

Diffraction Patterns
Of Some
Metallic Derivatives
Of
Anthranilic Acid

A thesis presented to the Graduate
School of The University of Houston in
partial fulfillment of the requirements
for the Degree of Master of Science.

M. D. ANDERSON MEMORIAL LIBRARY
UNIVERSITY OF HOUSTON

by

William C. Herrmann

April 15, 1951

64979

Table of Contents

Summary

Section I

Chelate rings ----- 1

Syntheses ----- 3

Section II

Introduction ----- 4

Production of x-rays ----- 6

Equipment ----- 12

Procedure ----- 15

Data ----- 16

Diagrams ----- 24

Photographs ----- 27

Conclusion and Errors ----- 28

Graph ----- 29

Summary

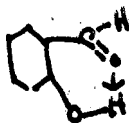
Metallic derivatives of anthranilic were synthesized. These derivatives are classified as chelate rings. X-ray diffraction patterns of these metallic derivatives were obtained. Investigation of these patterns revealed that each diffraction pattern was unique, but that the patterns were related by the atomic number (Z) of the incoming metal. These diffraction patterns provide a quick and positive means of identification. A plot of atomic number against intense characteristic lines enables the approximate prediction of strong characteristic lines of other metallic derivatives of anthranilic acid.

Section I

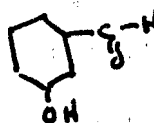
Introduction

The term chelate ring denotes a cyclic structure that arises from the intramolecular coordination in a system formed as a result of the presence of a donor and acceptor atom within the same molecule.

Chelation had been detected as a result of stereoisomerism, ionization, and absorption data. The last criterion came into prominence in the year 1935 with the study of the infra-red absorption of certain molecules containing hydroxyl groups. Hilbert, Wulf, Hendricks, and Liddel⁽¹⁾ were prominent in these absorption studies. They noted that an organic compound, aliphatic or aromatic, containing a free hydroxyl group showed a symmetrical absorption band at or near 2.7μ . It was observed that this characteristic band was absent in those molecules which, from other evidence, were presumed to undergo hydrogen bond formation. Later it was established that the characteristic hydroxyl band was replaced by a broad band extending from 2.9μ to 3.3μ , which was characteristic of the $\text{O-H}\cdots\text{O}$ linkage.



Absorption band $(2.9-3.3)\mu$

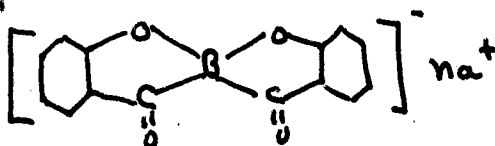


Maximum absorption 2.7μ

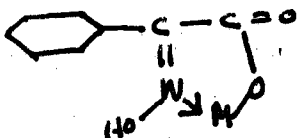
(2)

According to Sidgwick, chelate rings may be classified into three distinct classes.

Class A chelate rings result from a combination of ions with a central atom and arise from the inability of that atom to form additional covalent links except by coordination. These compounds are highly ionized in aqueous solutions and are generally very stable. An example of this structure is a boro-salicylate.



Class B chelate rings are systems in which the central atom forms one normal covalent link and one coordinate covalent link. These systems are non-ionized in aqueous solutions and are generally less stable than is the class A structure. Depending upon the central atom, these ring systems decompose at temperatures ranging from 150°C. to 230°C. An example of this structure is the oxime of phenylglyoxalic acid. (3)



Class C chelate rings are systems in which the central atom forms two coordinate covalent links. These systems are unstable. Their instability arises from the fact that stable ions or molecules are formed when the ring is broken. An example of this structure is Al_2Cl_6 .



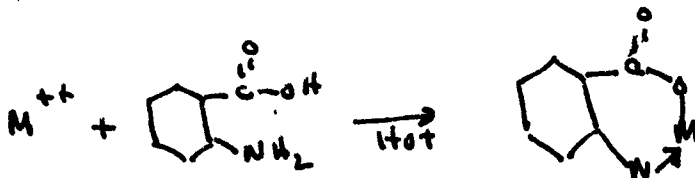
Syntheses

Manganese, nickel, copper, zinc, mercury, and lead derivatives of anthranilic acid were synthesized. These syntheses were performed as reported by the following authors.

- (4)
- Ni, Cu derivatives--- H. Funk and M. Ditt
- (5)
- Pb, Hg derivatives--- H. Funk and F. Römer
- (6)
- Mn, Zn derivatives--- H. Funk and F. Römer

A typical synthesis consisted of adding a 3% (by weight) anthranilic solution (3 grams of anthranilic acid dissolved in 123 c.c. of ethyl alcohol) to a hot, dilute water solution of the metallic salt. The resulting mixture was removed from the burner and allowed to stand one hour. The mixture was filtered through a Buchner funnel (with suction) and washed three times with the reagent and twice with ethyl alcohol. The precipitate was dried at a temperature of 100°C. for one hour.

The reaction proceeded as is indicated by the following equation.



The reaction proceeds readily and is used for the quantitative determinations of many metallic ions.

Section II

Introduction

The history of x-rays dates back to the year 1895. In this year, Wilhelm Conrad Roentgen was conducting experiments to determine the properties of cathode rays. In the course of his study, he noted a hitherto unknown type of radiation associated with cathode rays when the cathode rays were rapidly decelerated. He called these unknown radiations, x-rays.

In the year 1912, M. Laue, W. Friedrich, and P. Knipping noted that x-rays can be diffracted by crystals. This work was carried on by Bragg, who later propounded the relation between the wavelength of the incident radiation (λ), the angle between the crystal face and the incident radiation (θ), the order of reflection (n), and the distance between two successive planes in the crystal (d). This relation is known as Bragg's law and is given by the equation

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

With the application of this law, interatomic distances can be accurately calculated. (λ) varies directly as the $\sin \theta$; in order to obtain values of (θ) that are large enough for accurate measurement, (λ) should be relatively large. For this reason, in diffraction work, soft x-rays are employed. Soft x-rays denote x-rays whose wavelengths are situated between 10 angstrom units and 1 angstrom unit. The importance of intense, monochromatic (nearly) radiation is at once evident.

These intense, nearly monochromatic x-rays are obtained by appropriately filtering the general radiation so as to leave the characteristic x-ray beam. The radiation used in obtaining the diffraction patterns of these metallic derivatives of anthranilic acid was the characteristic K_{α} doublet. (2.81 Å)

Bragg obtained diffraction patterns from a single large crystal. Somewhat later, Debye and Scherrer⁽⁷⁾ developed the powder method of crystal analysis (the method used in this laboratory), whereby a finely divided sample of myriads of randomly oriented crystals was substituted for a single large crystal. In this method, a small, nearly parallel beam of monochromatic x-rays is allowed to pass through the sample. The x-rays will be reflected only from planes, of the minute crystals, that are properly oriented. Any crystal that is not properly oriented, will give rise to interference of x-rays reflected from the different layers. Thus, the reflected x-rays will constitute a family of right circular cones with their axes coincident with the primary x-ray beam and their apices at the sample.

(8)
A Debye-Scherrer type camera was used to record the diffraction patterns. This camera consists of a right circular cylindrical shell with an enclosed base and a demountable, light proof top. The narrow x-ray beam entered parallel to the base and was incident upon the sample which was placed coaxial with the cylindrical camera. Photographic film was mounted around the inner periphery of the camera. The inter-

section of the reflected radiations (family of right circular cones) and the right circular cylinder described a family of curves which were symmetrical about the intersection of the primary x-ray beam and the cylindrical camera.

The camera was built with an inner diameter of 114.6 mm. One mm. measured along the axis of the film corresponded to a 1 degree angular displacement. From geometric considerations, it is evident that the Bragg angle (Θ) is one half the angular displacement obtained from the film.

Production of x-rays

To propound adequately the theory of the production of x-rays is beyond the scope of this paper. The following paragraphs are offered only as a survey of some of the more pertinent facts.

When high speed electrons are rapidly decelerated, x-rays are produced. The frequency of these electro-magnetic radiations is determined by the structure of the atom (target) causing the deceleration and the potential difference through which the electrons are accelerated.

In diffraction work, characteristic x-rays are of major importance. Continuous x-rays, which are produced in conjunction with characteristic x-rays, interfere with the diffraction process and must be eliminated, as far as possible, by filters.

To initiate the discussion of the production of characteristic x-rays, it is helpful to examine the wave aspect of matter.

(9)

As early as 1828, Sir William Hamilton associated characteristic wave motion with every material particle. We assume a particle of mass (m) moving with velocity (v) from point A to point B. Associated with the particle is a characteristic wave with velocity (μ). (μ) is the phase velocity of the wave, i. e., the velocity with which a single wave is propagated. If the two paths from A to B are to be identical, (v) must be proportional to ($\frac{1}{\mu}$).

$$(2) \quad \mu = \frac{c}{mv}$$

(C) must be independent of distance, i. e., independent of coordinates.

If the particle is to correspond to a group of waves, it is not the velocity of propagation of the individual waves, but that of the boundaries of the wave groups which should be equal to the velocity of the particle. Treatises on wave motion show that the group velocity (w) is expressed by the equation

$$(3) \quad \frac{1}{w} = \frac{d}{dv} \left(\frac{v}{\mu} \right)$$

where (v) is the frequency of the wave.

To differentiate (3), (μ) must be expressed as a function of (v). DeBroglie accomplished this by placing the arbitrary constant (C) equal to the total energy (W) of the moving particle and assuming that

$$(4) \quad C = W = h\nu$$

in accordance with the quantum theory. Upon performing the differentiation, De Broglie found that the speed of the wave group (μ) is identical with the speed of the corresponding particle.

The wave length of the wave associated with the particle is $\lambda = \frac{h}{p}$. Solving (3) and (1) for (μ) we obtain, $\mu = \frac{h\nu}{mv}$.

Therefore:

$$(5) \quad \lambda = \frac{h}{mv}$$

Equation (5) describes the wavelength of a de Broglie wave. The wave train corresponding to the moving particle has a wave length inversely proportional to the momentum of the particle.

The wave mechanics of de Broglie showed that large scale mechanics might be considered either the propagation of wave groups or the motion of particles. Schrödinger extended wave mechanics to cover problems without restriction as to dimensions.

The following differential equation can be used to represent a wave corresponding to the motion of a particle in a field of force,

$$(6) \quad \text{or} \quad \frac{\partial^2 \psi}{\partial x^2} + \frac{\partial^2 \psi}{\partial y^2} + \frac{\partial^2 \psi}{\partial z^2} - \frac{1}{\mu^2} \frac{\partial^2 \psi}{\partial t^2} = 0$$

$$\nabla^2 \psi - \frac{1}{\mu^2} \frac{\partial^2 \psi}{\partial t^2} = 0$$

where (ψ) is any well behaved function. Assume the wave associated with the particle to have a velocity (V). The wave length will be given by equation (5).

$$\lambda = \frac{h}{mv}$$

The frequency of the wave is $\nu = \frac{u}{\lambda} = \frac{mv}{h}$.

If (ψ) is a wave of frequency (ν) , i.e., $\psi = A \sin(2\pi\nu t + \phi)$,

$$\frac{\partial^2 \psi}{\partial t^2} = -4\pi^2 \nu^2 \psi$$

whence

$$\frac{1}{u^2} \frac{\partial^2 \psi}{\partial t^2} = -\frac{4\pi^2 m^2 \nu^2}{h^2} \psi.$$

De Broglie has shown that $\nu = \frac{1}{h}(W - V)$, where (W) is the total energy of the particle and (V) is the potential energy of the particle. Thus:

$$\frac{1}{u^2} \frac{\partial^2 \psi}{\partial t^2} = -\frac{8\pi^2 m}{h^2} (W - V) \psi.$$

Equation (6) becomes

$$(7) \quad \nabla^2 \psi + \frac{8\pi^2 m}{h^2} (W - V) \psi = 0$$

which is the foundation of Schrödinger's wave mechanics.

Schrödinger's wave mechanics may be applied to the stationary states of a simple atom. Consider a simple atom of massive positive nucleus of charge $+Ze$ about which exists an electron of charge $-e$. Clearly, the potential energy of the electron is $-\frac{Ze^2}{r}$. Inserting this value of (V) in equation (7), we obtain

$$(8) \quad \nabla^2 \psi + \frac{8\pi^2 m}{h^2} \left(W + \frac{Ze^2}{r} \right) \psi = 0$$

Schrödinger has shown solutions exist for this differential equation only if

$$(9) \quad W_n = \text{Const} - \frac{2\pi^2 Z^2 m e^2}{h^2 n^2} \quad [n=1, 2, 3, \dots]$$

Equation (9) represents the possible energy levels (shells) in an atom.

When n	1	shell is noted as the K shell
" "	2	" " " " " L "
" "	3	" " " " " M "
" "	4	" " " " " N "

etc.

Corresponding to any particular value (W_k) of (W_n) , the solution of (8) becomes

$$(10) \quad \psi_k = C_k \psi_k e^{2\pi i(\nu_k t + \theta_k)}$$

where (C_k) and (θ_k) are arbitrary constants and (ν_k) is defined by the relation $h\nu_k = W_k$. The quantity (ψ) oscillates with a frequency (ν_k) .

The most general solution of (8) for a system is the sum of all the solutions of the type (10).

$$(11) \quad \psi = \sum_{k=1}^{\infty} C_k \psi_k e^{2\pi i(\nu_k t + \theta_k)}$$

We assume that the density of electric charge, at any point, is proportional to the value ... at this point of $\psi \bar{\psi}$, where $(\bar{\psi})$ is the complex conjugate of (ψ) ; we assume

$$(12) \quad \rho = -e \psi \bar{\psi} \\ = -e \sum_{k_1} \sum_{k_2} C_{k_1} C_{k_2} \psi_{k_1} \bar{\psi}_{k_2} e^{2\pi i[(\nu_{k_1} - \nu_{k_2})t + \theta_{k_1} - \theta_{k_2}]}$$

where (e) is the electronic unit charge. It is clear that (ρ) depends upon the time only through the exponential factor of which the real part is $2\pi[(\nu_{k_1} - \nu_{k_2})t + \theta_{k_1} - \theta_{k_2}]$. Equation (12) represents a fluctuation of charge densities at any point with frequencies,

$$(13) \quad \nu_{k_1} \nu_{k_2} = \nu_{k_1} - \nu_{k_2} = \frac{1}{h} (W_{k_1} - W_{k_2})$$

From the preceding discussion it is noted that permissible changes from one energy state to another energy state occur with the emission or absorption of radiation of a definite frequency. These frequencies are characteristics of each element.

The preceding discussion has evolved about a hydrogen like atom. Moseley's law is the experimental evidence of the quantum theory. Moseley was able to use an x-ray spec-

trometer to study the relation between the frequency of characteristic radiation (ν) and the atomic number of the target (Z). He found that

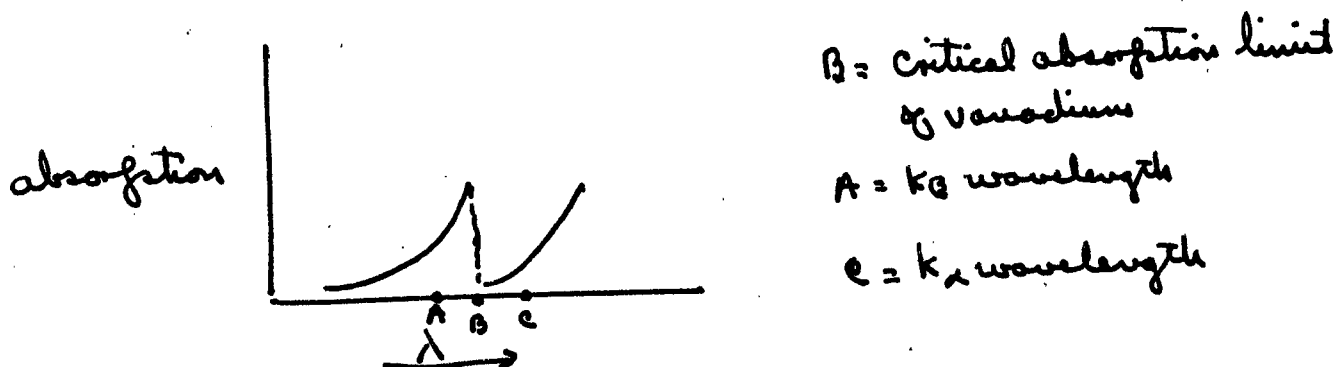
$$(14) \quad \nu^{1/2} = k(Z - \sigma)$$

where k and σ are constants for a certain set of energy level displacements.

A certain minimum of energy will be necessary to knock an electron from a specific energy level. If the filament electron is to supply this energy, it necessarily must be accelerated through a definite potential difference. In order to knock a K shell electron out of the atom, the filament electron will have to be accelerated through a certain potential difference; this potential difference is called the excitation potential for the K series. When the K shell electron is displaced, its initial energy state may be filled by a L, M, shell electron. If the L shell electron makes the transition, the radiated photon is noted as the K_{α} characteristic radiation. If the M shell electron makes the transition, the radiated photon is noted as the K_{β} characteristic radiation. L series characteristic radiations are denoted in a similar manner.

To obtain nearly monochromatic x-rays, critical absorption data were employed. In this diffraction problem, the characteristic K_{α} doublet of chromium was employed. The characteristic chromium K_{β} line is of high intensity and has a wave length 0.80 angstrom units shorter than the K_{α} line. ($k_{\alpha} = 2.28 \text{ \AA}^{\circ}$) To reduce the K_{β} line to a negligible
 $k_{\beta} = 1.48 \text{ \AA}^{\circ}$

value, a vanadium pentoxide filter was used. The K_α line is readily absorbed by the vanadium because its wave length falls just inside the critical absorption limit of vanadium.



The filter practically eliminates the K_α line and decreases the intensity of the higher frequency radiations of the continuous spectrum.

The mass absorption coefficient (μ_m) of vanadium near its critical absorption edge is $445 \frac{\text{cm}^2}{\text{gm}}$ ⁽¹²⁾. The thickness of vanadium required for the standard 9/10 absorption of the K_α line is given by the absorption equation.

$$(15) \quad \log I = -\mu x + \log I_0 \quad \left| \begin{array}{l} \mu = \text{linear absorption coefficient} \\ x = \text{distance} \end{array} \right.$$

The grams of vanadium per unit area necessary to affect 9/10 absorption may be obtained from the relation

$$(16) \quad m = \frac{A}{\mu_m} \log \frac{I_0}{I}$$

where (A) is unit area.

Equipment

The high voltage supply was adapted from a Westinghouse #67438 transformer. It provided full wave rectification and a peak voltage of 50 K.V. A circuit diagram of the transformer is given on page #24.

The control unit was adapted from a Westinghouse Dynex B control unit. The circuit diagram given on page #15 is self explanatory. It should be noted that every control the operator touches is grounded. To improve the clarity of the circuit diagram, the ground leads are omitted.

The diffraction tube (x-ray) was designed in this laboratory. It is a hot cathode, continuously pumped, demountable x-ray tube.

The original design of the diffraction tube was altered many times before a functional tube was developed. The tube, as it stands today, will operate over long periods of time with a current of 9 milli-amps at 30 K.V.

The metal part of the tube was constructed from a brass tube 2 inches in diameter and 4 inches long. It consists of a water cooled target and a beryllium window⁽¹³⁾ mounted in the wall of the tube. The target is essentially a thin brass plate, (1 mm. in thickness) plated with 20/1000 in. of chromium and placed perpendicular to the axis of the tube. The beryllium window is a circular piece of that metal, 10 mm. in diameter and 1.4 mm. thick, mounted in the wall of the tube adjacent to the chromium target. On page #16 is a drawing of the tube.

The glass part of the diffraction tube is a tube 25 cm. long in which two electrodes are mounted. To evacuate the tube there is a male connection situated at the bottom, which leads to a two stage diffusion pump and a fore pump.

At the outset, the filament (a tungsten spiral 15 mm. long) was placed 1 cm. from the target. The filament was enclosed

in a cylindrical aluminum shield; an aperture, the dimensions of the filament, was cut out of the top portion of the shield. Upon running the tube for a short time, a deposition of metal was noted on the glass walls of the tube. A subsequent analysis of the deposited metal yielded 90% aluminum and 10% tungsten. The vapor pressure of aluminum, at high temperatures and low pressures, was too great to render aluminum a functional metal for use as a shield. This condition was remedied by replacing the aluminum shield with a nickel shield.

The problem of the deposition of tungsten remained. The trouble was in the proximity of the filament to the target. The vapor pressure of tungsten at 2500°C. is approximately 10^{-7} microns. Tungsten, at the same temperature, in a intense electric field (as in an x-ray tube) has a vapor pressure much greater than the expected value. ⁽¹⁴⁾ The nearness of the tungsten filament to the target is the controlling factor. A tungsten filament placed within 1 cm. of the target has a vapor pressure 10^5 (approximately) times the expected value. Placing the filament at greater distances from the target decreases the vapor pressure of the tungsten. The reason for this unusual behavior of tungsten has not as yet been fully explained in the literature. The deposition of tungsten was, for all practical purposes, eliminated by placing the filament 2.2 cm. from the target.

Since these innovations were made, the x-ray tube operates satisfactorily.

Extent of the

The vacuum of the x-ray tube system was obtained from a calibrated thermocouple gage that was mounted in the system.

The filter was made of finely divided vanadium pentoxide particles affixed to a thin piece of cellophane. The mass of vanadium per unit area was determined by equation (16). To determine the mass of vanadium pentoxide equivalent to the determined mass of vanadium, the value obtained from (16) was multiplied by the ratio of the molecular weights of V_2O_5 and V, i.e., $\frac{V_2O_5}{2V}$.

The Debye-Scherrer camera used for recording the diffraction patterns has been discussed in a previous section.

Photographs of the camera, diffraction tube, and control unit are on page # 27A.

Procedure

The diffraction tube was degassed as much as possible and evacuated to a pressure of the magnitude of 10^{-3} microns.

The sample was mixed with collodion and molded into the shape of a right circular cylinder with a diameter of 1 mm. The sample was placed coaxial with the Debye-Scherrer camera, i.e., in the path traversed by the x-ray beam. 35 mm. Kodak no-screen x-ray safety film was mounted on the inner periphery of the camera. The camera was mounted so that x-ray beams emitted at a 6 degree angle (with respect to the plane of the target) would be incident upon the sample. The target area, when viewed from 6 degrees, presents a nearly square source. The vanadium pentoxide filter was then placed over the beryllium

window.

High voltage was applied (30 K.V.) and the filament current increased until an emission current of 9 ma. was attained. The average exposure time was 6 hours.

The exposed film was developed according the procedures outlined by the Kodak Co. for their developing solutions.

The Bragg angles of the various lines of the diffraction patterns were determined with an appropriately adapted traveling microscope.

Data

The Bragg angles of the various lines of the diffraction patterns are listed with their corresponding values of $\frac{d}{n}$. The intensities of these lines were visually estimated. A value of 10 was arbitrarily assigned the most intense line in each pattern.

Anthranilic Acid; 6 hour exposure at 30 K.V. and 9 ma.

Bragg angle	Intensity	d/n
degrees	#	A°
7.95	7	10.4
10.95	10	7.60
13.68	1	6.10
15.00	1	5.57
16.13	9	5.20
17.27	3	4.84
18.68	2	4.51
19.67	110	4.28
20.80	2	4.06
21.90	2	3.87
23.34	2	3.64
25.00	2	3.42
29.23	3	2.95
30.30	2	2.86
31.26	1	2.78
32.00	2	2.72
34.07	1	2.57

Manganese derivative; 6 hour exposure at 30 K.V. and 9 ma.

Bragg angle	Intensity	d/n
degrees	#	$\text{\AA}^\circ$
5.20	9	15.8
9.90	2	8.47
14.08	2	5.93
15.22	10	5.51
16.50	1	5.09
17.96	2	4.67
19.43	1	4.33
20.89	8	4.03
26.12	1	3.27
28.30	1	3.04
28.78	1	2.99
32.40	2	2.96
33.70	2	2.60
35.00	1	2.51
36.02	1	2.45
38.32	1	2.33
43.28	1	2.00

Ni derivative: 6 hour exposure at 30 K.V. and 9 ma.

Bragg angle	Intensity	d/n
degrees	#	Å ⁰
5.10	9	16.3
9.70	2	8.53
13.64	1	6.10
14.82	10	5.63
15.38	8	5.43
17.44	2	4.82
19.51	2	4.32
20.40	8	4.13
26.42	1	3.24
29.08	2	2.97
30.96	2	2.81
32.55	2	2.67
33.10	2	2.64
34.48	1	2.54
35.54	2	2.47
37.00	1	2.39
38.28	2	2.33
41.85	2	2.16
43.45	1	2.09

Copper derivative: 6 hour exposure at 30 K.V. and 9 ma.

Bragg angle	Intensity	d/n
degrees	#	$\text{\AA}^\circ$
5.04	9	16.4
5.30	7	15.6
9.10	2	9.12
9.80	2	8.46
14.64	10	5.71
15.26	7	5.47
16.21	2	5.17
18.11	4	4.64
19.06	3	4.42
19.71	2	4.27
20.15	8	4.18
21.15	2	4.00
24.35	1	3.50
26.06	3	3.28
26.81	2	3.20
28.11	4	3.06
30.16	3	2.87
32.26	2	2.70
34.90	2	2.52
38.34	4	2.33
41.56	2	2.15

Zinc derivative; 6 hour exposure at 30 K.V. and 9 ma.

Bragg angle	Intensity	d/n
degrees	#	A°
5.00	9	16.5
5.40	6	15.3
8.95	2	9.27
9.85	2	8.43
14.32	2	5.82
14.50	10	5.76
15.10	6	5.52
16.38	1	5.12
17.35	2	4.83
19.26	3	4.36
19.90	8	4.23
25.14	1	3.40
26.25	1	3.26
28.63	1	3.00
30.17	1	2.97
30.70	2	2.92
32.30	3	2.70
44.30	1	2.06

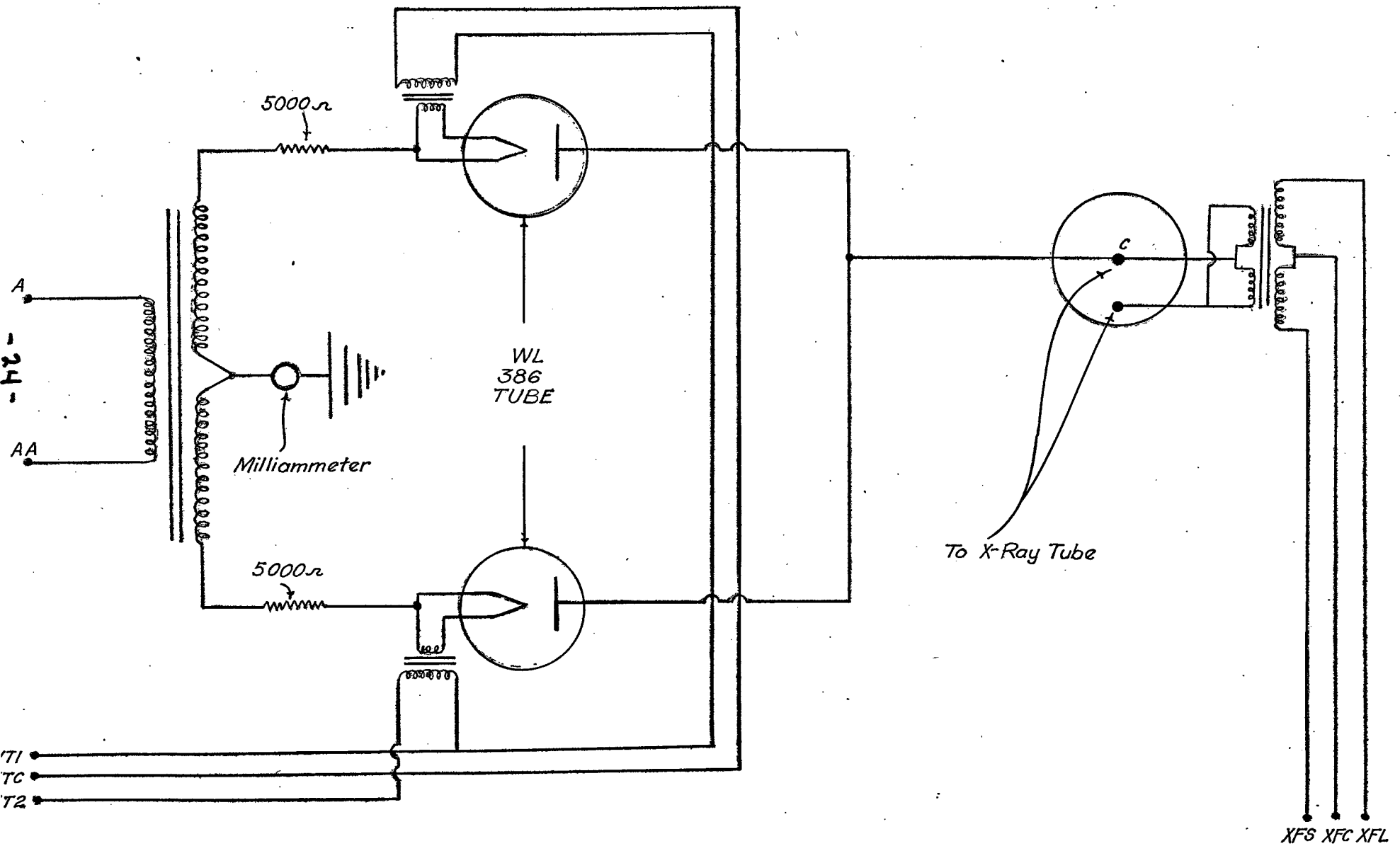
Mercury derivative: 4 hour exposure at 30 K.V. and 9 ma.

Bragg angle	Intensity	d/n
degrees	#	$\text{\AA}^\circ$
4.28	9	19.7
10.75	10	7.73
16.15	8	5.18
21.00	5	4.02
24.85	5	3.42
25.35	3	3.36
27.55	3	3.11
29.65	3	2.91
30.30	2	2.85
31.00	2	2.80
33.75	4	2.69
35.78	3	2.46
37.09	2	2.38
41.13	3	2.19
41.87	2	2.16
49.25	2	1.91

Lead derivative: 4 hour exposure at 30K.V. and 9 ma.

Bragg angle	Intensity	d/n
degrees	#	Å°
4.26	9	19.8
8.37	2	9.89
10.70	10	7.77
16.10	8	5.19
18.88	4	4.35
21.41	4	3.95
23.00	4	3.69
27.07	5	3.16
27.81	5	3.08
33.20	5	2.63
39.22	6	2.27
42.33	1	2.14
45.45	4	2.02

CIRCUIT DIAGRAM OF HIGH TENSION TRANSFORMER



CIRCUIT DIAGRAM OF CONTROL UNIT

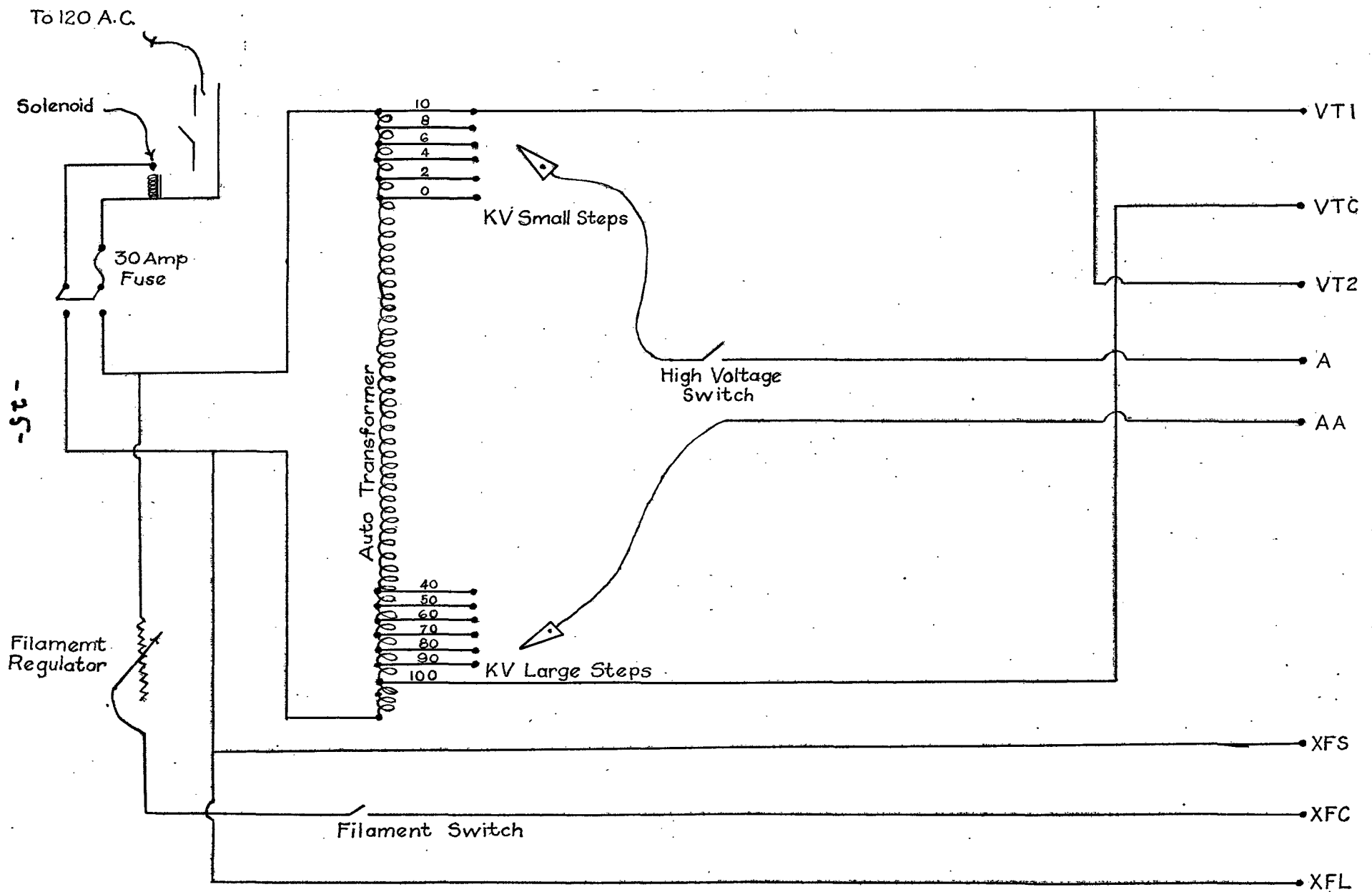
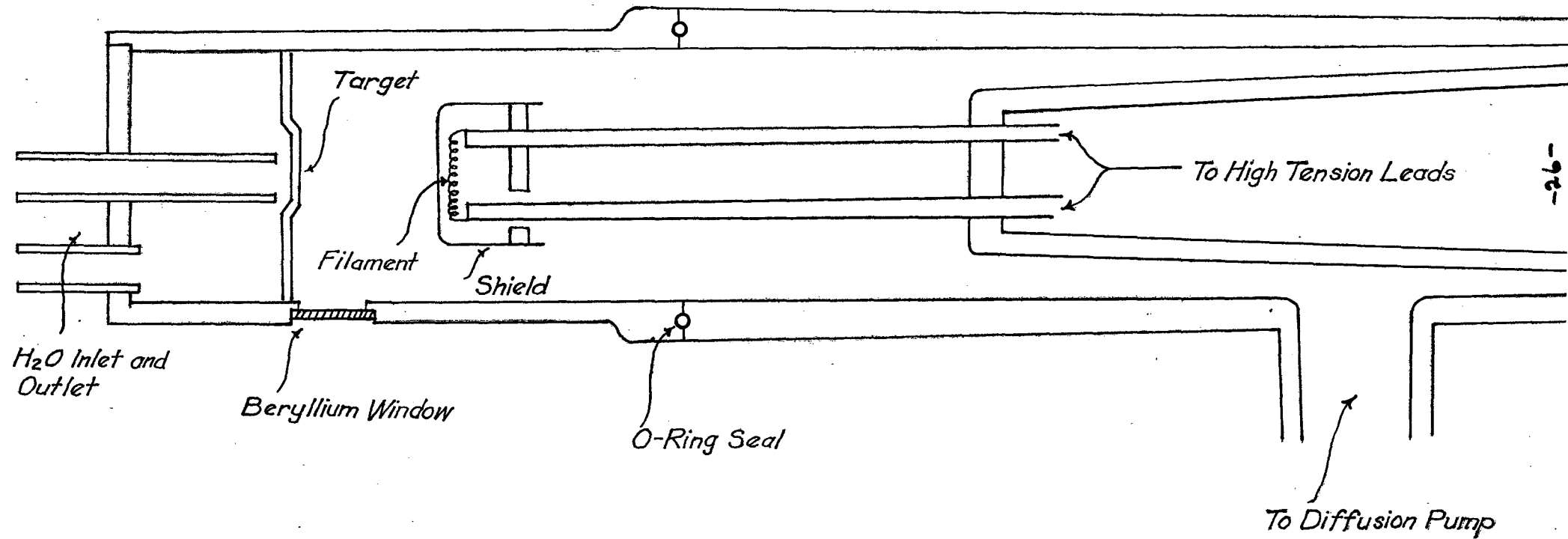
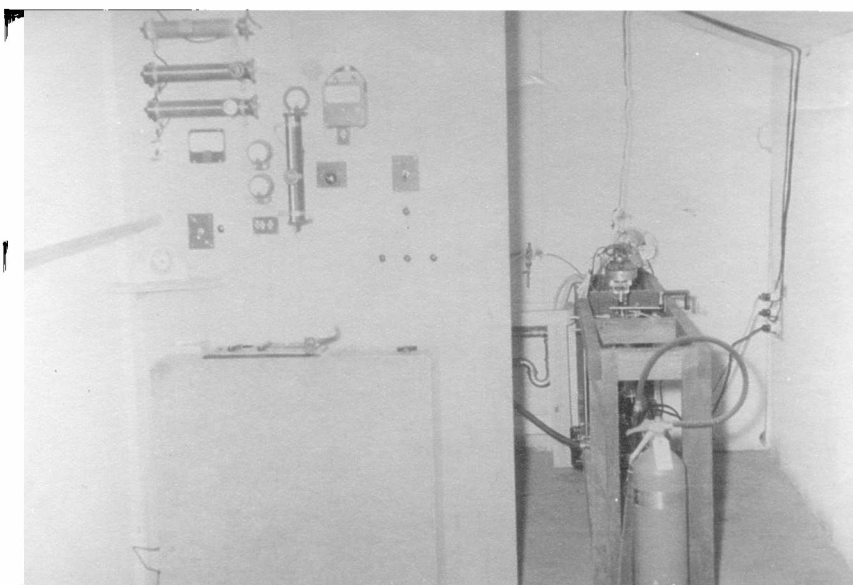
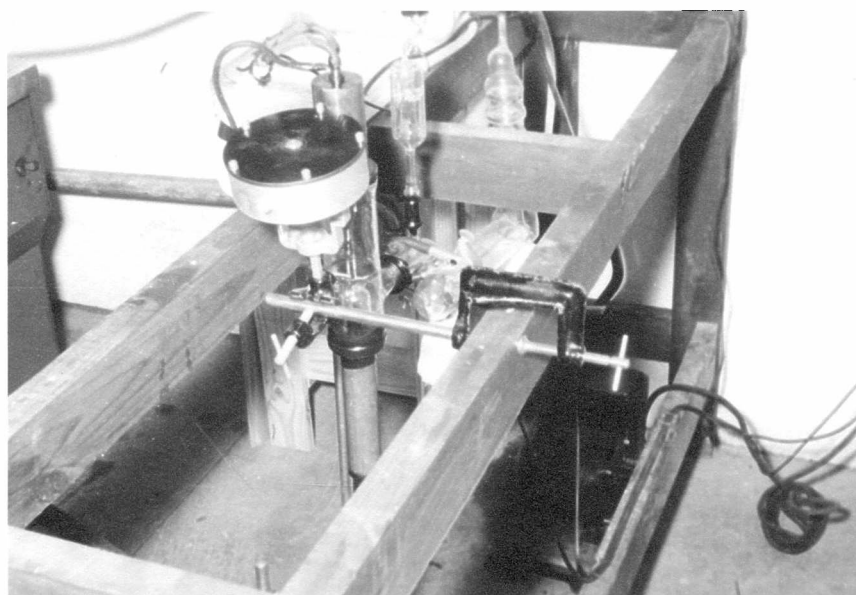


DIAGRAM OF X-RAY TUBE
ACTUAL SIZE

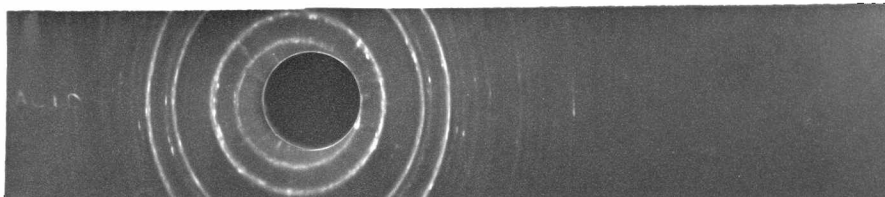




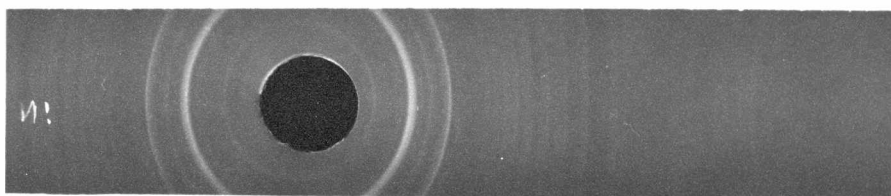
Control unit



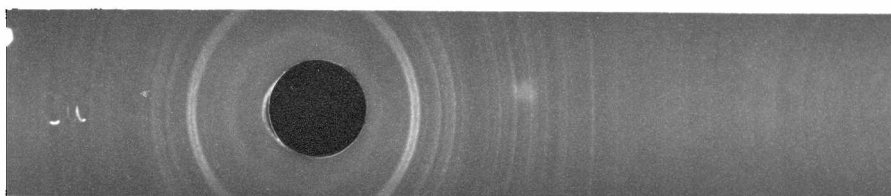
Tube and Camera



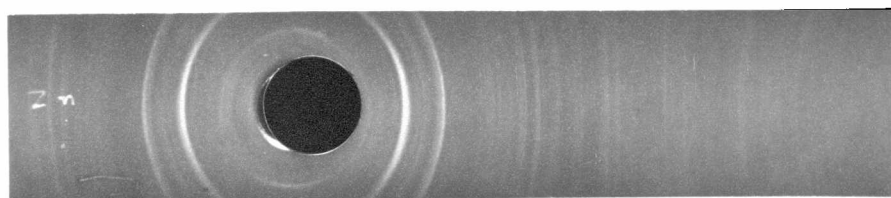
anthranilic acid



Nickel derivative



Copper derivative



zinc Derivative

Conclusions

Each diffraction pattern obtained was unique although the general distribution of lines was somewhat similar. On page#218 there are diminished, photographic reproductions of the diffraction patterns of some of the metallic derivatives of anthranilic acid. The similarity in the distribution of the diffraction lines infers that the general structures of these derivatives are similar.

The unique distribution of lines in each pattern affords a positive means of identifying each of these compounds.

The plot of the Bragg angle of the prominent diffraction lines against the atomic number shows a smooth curve, which enables one to predict the Bragg angle of other substitutions. This plot is shown on page#29.

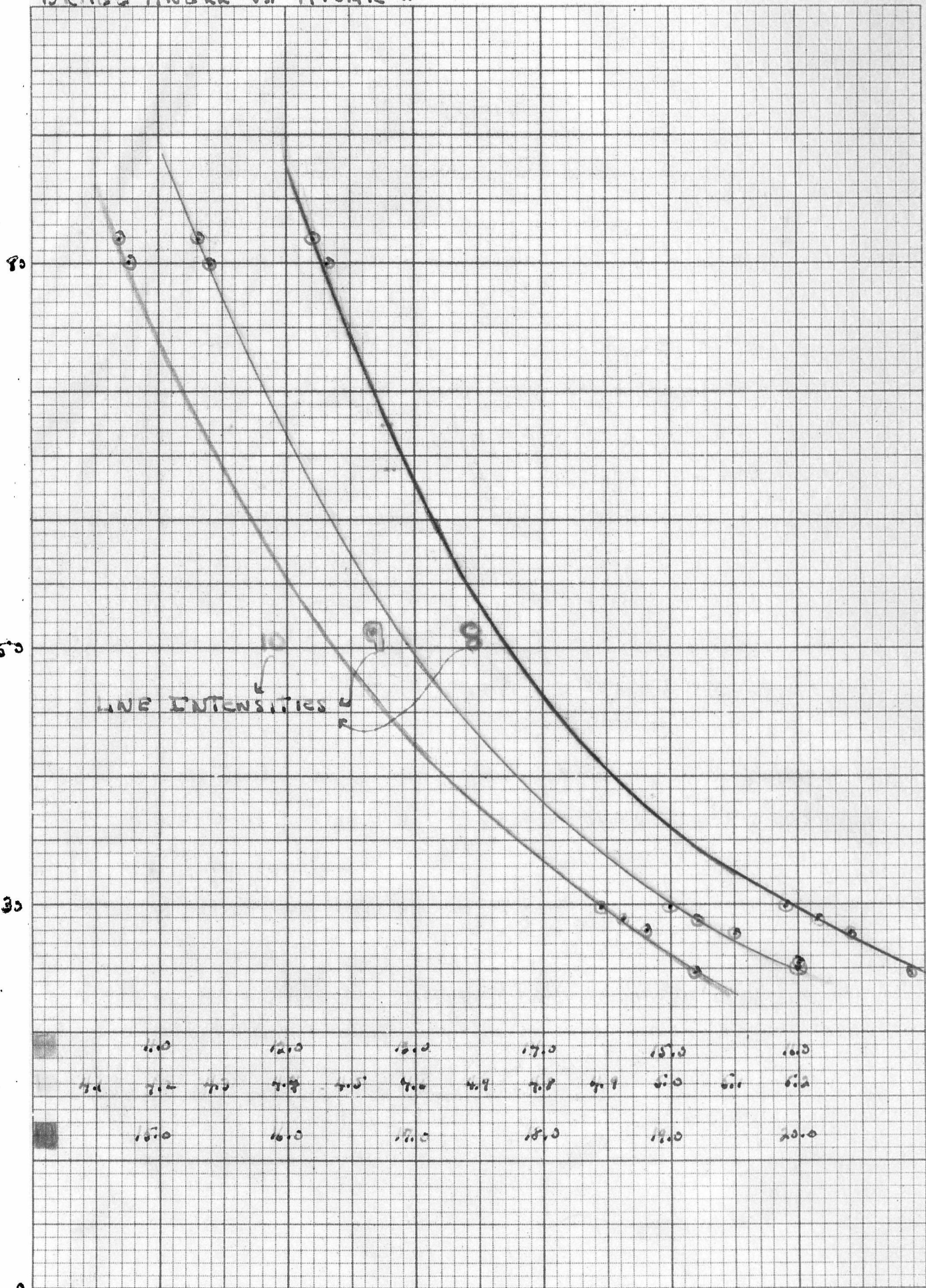
Errors

The relative intensities of the diffraction lines are somewhat in error. Following the standard practice, the line intensities were estimated visually.

The possible error in the Bragg angle (due to thickness of the sample) varies as the angle varies. The error is maximum at a Bragg angle of 45 degrees and minimum at Bragg angles of 0 degrees and 90 degrees. With the equipment used in this project, the estimated possible error at a Bragg angle of 23 degrees is 0.07 degrees and at a Bragg angle of 12 degrees is 0.04 degrees.

In conclusion, I wish to thank Dr. Mc Elrath and Dr. Long for their interest and suggestions in this work.

BRAGG ANGLE VS. ATOMIC



Bibliography

- (1). Hilbert, Wulf, Hendricks, and Liddel, J. Am. Chem. Soc., 58, 548, (1936)
- (2). Gilman, "Organic Chemistry" (John Wiley and Sons) Volume II 1948, pp. 1868-78)
- (3). Saburo Ishimarcu, J. Chem. Soc., Japan, 55, 288-92 (1934)
- (4). H. Funk and M. Ditt, Z. anal. chem. 93, 241-7 (1933)
- (5). H. Funk and F. Römer, Z. anal. chem. 101, 85-8 (1935)
- (6). H. Funk and F. Römer, Z. anal. chem. 96, 385-8 (1934)
- (7). Compton and Allison, "X-rays In Theory" (Van Norstrand) 1935, pp. 405-15
- (8). Sproull, "X-rays In Practice" (Mc Graw-Hill) 1946 pp. 391-419
- (9). Compton and Allison, "X-rays In Theory" (Van Norstrand) 1935, p. 61
- (10). L. de Broglie, Comptes Rendus 177, pp. 507 and 548 (1923)
- (11). Compton and Allison, "X-rays In Theory" (Van Norstrand) 1935, p. 32
- (12). Compton and Allison, "X-rays In Theory" (Van Norstrand) 1935, p. 802
- (13). T.H. Rogers, Proceedings Of The I. R. E., Vol. 35, No. 3 March 1947
- (14). J. Sci. Instruments, 22, 67 (1945)