amorphous complex of silica and alumina. The X-ray pattern of kaolin at 700° C shows diffuse diffraction bands characteristic of amorphous silica as well as faint lines due to heat treated mica. At higher temperatures both the partial crystallization of the alumina as γ - alumina and the formation of the compound mullite are observed. Growth of mullite crystals with temperature can be followed by sharpening of X-ray lines. Diffraction patterns also indicated differences in mullites from different sources.

In selecting silica for refractories diffraction patterns show the relation between crystalline texture of the raw material and its suitability for use in the manufacture of high grade silica brick.

X-ray patterns show the relative amounts of residual quartz, and newly formed cristobolite and tridymite in rock samples for brick manufacture after firing at 1450° C.

X-ray analysis of fire clays is used to determine correct firing temperatures. By X-ray means it is possible to assess consistency of supplies and to follow service behavior with changes in alumina-silica ratio and firing temperature.

Miscellaneous Chemical Reactions

X-ray diffraction was used throughout a laboratory investigation of boiler scale formation to determine the rate of reaction between various constituents of the scale. Calcium carbonate and sulphate scales could be identified by measurement of refractive indices with the microscope, but silicate and phosphate scales could only be studied by X-ray technique.

In the manufacture of one type of thermionic cathode, nickel wire is coated with barium and strontium oxides. X-ray diffraction showed that for maximum thermionic emission, the coating of the cathode before heat treatment should be in the form of mixed crystals of the carbonates. Study of the cathode after operation indicated preferential loss of barium oxide. The correct rating for the cathode, i.e., the temperature range over which the cathode operates without serious loss of barium, is determined from X-ray diffraction data.

Chemical and physical changes accompanying adsorption of substances on charcoal or silica gel are revealed by X-ray diffraction.

The question of the constitution of bleaching powder was solved by powder pattern methods when all chemical techniques failed.

Reactions in lead storage batteries were traced by X-ray diffraction studies of material taken from the positive and negative plates of batteries when fully charged and in the completely discharged condition.

Powder diffraction was used to follow the process of dehydration of gypsum to Plaster of Paris and to identify reactions accompanying the setting of plaster.

Some processes in the softening of water by chemical agents were completely understood only after X-ray diffraction analysis.

CIRCUIT CONSTANTS FOR COUNTER CIRCUIT F 653A

R1	150,000 ohms	R18	20,000 ohms
R2	70,000 "	R19	25,000 "
R3	50,000 "	R20	1 megohm
R4	30,000 "	R21	100,000 ohms
R5	20,000 "	R22	2 megohm
R6	150,000 "	R23	50,000 ohms
R7	50,000 "	R24	50,000 "
R8	250,000 "	R25	50,000 "
R9	20,000 "	R26	2 megohm
R10	25,000 "	R27	25,000 ohms
R11	5,000 "	R28	5,000 ohms
R12	12,500 "	R29	10,000 "
R13	12,000 "	R30	3 megohm
R14	33,000 "	R31	2 "
R15	2 megohm	R32	1 "
R16	50,000 ohms	R33	5 "
R17	30,000 "		

C1	100 mmf	C10	8 mf
C2	50 mmf	C11	20 mf
C3	50 mmf	C12	40 mf
C4	20 mf	C13	25 mf
C5	20 mf	C14	.25 mf
C6	50 mmf	C15	20 mf
C7	5000 mmf	C16	2 mf
C8	50 mmf	C17	4 mf
C9	50 mmf	C18	.005 mf

ABSORPTION OF X-RAYS BY GASES FOR VARIOUS WAVE LENGTHS AND PRESSURES

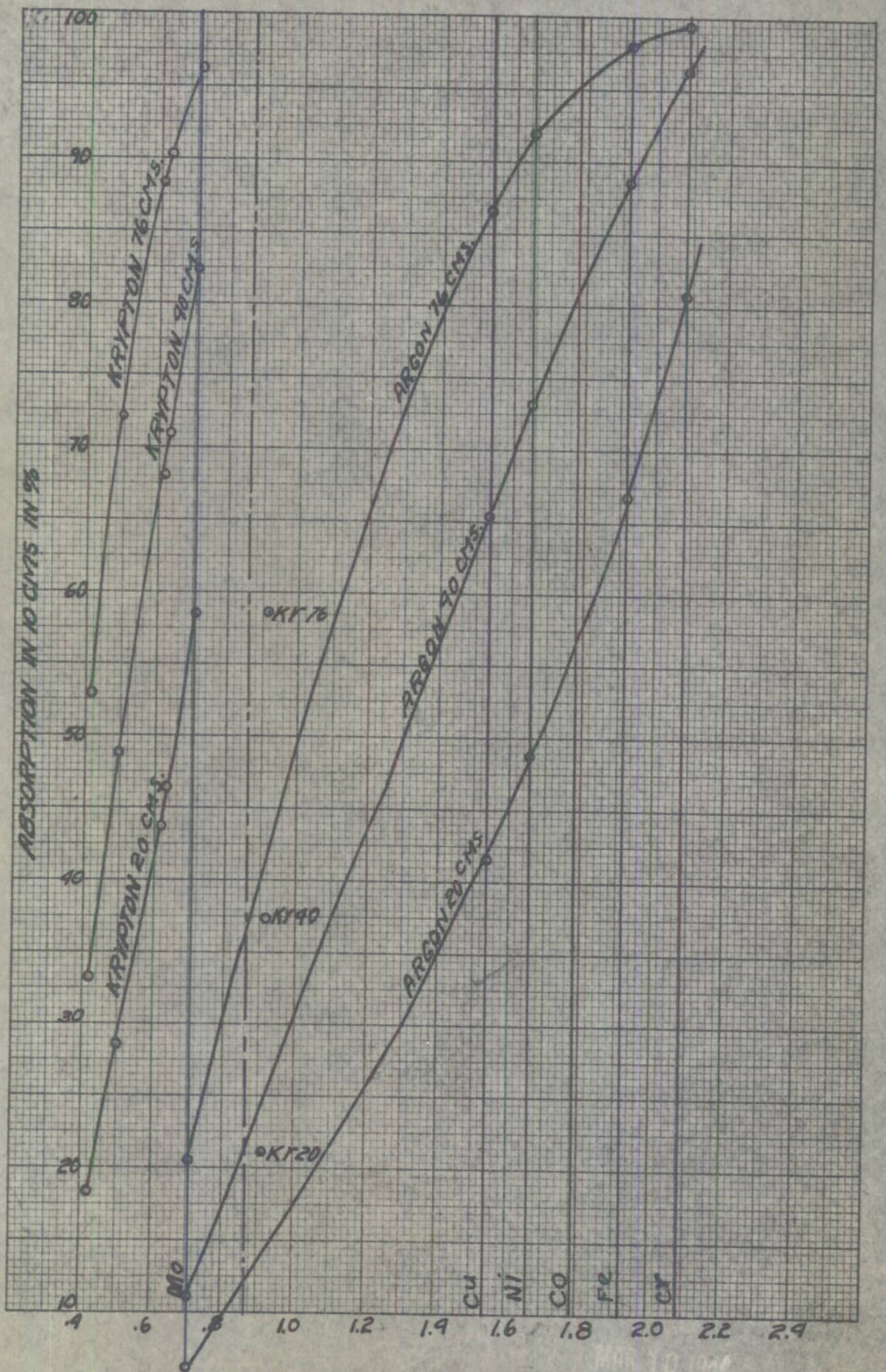


PLATE I

NO. 3110. 20 DIVISIONS PER INCH BOTH WAYS. 120 BY 180 DIVISIONS.
CODEX BOOK COMPANY, INC., NORWOOD, MASSACHUSETTS.
PRINTED IN U.S.A.

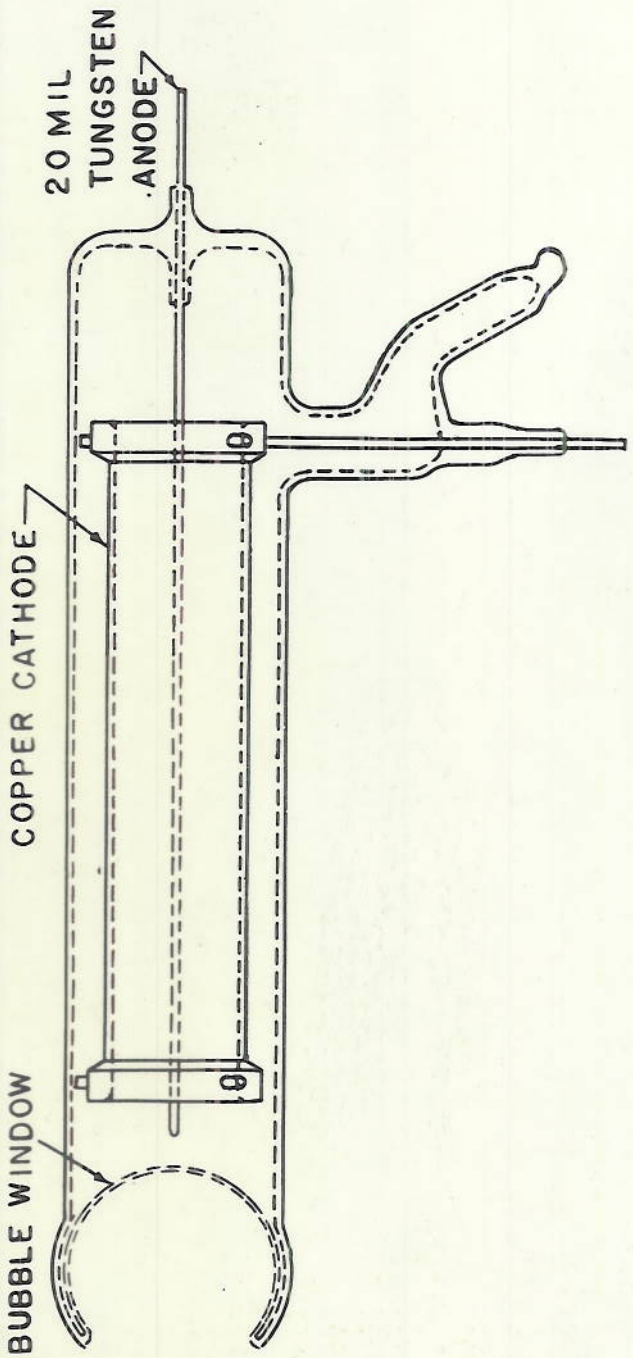


PLATE 2

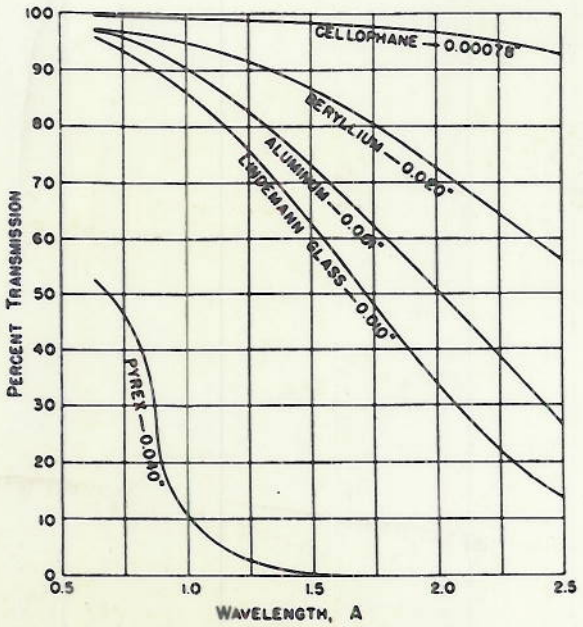


FIG. 1. X-ray transmission of windows used in diffraction tubes.

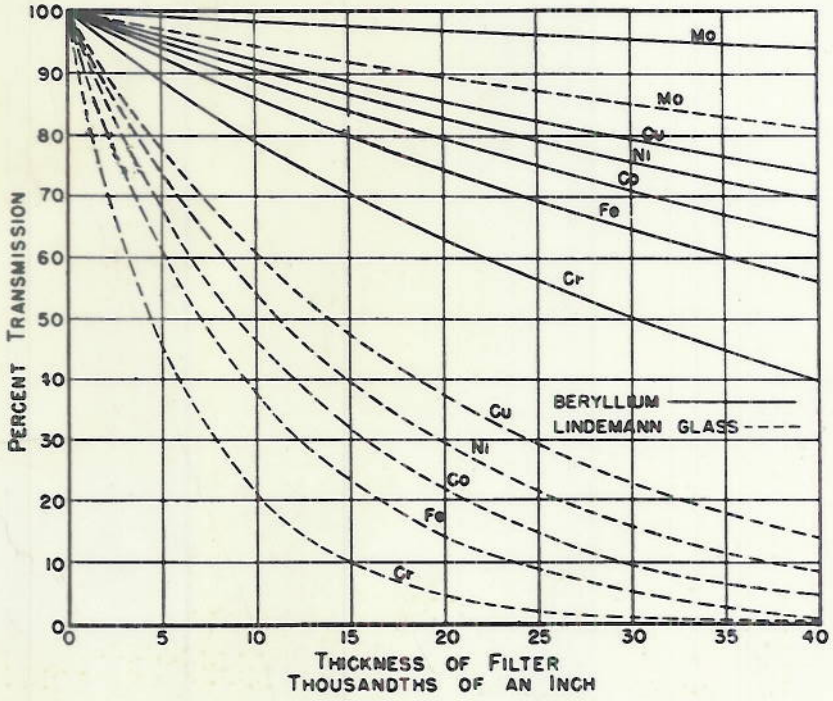
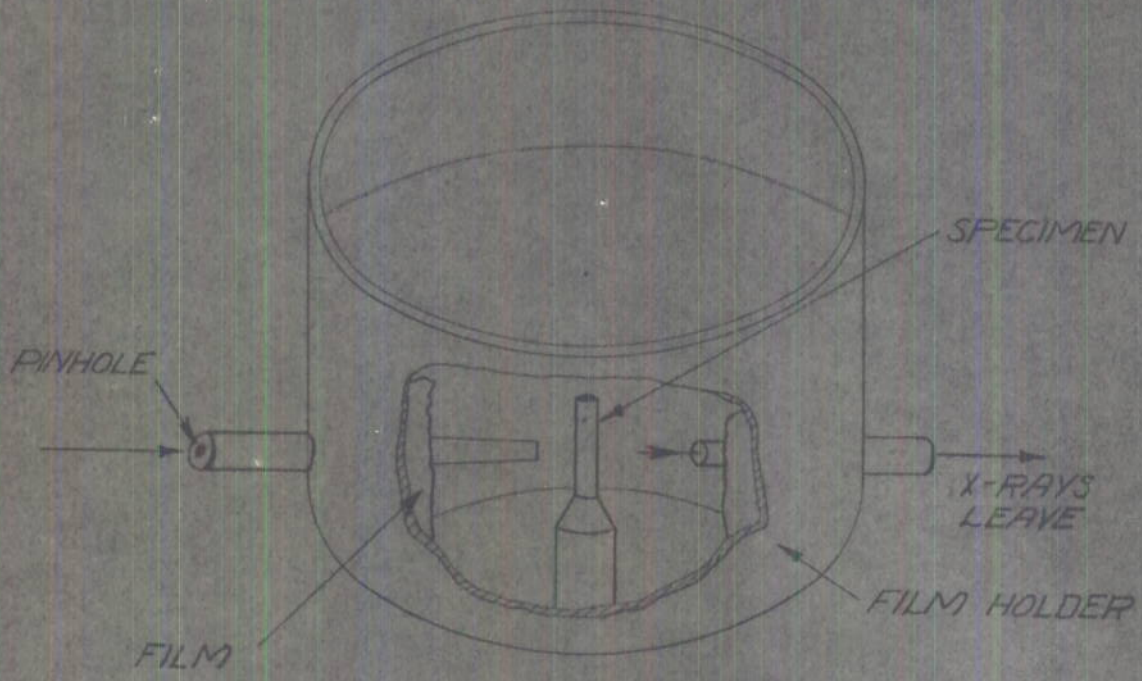


FIG. 2. X-ray transmission of beryllium and Lindemann glass for the $K\alpha_1$ radiations commonly used in diffraction work.

PLATE 3



POWDER CAMERA

FIG. 1

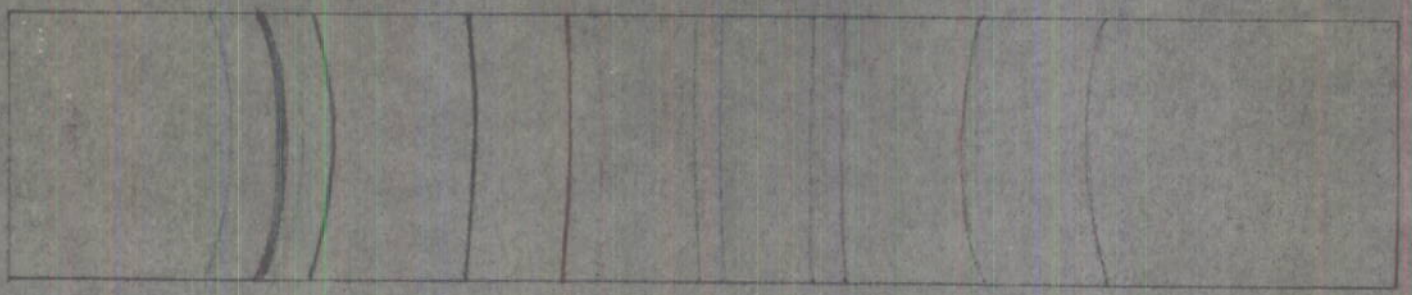


FIG. 2

PLATE 4

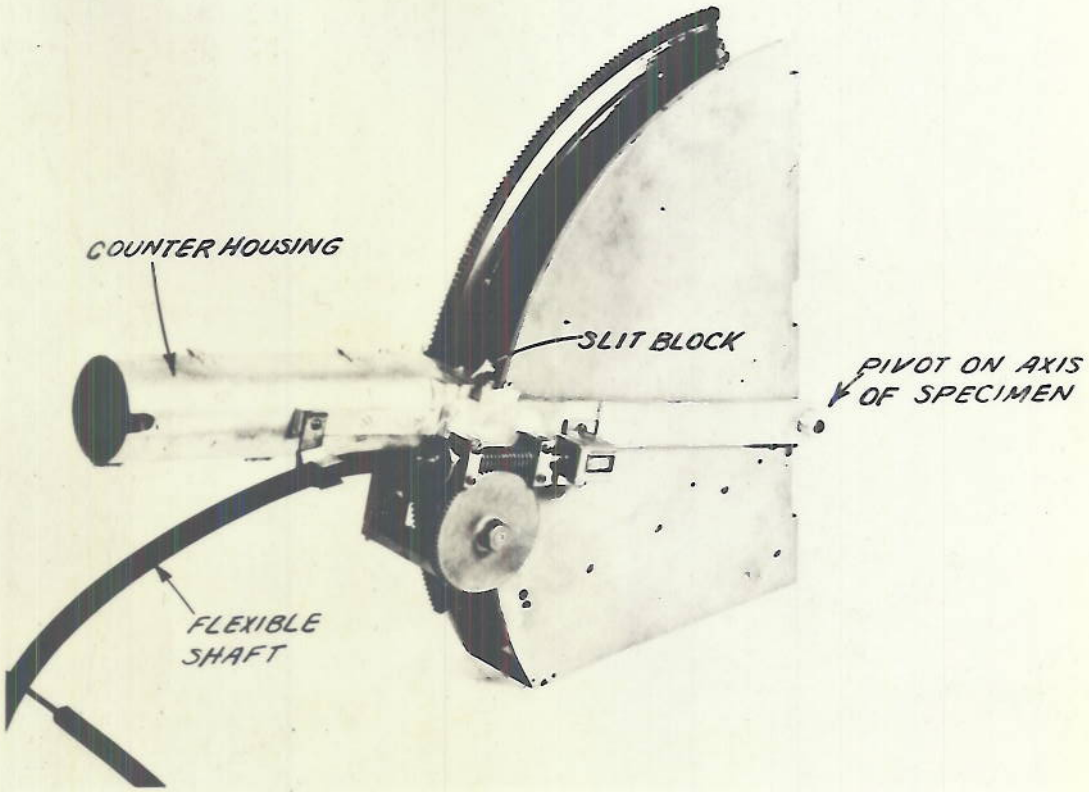
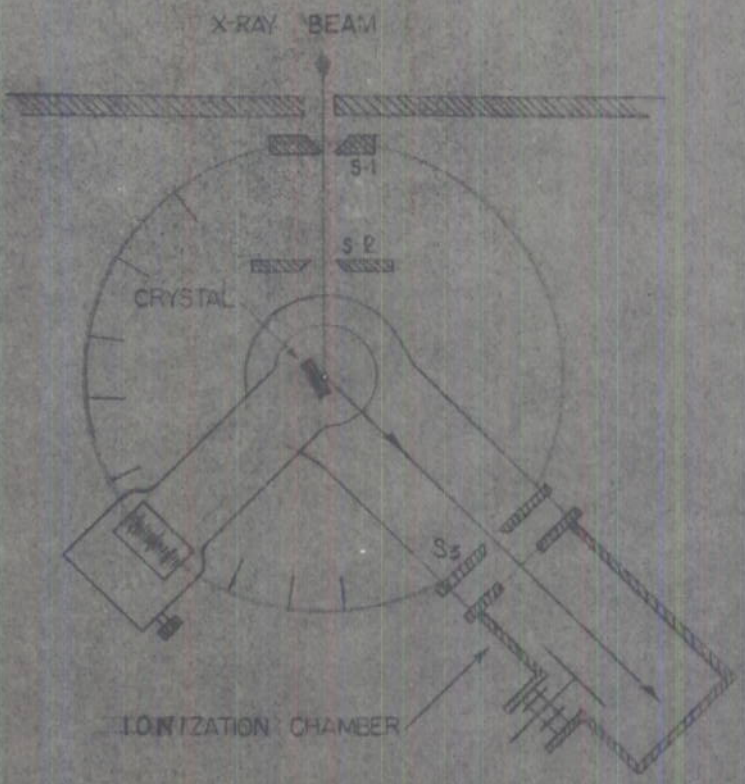

COUNTER HOUSING
SLIT BLOCK
PIVOT ON AXIS OF SPECIMEN
FLEXIBLE SHAFT

PLATE 5



BRAGG IONIZATION SPECTROMETER

FIG. 1

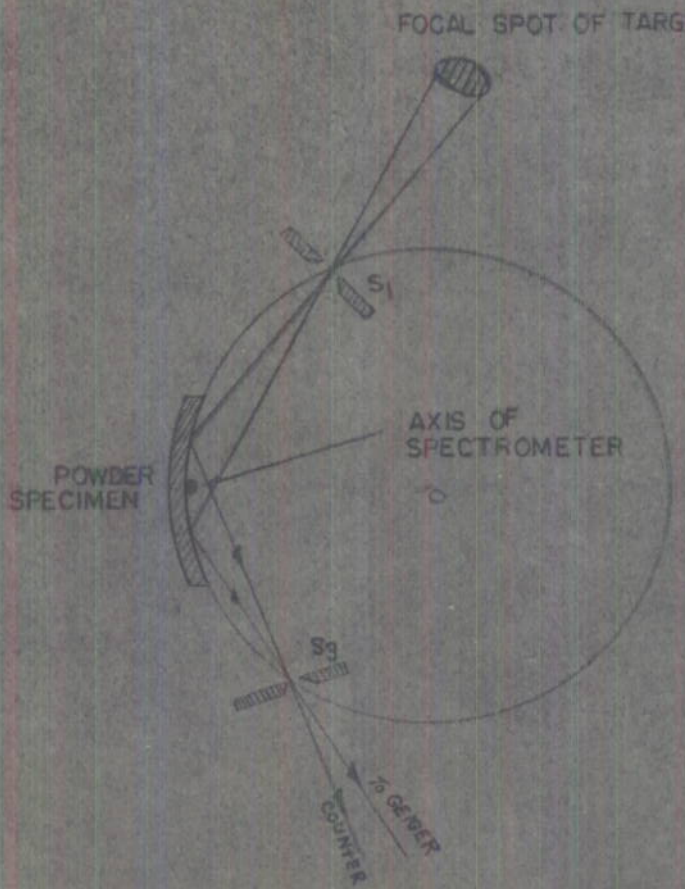


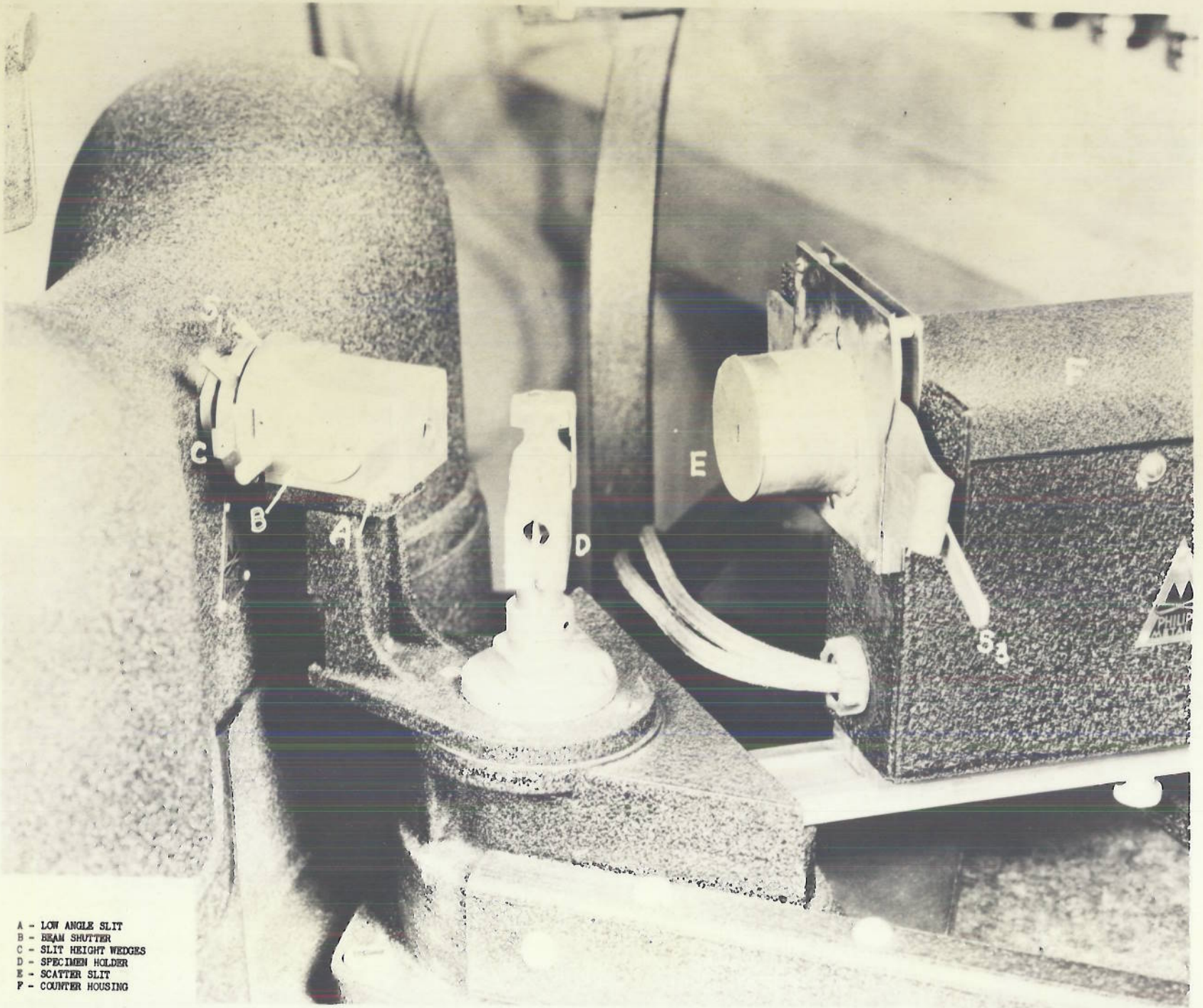
FIG. 2

FOCUSING ACTION OF GEIGER COUNTER SPECTROMETER

PLATE 6

A - IMPULSE COUNTER
B - 0-400 SCALE
C - MICROMETER DIAL
D - X-RAY TUBE HOUSING
E - FREQUENCY METER OUTPUT
F - SPECIMEN SLIDE

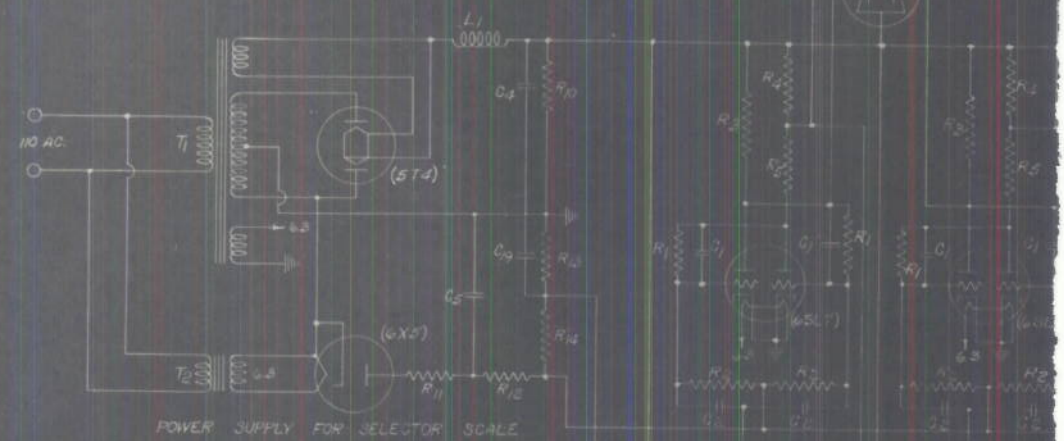
PLATE 7

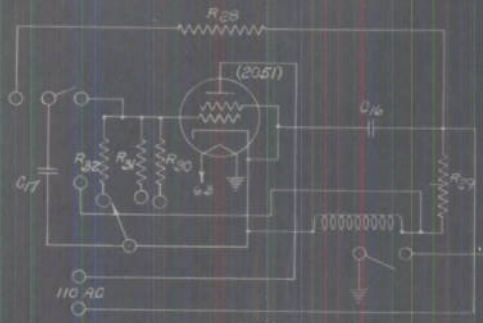


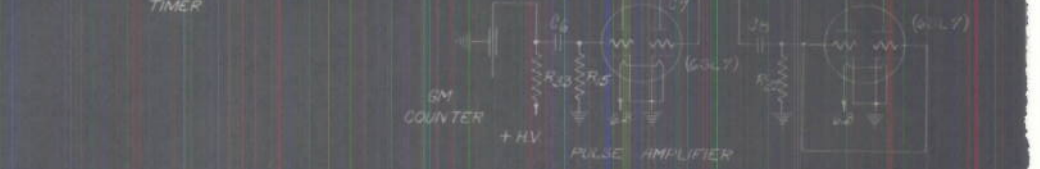
A - LOW ANGLE SLIT
B - BEAM SHUTTER
C - SLIT HEIGHT WEDGES
D - SPECIMEN HOLDER
E - SCATTER SLIT
F - COUNTER HOUSING

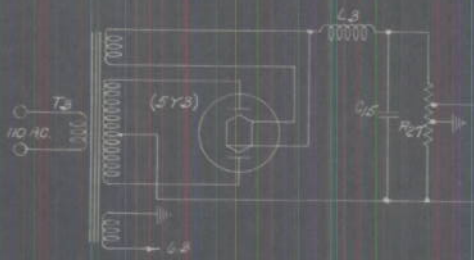
PLATE 8

N. R. L. 66

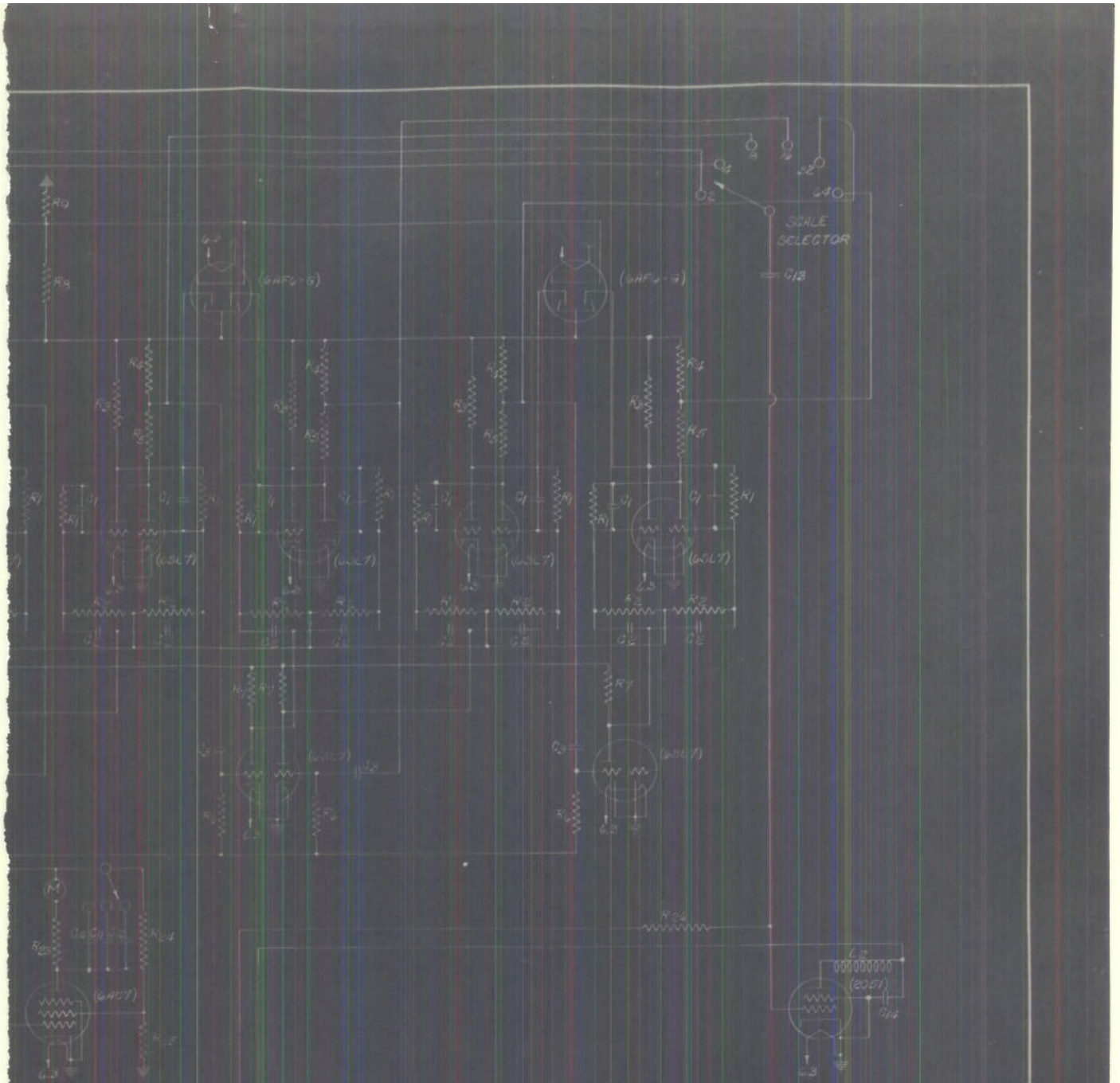

L1
110 AC.
T1
(5T4)
(6X5)
(6AF8-G)
(6SL7)
POWER SUPPLY FOR SELECTOR SCALE


(2051)
110 AC.
TIMER


GM COUNTER
+ H.V.
PULSE AMPLIFIER
(6SL7)
(6SL7)


L3
(5Y3)
110 AC.
POWER SUPPLY
FOR IMPULSE COUNTER CIRCUIT

MAR 1



ALTERATION TABLE

SYMBOLS AND THEIR EQUIV. TOLERANCES (UNLESS OTHERWISE NOTED)	
SYMBOL 1	±.0005
SYMBOL 2	±.0010
SYMBOL 2½	±.0030
SYMBOL 3	±.0050
SYMBOL 3½	±.0100
SYMBOL 4	±.0250
SYMBOL 5	

DELINEATOR	J.E. KING	IN CHARGE OF DIV. DRAFTING	CHIEF DRAFTSMAN
TRACER			
CHECKER			

APPROVAL	
PHYSICIST ENGINEER	SUPT. OF DIVISION
H. Friedman	
FOR DIRECTOR	
	COMDR. U.S.N.
BUREAU OF SHIPS	REFERENCE

U. S. NAVAL RESEARCH LABORATORY
"BELLEVUE," ANACOSTIA, D. C.

COUNTER CIRCUIT SCHEMATIC

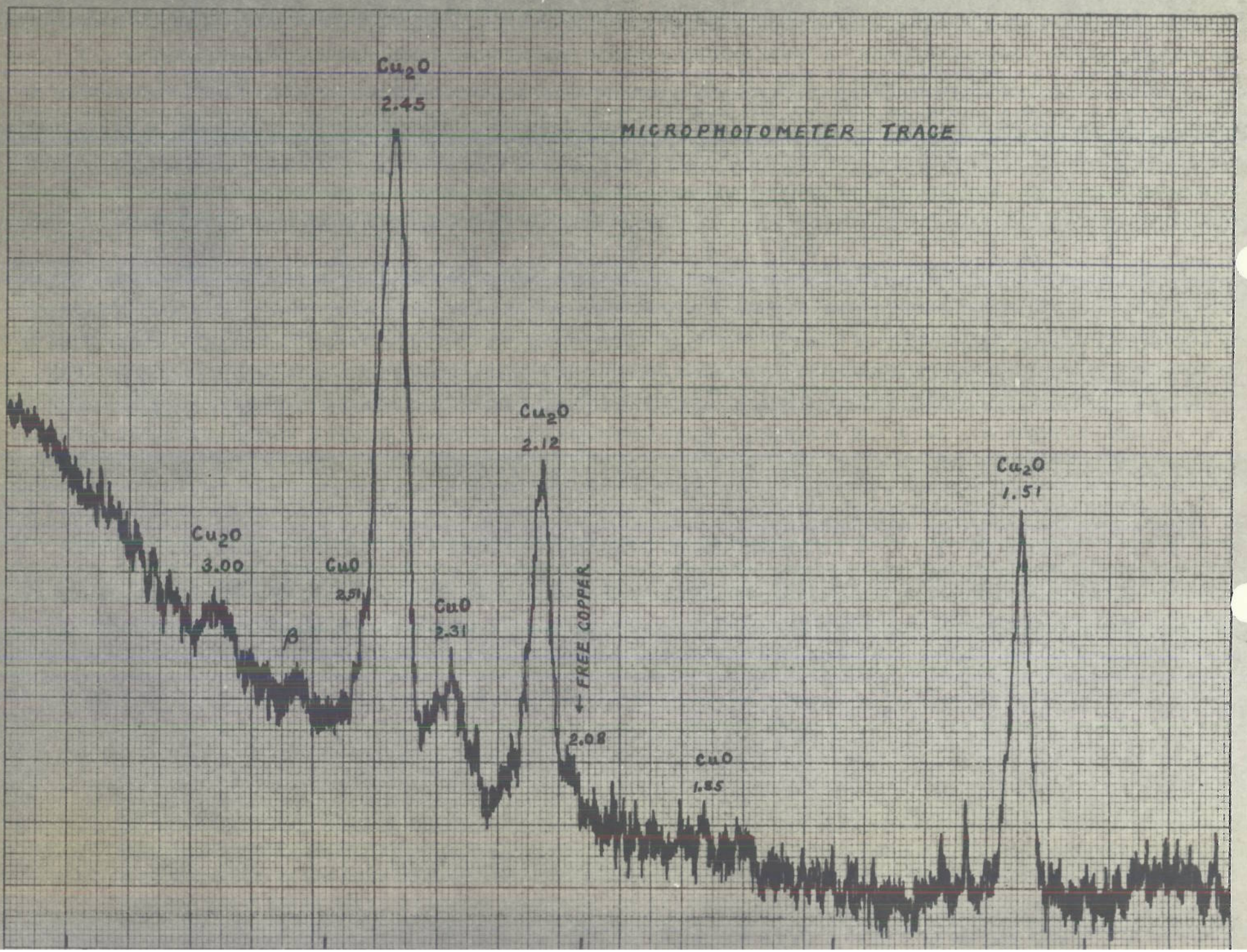
SCALE

DATE NOV. 11, 1943

F 653A

PLATE 9

1944


MICROPHOTOMETER TRACE
Cu2O
2.45
Cu2O
2.12
Cu2O
1.51
Cu2O
3.00
β
CuO
2.51
CuO
2.31
+ FREE COPPER
2.08
CuO
1.85

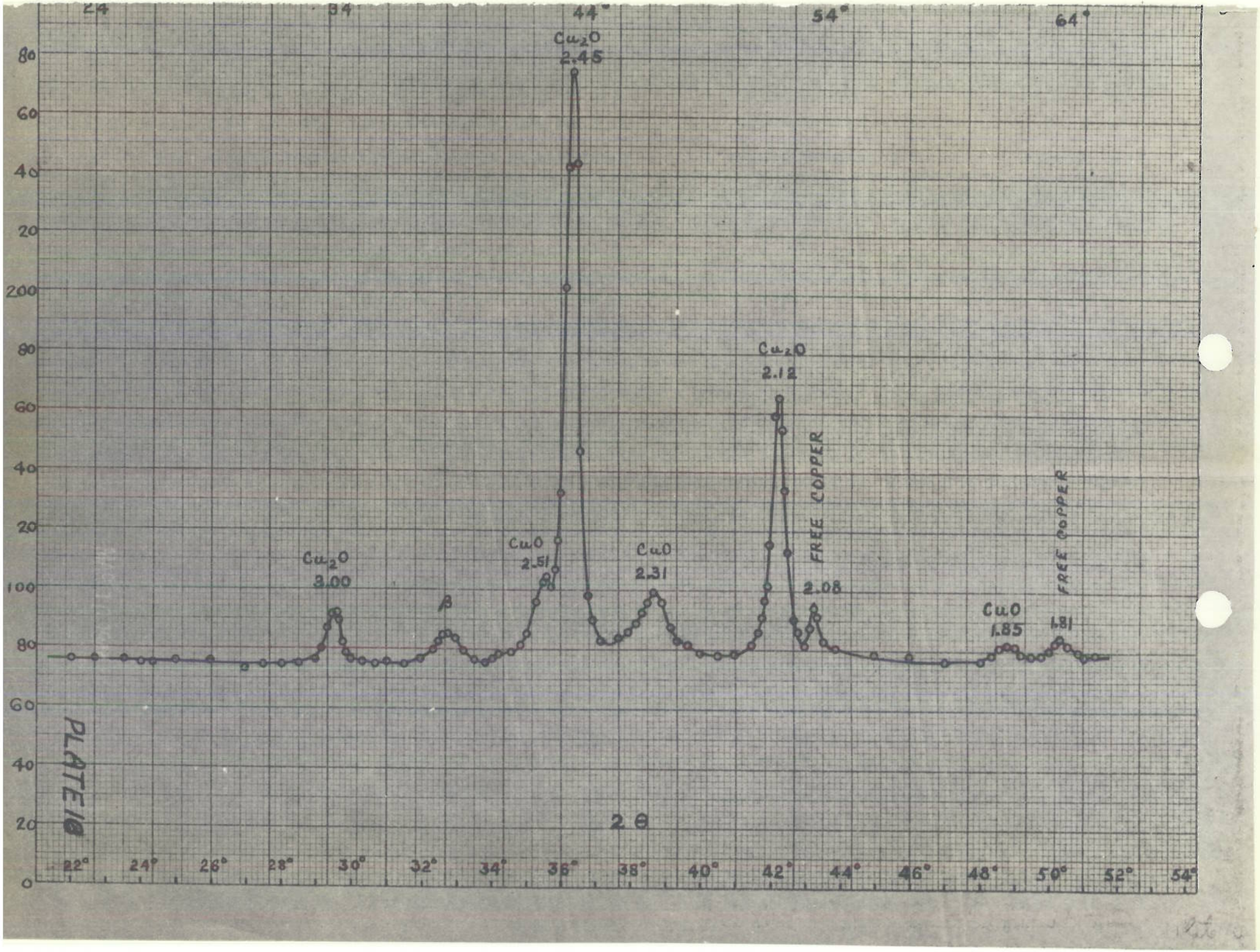
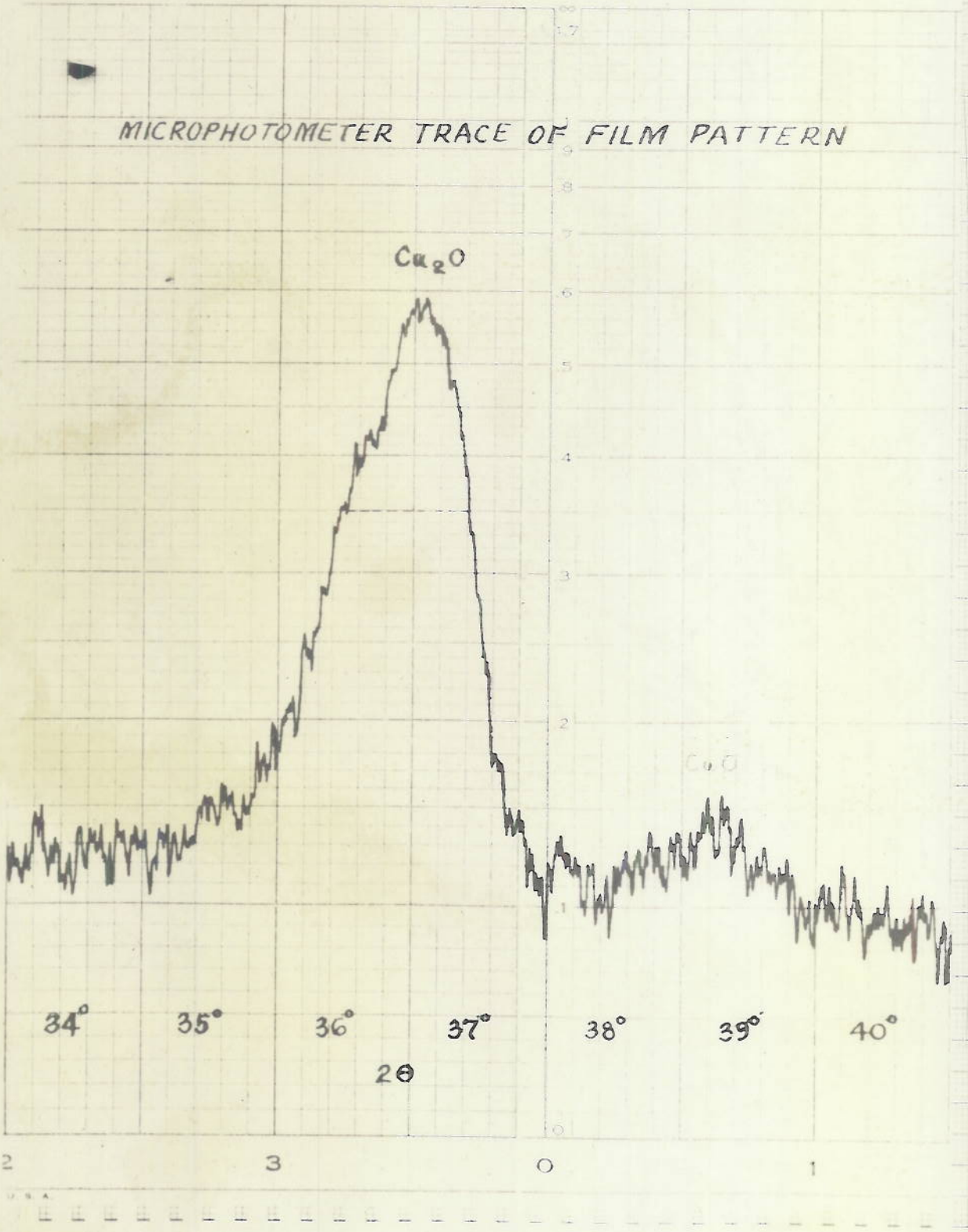


PLATE 10
2θ
Cu2O 3.00
Cu2O 2.45
Cu2O 2.12
CuO 2.51
CuO 2.31
CuO 1.85
FREE COPPER 2.08
FREE COPPER 1.81
A
22° 24° 26° 28° 30° 32° 34° 36° 38° 40° 42° 44° 46° 48° 50° 52° 54°

MICROPHOTOMETER TRACE OF FILM PATTERN



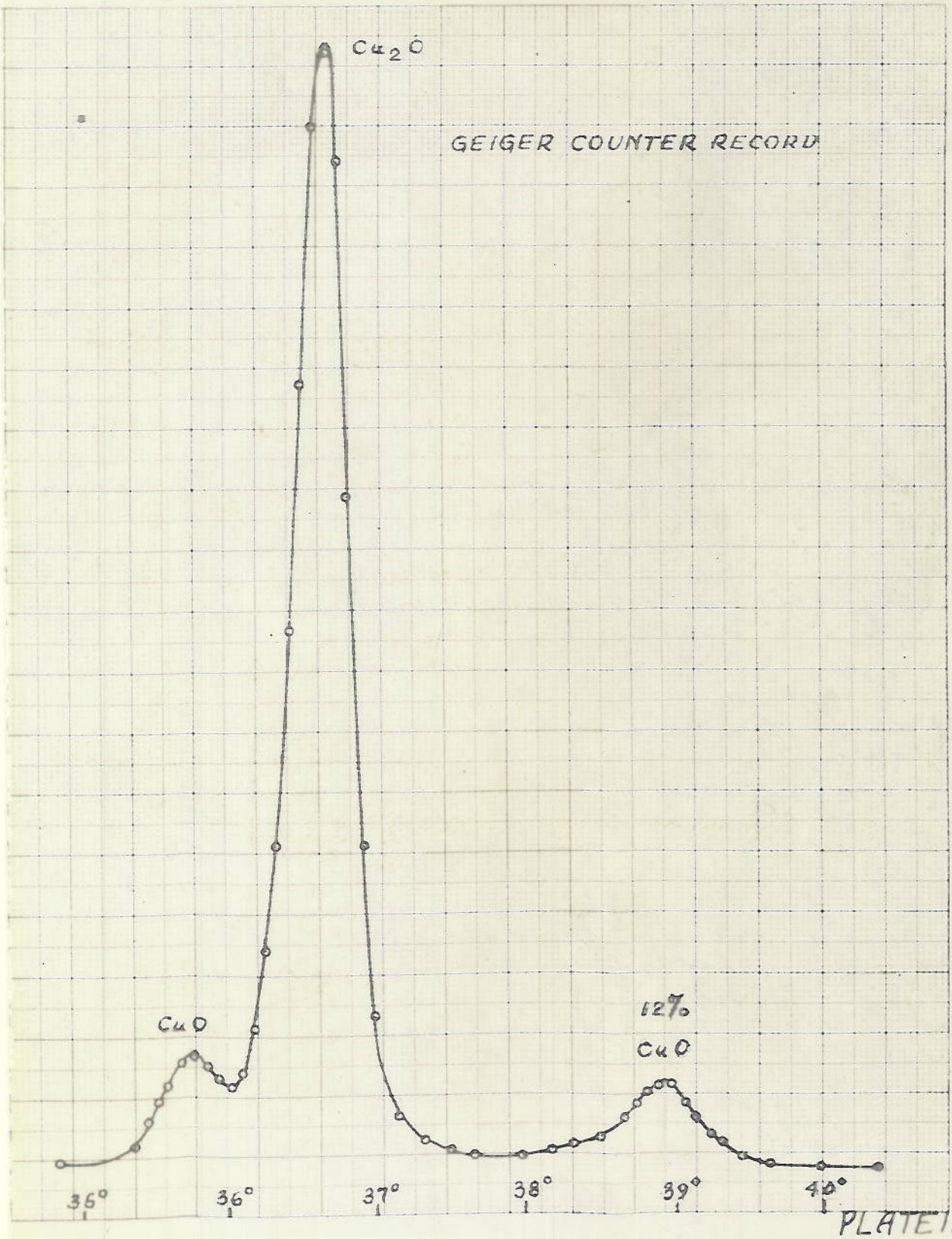
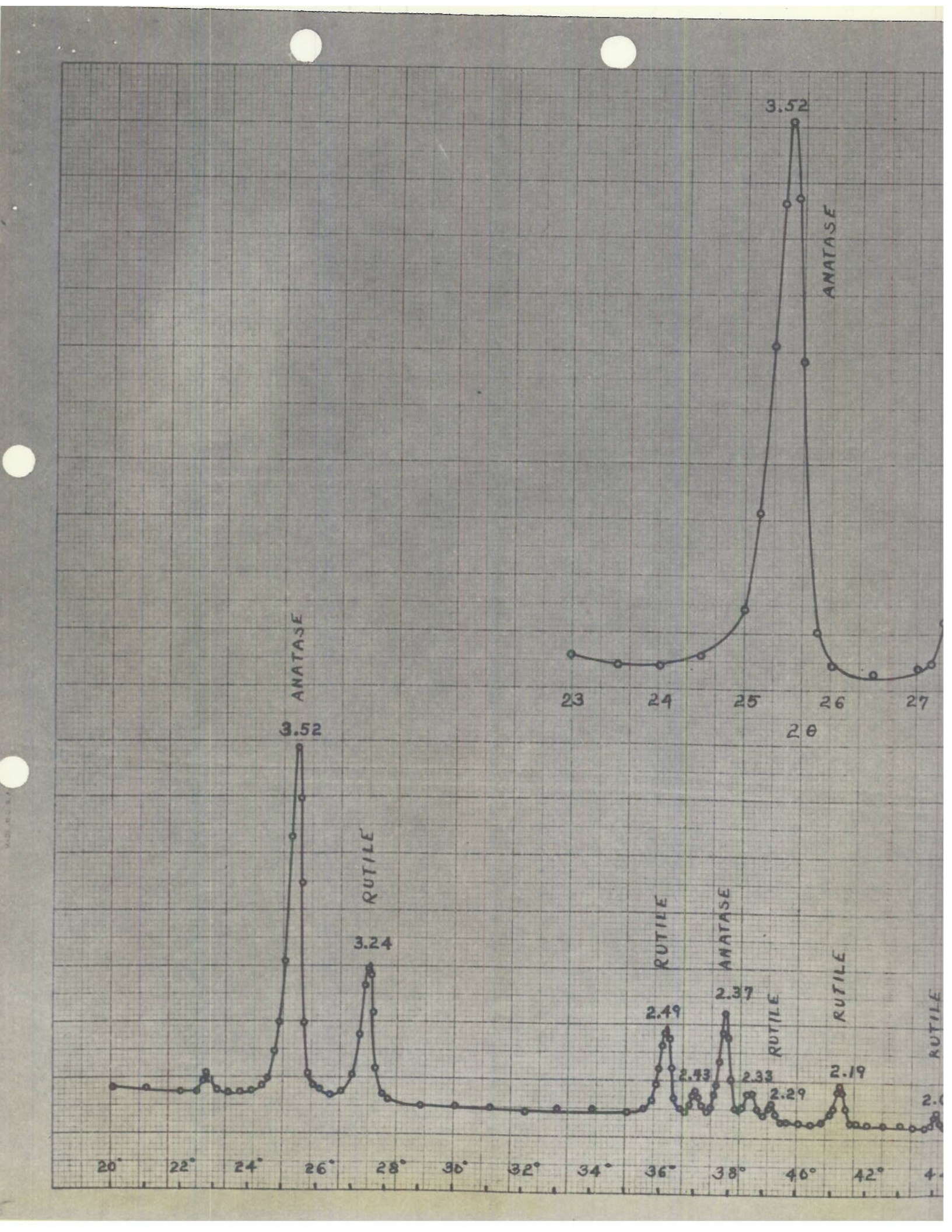


PLATE II


3.52
ANATASE
23
24
25
26
27
2θ
ANATASE
3.52
RUTILE
3.24
RUTILE
2.49
ANATASE
2.37
RUTILE
2.43
2.33
2.29
RUTILE
2.19
RUTILE
20°
22°
24°
26°
28°
30°
32°
34°
36°
38°
40°
42°

TITANIUM DIOXIDE

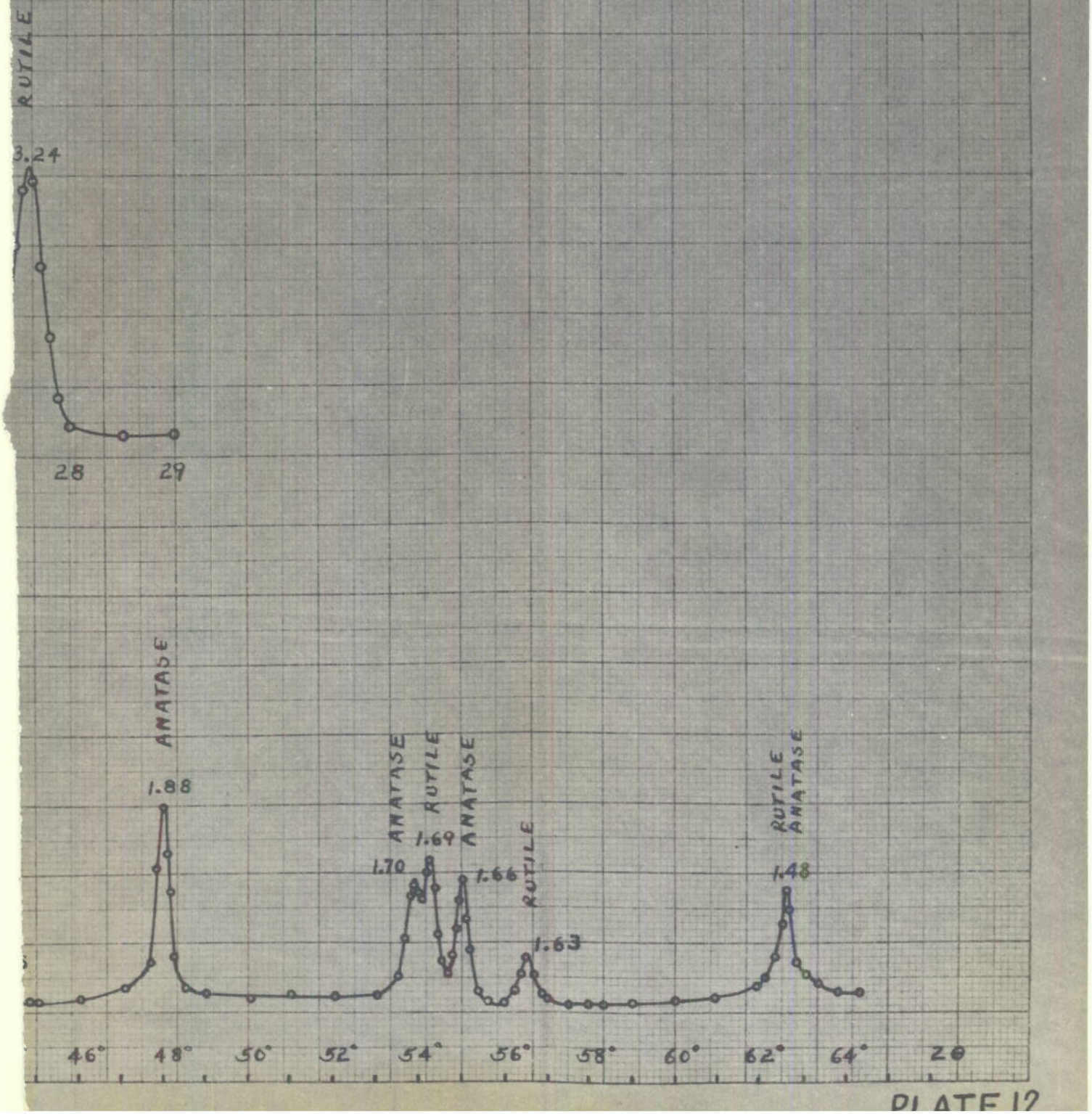
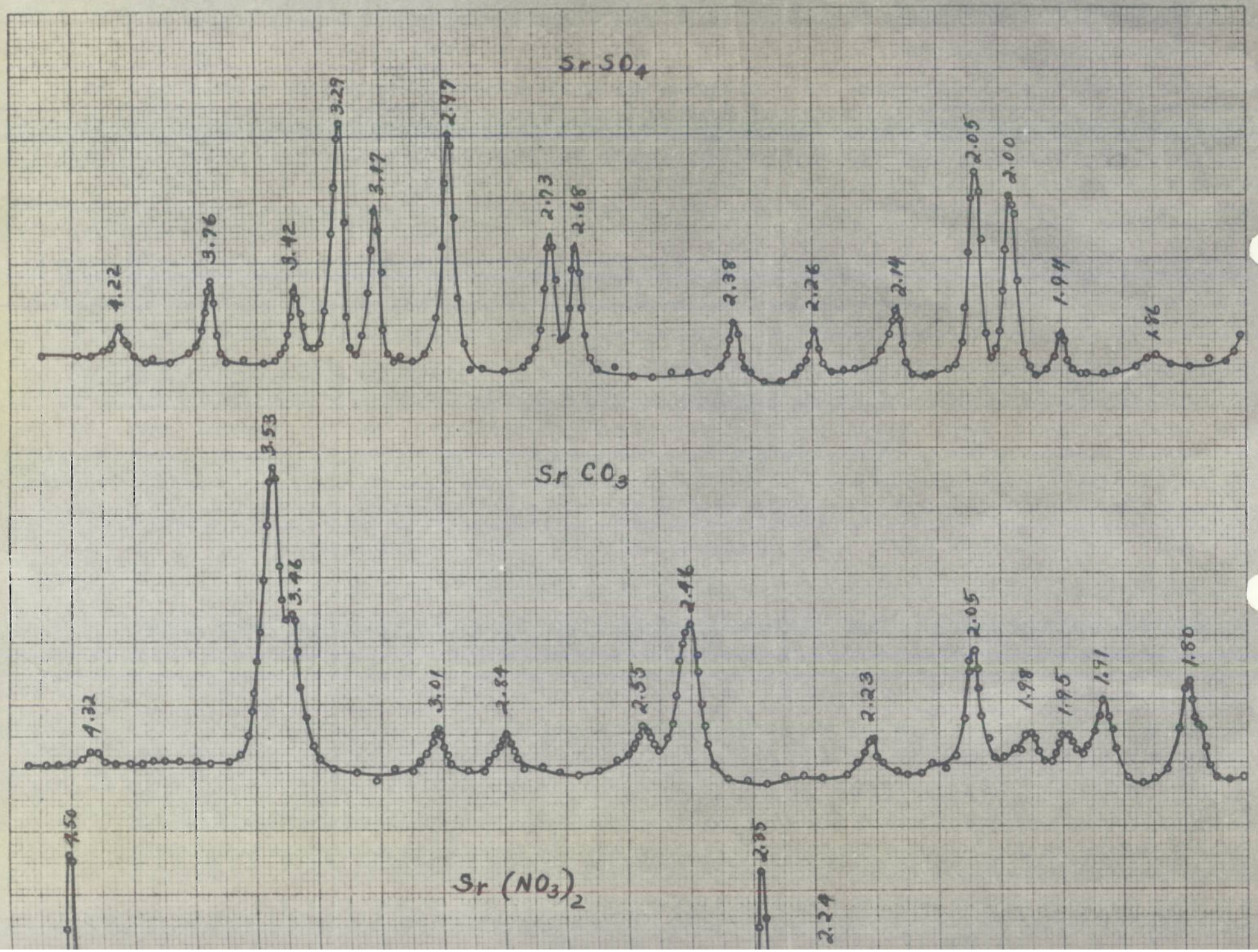


PLATE 12


Sr SO4
4.22
3.76
3.42
3.29
3.17
2.97
2.73
2.68
2.38
2.26
2.14
2.05
2.00
1.94
1.82
Sr CO3
3.53
3.46
4.32
3.01
2.84
2.53
2.46
2.23
2.05
1.98
1.95
1.91
1.80
Sr (NO3)2
4.50
2.85
2.29

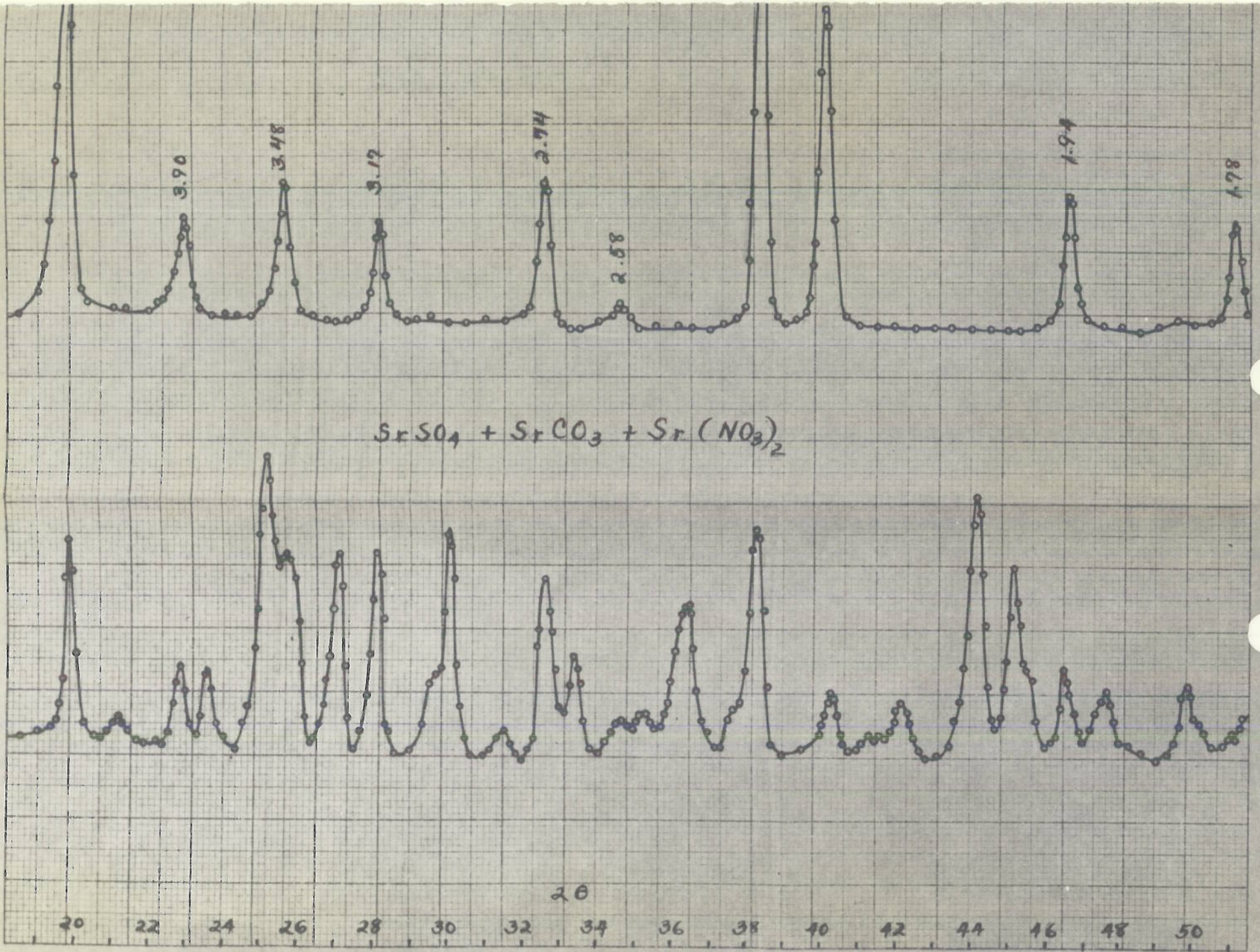

3.90
3.48
3.12
2.44
2.28
1.91
1.78
$SrSO_4 + SrCO_3 + Sr(NO_3)_2$
2θ
20
22
24
26
28
30
32
34
36
38
40
42
44
46
48
50

PLATE 13

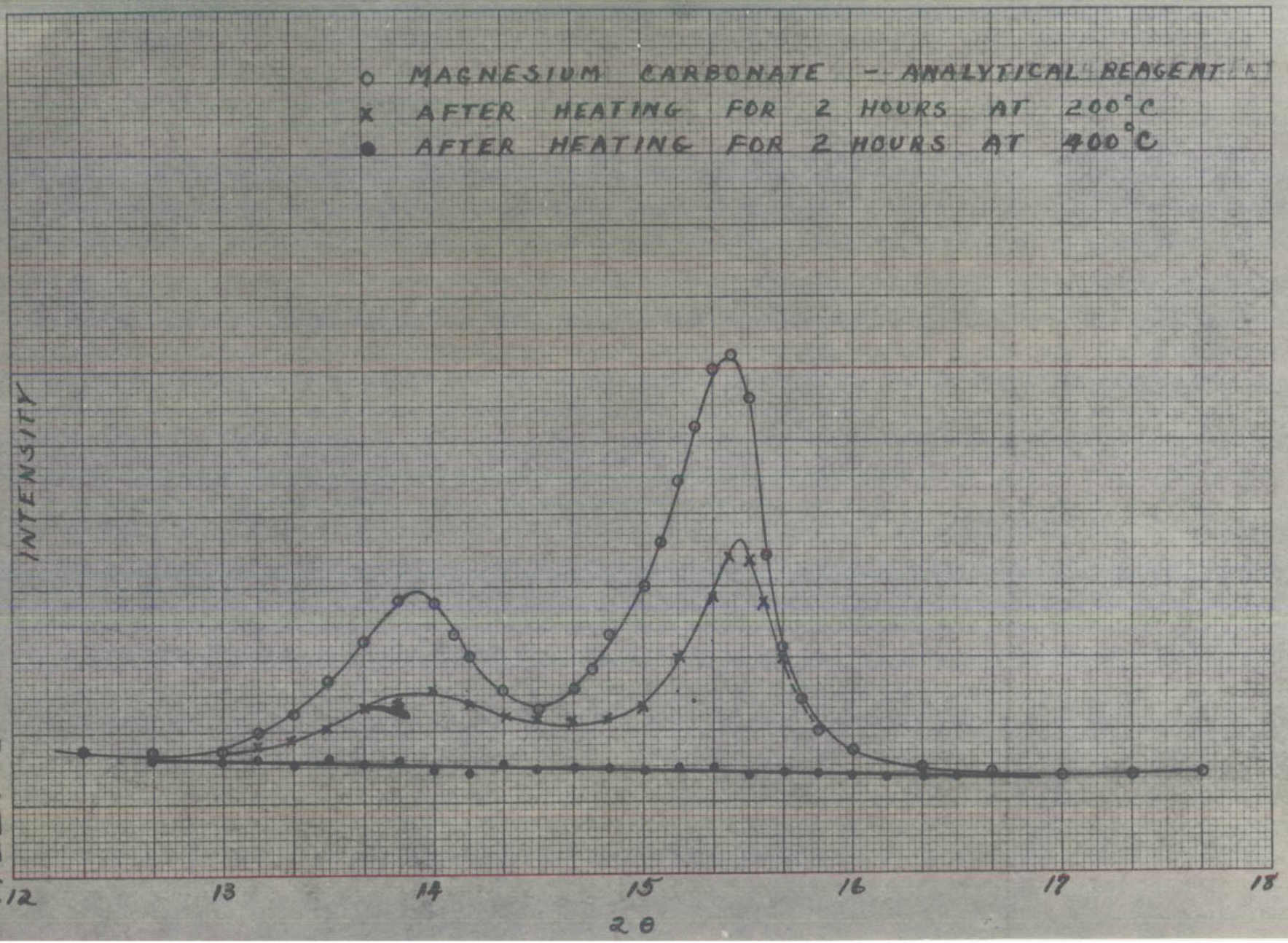
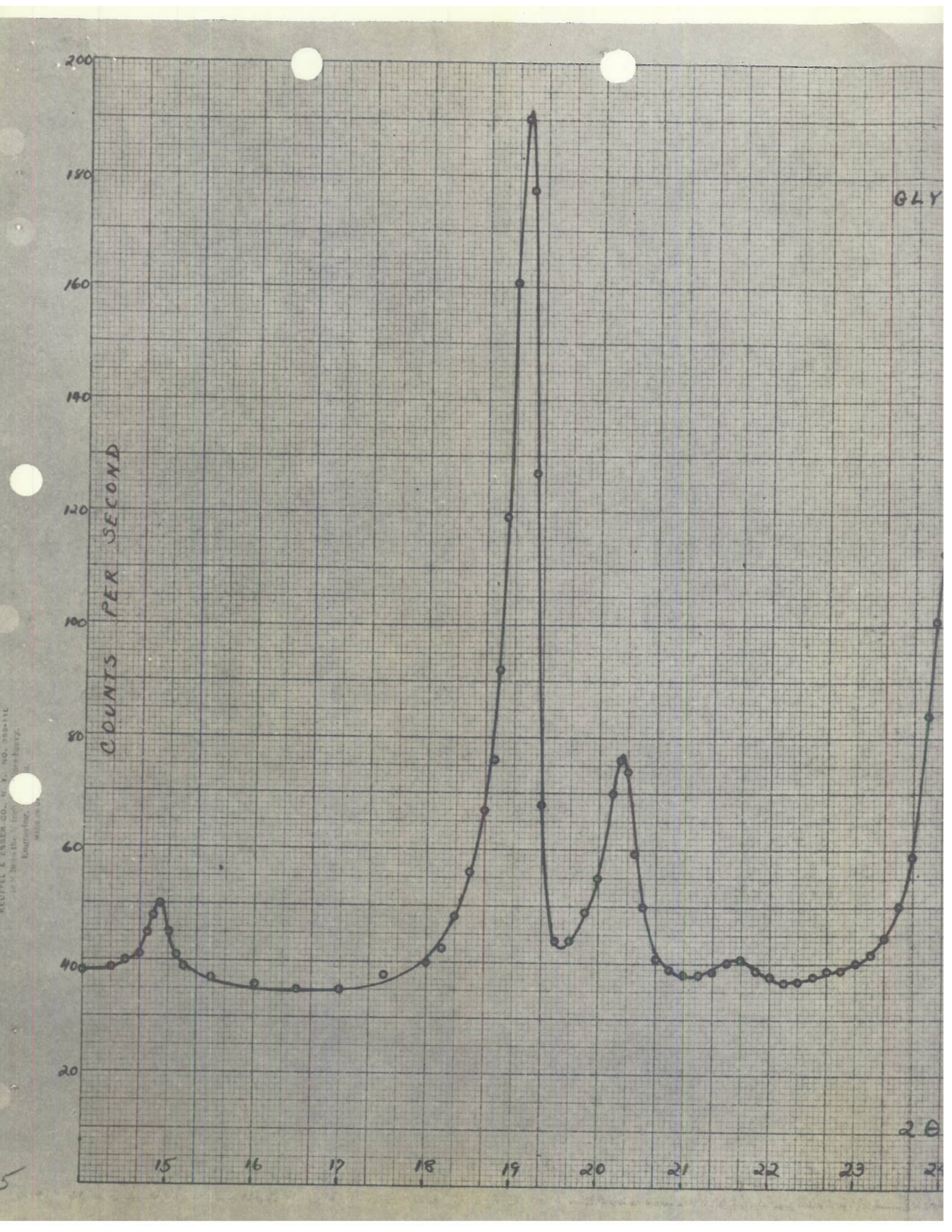

MAGNESIUM CARBONATE - ANALYTICAL REAGENT
AFTER HEATING FOR 2 HOURS AT 200°C
AFTER HEATING FOR 2 HOURS AT 400°C
INTENSITY
12
13
14
15
16
17
18
2θ

PLATE 14


200
180
160
140
120
100
80
60
40
20
COUNTS PER SECOND
GLY
15
16
17
18
19
20
21
22
23
2θ

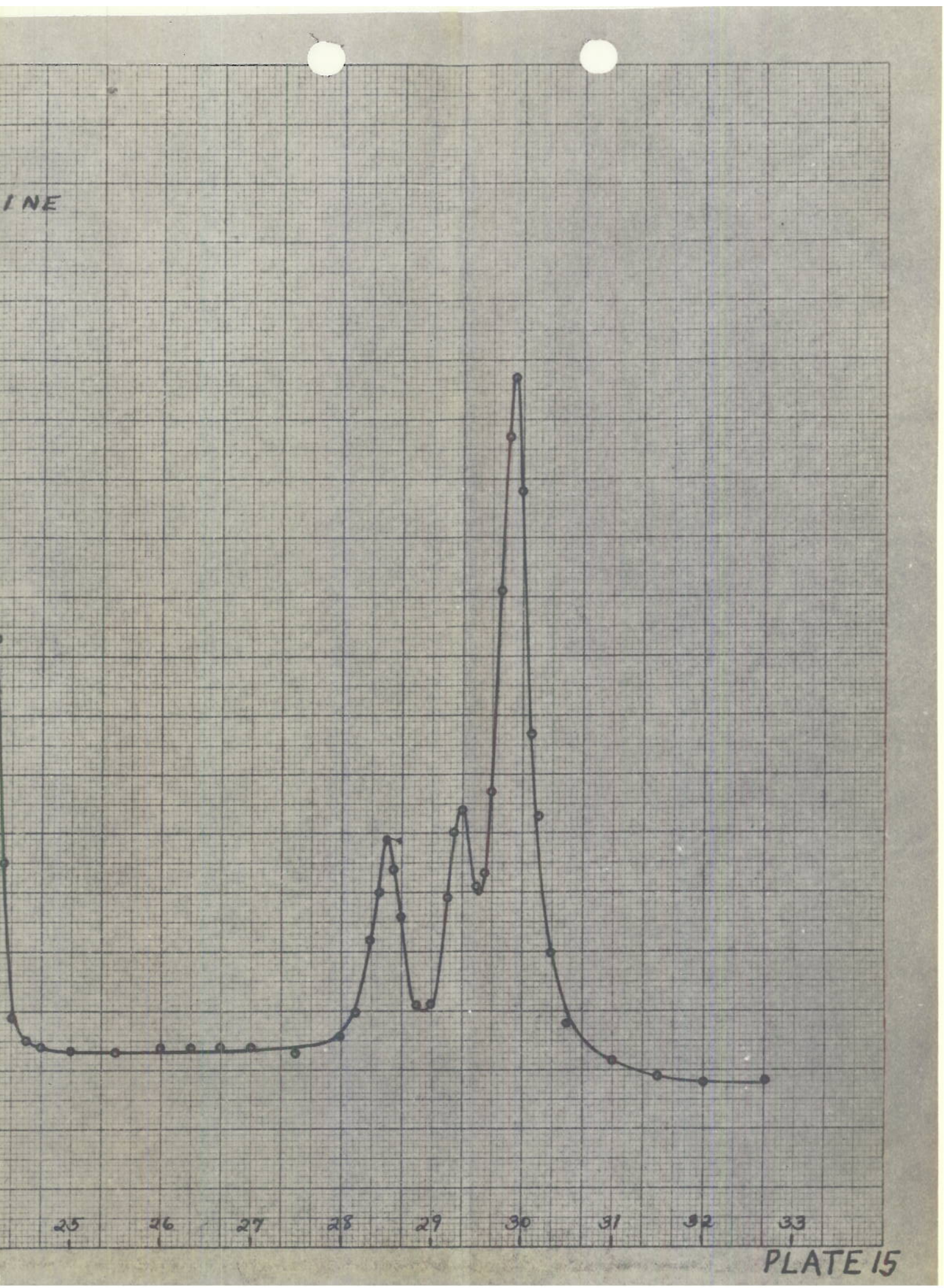
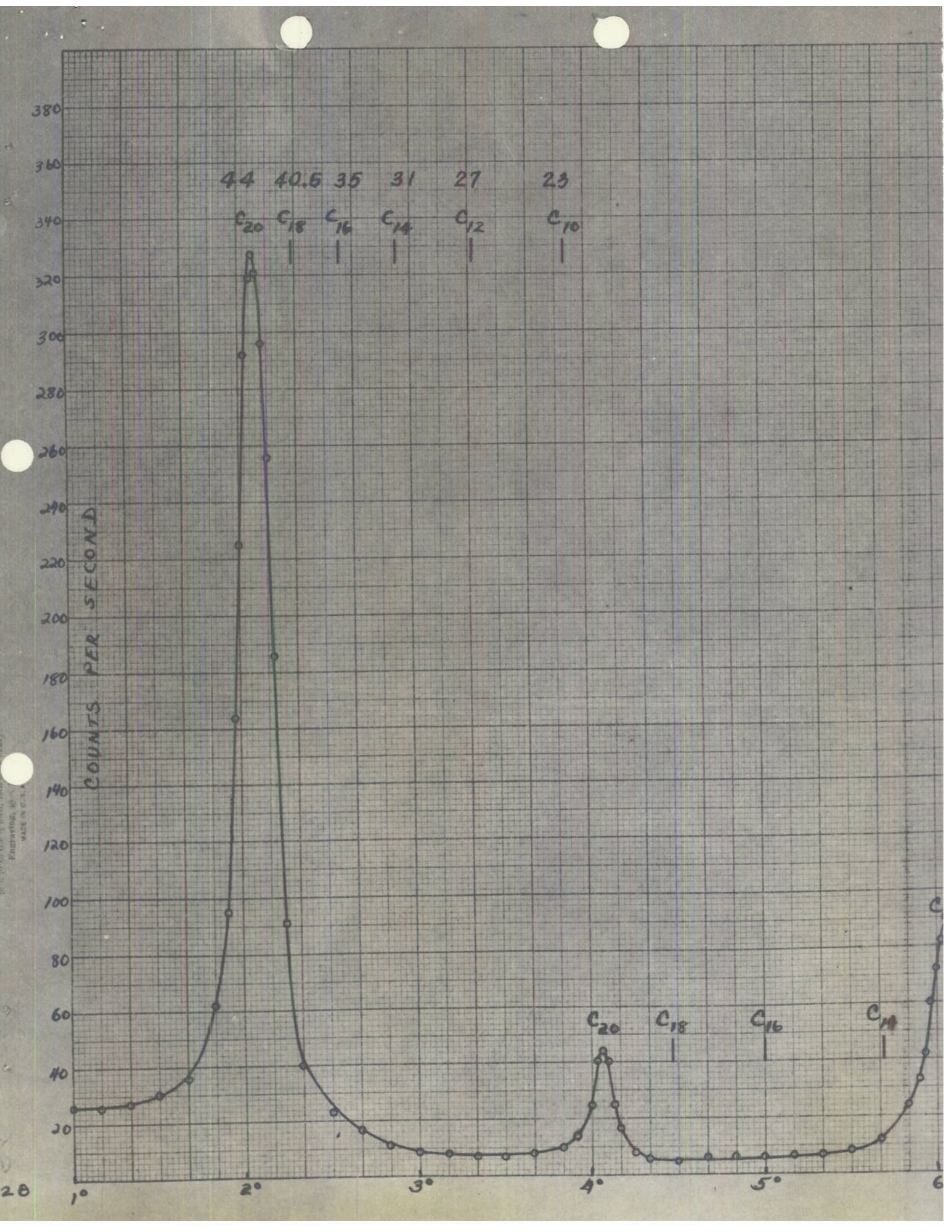


PLATE 15


COUNTS PER SECOND
380
360
340
320
300
280
260
240
220
200
180
160
140
120
100
80
60
40
20
44
40.5
35
31
27
23
C_{20}
C_{18}
C_{16}
C_{14}
C_{12}
C_{10}
C_{20}
C_{18}
C_{16}
C_{14}
1°
2°
3°
4°
5°

ALIPHATIC ACIDS

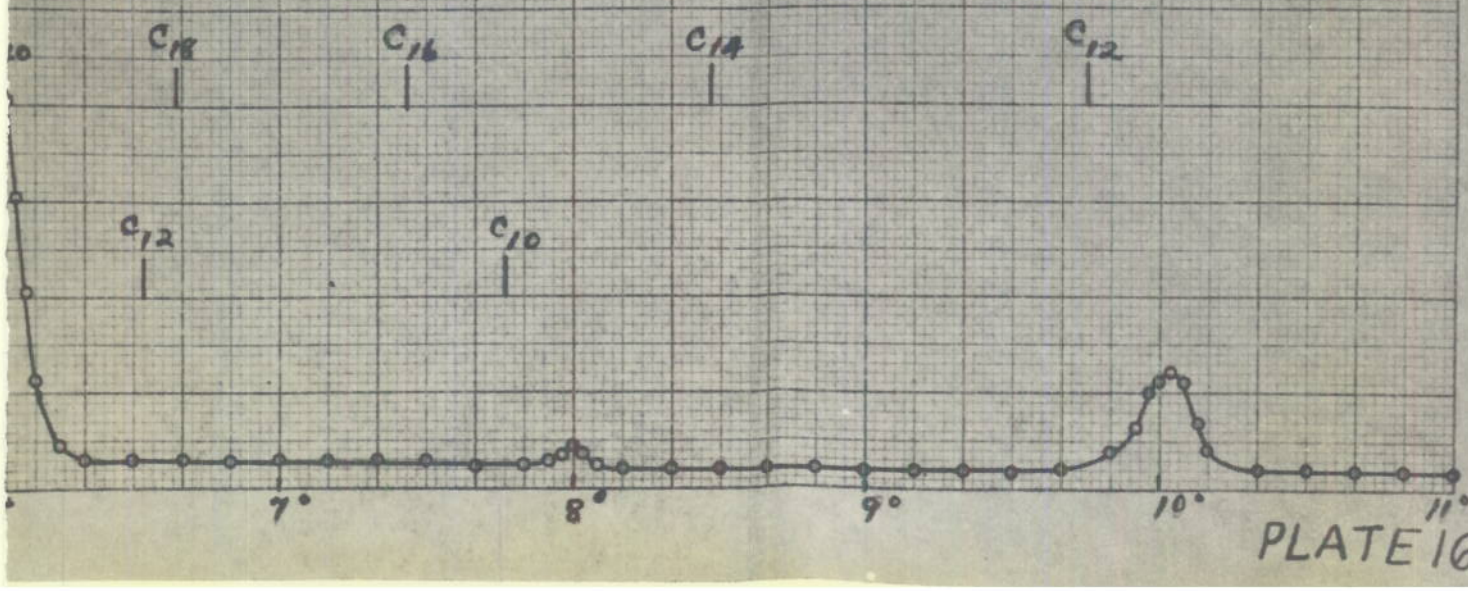
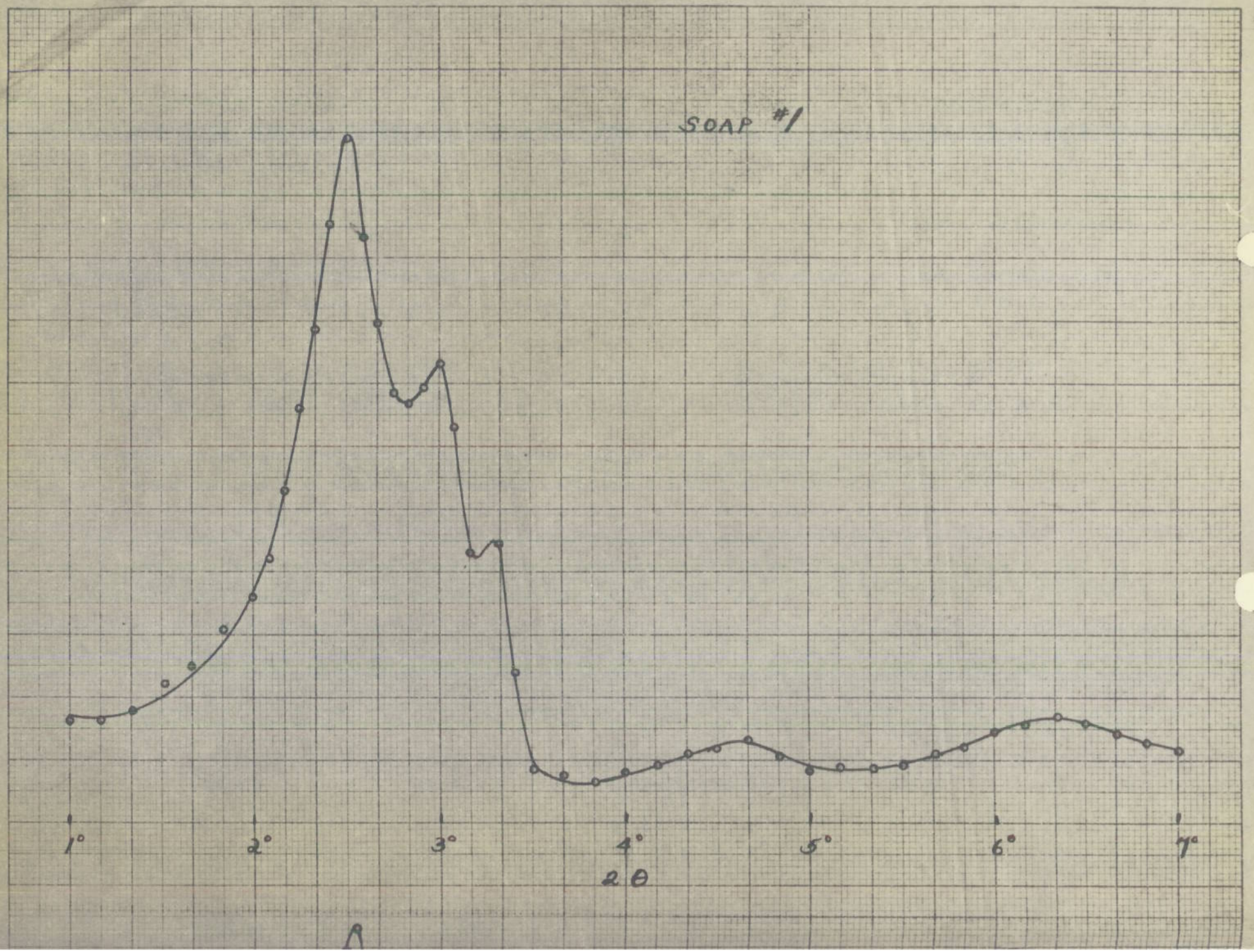


PLATE 16

PLATE 17


SOAP #1
1°
2°
3°
4°
5°
6°
7°
2θ

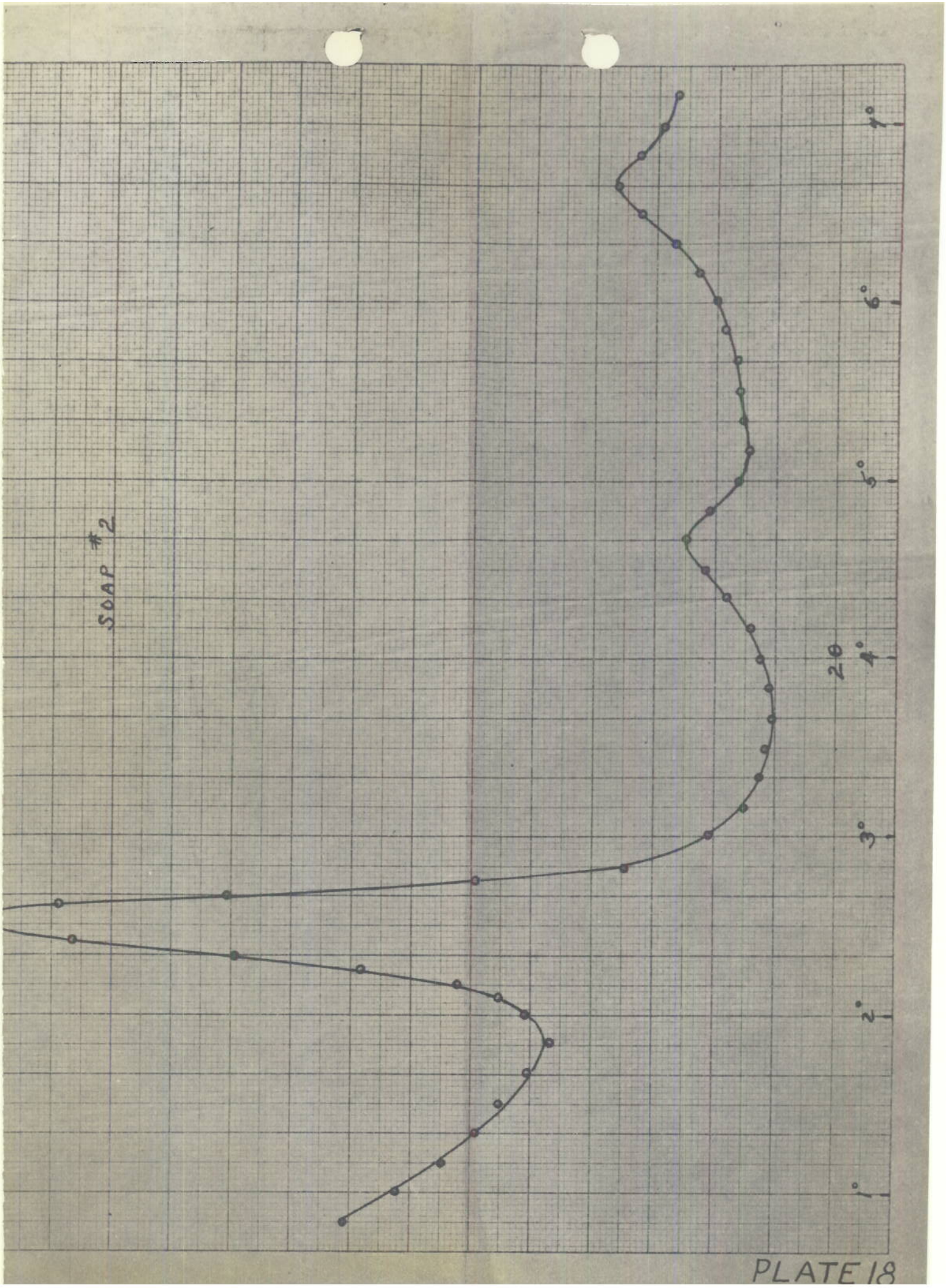


PLATE 18