# **ELECTRON DIFFRACTION**

Thesis for the Degree of M. S. MICHIGAN STATE COLLEGE John Hubert Muller 1954



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## ELECTRON DIFFRACTION

By

John Hubert Muller

### A THESIS

Submitted to the School of Graduate Studies of Michigan State College of Agriculture and Applied Science in partial fulfillment of the requirements

for the degree of

MASTER OF SCIENCE

## Department of Physics

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## STATEMENT OF THE PROBLEMS

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I

## Statement of the Problems

This work actually consists of three closely related problems in electron diffraction. The first of these that was tried was an experiment directed towards the possible construction of a simple means of measuring the energy of internal conversion electrons emitted by radioactive materials. Generally such measurement requires a large magnet with a carefully regulated current supply and some means of measuring the field with great precision. All this means a large project whenever one wants to make such an energy measurement. Electron diffraction on the other hand requires less in the way of a vacuum system, no large magnet, no precision method of field measurement, and no stable current supply. The calibration is furnished entirely by the known interatomic spacing of some crystalline material such as gold or aluminum, and the physical dimensions of the diffracting system.

The second problem was the construction and operation of a conventional electron diffraction apparatus using a heated cathode.

The third experiment was the diffraction of electrons at sufficiently low beam intensities that diffraction had to take place with essentially only one electron passing through the system at any given instant. This experiment was intended to show the dual character of matter,

specifically electrons, as both waves and particles. This would attempt to demonstrate that on a statistical basis, electrons had to obey a wave theory, even when there could be no interaction between any one electron and any other. Therefore, one had to assume that the wave property was inherent in each single electron; even though any particular electron was found to be localized when it gave its energy to a photographic plate. This localization at the photographic plate is one point that was assumed in this thesis as proven by the microscopic observation of tracks of individual electrons in photographic emulsions. See Sahni (18).

II

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## TECHNIQUE

#### Vacuum Technique

The mean free path required by the electrons in this instrument was estimated to be at least sixty centimeters. As the mean free path for electrons is somewhat longer than that for air molecules , a safety factor would result if the pressure in the vacuum system were low enough to provide the same mean free path for air molecules. This is satisfied by a pressure of one-tenth micron of mercury. It was evident that a diffusion pump backed by a mechanical pump would be necessary. Such a system was tried, but did not evacuate below fifty microns despite careful leak elimination. The difficulty was due to insufficient backing of the oil diffusion pump as noted by Strong, p.114 (21). Under insufficient backing the diffusion pump oil cannot rid itself of contamination from high vapor pressure liquids in the oil. This is because the temperature of the oil at the outlet of the diffusion pump is lower than the temperature at the inlet. High vapor pressure contaminants therefore are not removed so readily as they are accumulated. Strong suggests using two diffusion pumps, though this is not necessary for pressures of  $10^{-5}$  centimeters of mercury. A more adequate mechanical pump was the solution and one was obtained from the Cotrell project, through the courtesy of Dean T.H. Osgood. With the use of this Cenco Hypervac pump, the vacuum improved

tremendously, progressing to a pressure of twenty microns backing pressure and a high vacuum of about five-tenths of a micron.

Further reduction of pressure was then made through a combination of eliminating leaks which were not noticeable at the higher pressures, and the more careful adjustment of diffusion pump heating temperatures. These temperatures are not very critical at the higher pressures but affect the performance of the pump to a great extent at better vacua. Surprisingly enough, there seems to be no advantage in having an extremely high temperature, as this raises the vapor pressure of the oil or its possible contaminants sufficiently to overcome any improvement in the pumping force of the diffusion stream. There is slightly faster pumping speed at higher backing pressures with the greater supply of heat, but the ultimate vacuum is definitely poorer. In this case the optimum was found to be somewhat lower than the full heat intended for use with the pump as designed. The first major improvement in the vacuum by means of leak elimination and diffusion pump adjustment lowered the pressure to the desired region of one-tenth micron. A cold trap which had been incorporated in the original construction with the thought that it would be required in reaching the desired pressures was removed when it was found to have no appreciable affect on the vacuum. This also had the additional advantage of removing

a large number of seals which are natural liabilities as sources of leaks. Indeed it has not been found necessary to use refrigerated traps in this vacuum system in order to reach pressures as low as one-hundredth of a micron. By refrigerated traps is meant those operating at lower temperatures than fifteen degrees centigrade; as the diffusion pump used here had a water cooling system, not only to condense the oil vapor on the high pressure side of the jet, but also a spiral water cooled baffle in the high vacuum inlet to condense out any possible back-streaming vapor from the diffusion jet. In this connection Hickman (9) suggested that Octoil, the diffusion pump fluid used here was sufficiently well trapped by temperatures of tap water.

Similarly it should be noted that the commonly feared accident of air at atmospheric pressure coming in contact with hot diffusion pump oil (other than the inert silicones) is not such a serious matter as is usually supposed. When this occurred here, the removal of the oxidation products was attempted by merely leaving the system pumping overnight. In twenty-four hours the pressure was as low as it had been previously - about one-tenth micron. This obviously not only saved a great deal of work, but also avoided the introduction of other possible contaminants in the cleaning process, and possible leaks on rescaling the system. It may be, that with other fluids than Octoil, or with very much lower pressures, such a course of action would not be

sufficient, but it certainly was sufficient in this case.

Adequate cleaning of a new vacuum system seems to require the mechanical removal of gross rust, scale, and debris followed by thorough washing with soap or detergent and water and complete rinsing and drying. Special acids and chemical methods for rendering the surface inert did not appear necessary for pressures as low as one-hundredth of a micron. Some acids are indeed very useful in removing soldering fluxes or rust, but are otherwise unnecessary.

Pressures were measured in this system by means of thermocouple gauges for the higher pressures, and an ionization gauge for the lower pressures. The use of ionization gauges avoids many difficulties besetting other types. The pressure reading depends almost entirely on the number of gas molecules present, the geometry of the ion gauge, and the emission current which can be easily read and set to the required value. The fragile filament is a disadvantage. Another is the heating of the bulb by the filament which causes outgassing and therefore false readings. This difficulty can be nearly eliminated, however, by preheating the gauge measurement.

In the matter of construction, brass, aluminum, and magnesium plates and flanges, soldered or bolted together were satisfactory. Pipe joints proved unreliable and were eventually discarded for all except thermocouple gauge connections and at the forepump. These joints failed

under torque stress unless soldered together. Joints requiring great mechanical strength were brazed or silver soldered, and in some cases were wiped over with soft solder which generally wets better and forms a better seal.

All the soldered joints were covered initially with a hard wax. such as Picein or Apiezon "W", once the great effectiveness of these waxes was appreciated. Glyptal varnish did not seem as satisfactory for several reasons. It required about four hours to harden to a usable consistency. while Picein took only twenty minutes. Once hardened Glyptal could not be softened by flaming or any comparably simple technique to seal any overlooked gaps. According to Strong (21), the vapor pressure of the hard waxes was lower than any except baked glyptal, and this pressure was reached as soon as the joint was cool, far sooner than the Glyptal, which continues outgassing at a high rate, long after the joint is quite hard to the touch. Leakfinding is about the same with both materials but checking the presence of the leak by means of the torching of the Picein was so rapid as to constitute an invaluable aid in improving the vacuum.

Several conventional methods, including a halide torch, were used to detect leaks, but the most reliable system was one suggested by Miller (16). This consisted of painting the system with a solution of ordinary soap in water, nearly at the boiling point, and so concentrated

as to be solid at room temperature. Indeed, the solution melted only a little below the boiling point itself.

This was not painted on with the idea of discovering bubbles due to pressure from the inside, but was applied to the system when under the best vacuum attainable with the leaks present. The method was used in connection with any rapidly indicating pressure gauge. When a leak was covered, the solution would flow into the leak and there harden under two influences; the cooling due to the system being at room temperature, and the fact that any water in the solution would promptly evaporate into the vacuum. The pressure gauge would then show first, a sudden increase in pressure, and then a decrease in the pressure below the previous best value. The plugging of the leak was so effective that a large number of leaks could be found at one session because the effect of the larger leaks originally present would be so diminished. Once a series of leaks were noted one could wash off the soap solution on the outside of the system (though it is probable that most of the soap plugs would remain) and seal over the leaks with Picein.

All demountable joints were made with Neoprene "O" rings. The satisfactory qualities of these can hardly be overemphasized. Due to their round cross section there is a high pressure per unit area between the Neoprene and the flange surface even at small total sealing pressures. They

were used with both Octoil and vacuum grease as lubricant, though where motion was expected the grease was usually used. These gaskets provided both sliding and rolling seals and one was regularly used for both motions in the same joint. A glass to metal joint using a small "O" ring was capable of withstanding 15 degrees of intentional axial misalignment without leaking under high vacuum.

The original system contained a complete bypassing valve arrangement to avoid waiting for the diffusion pump to warm up or cool down. At that time it was thought that the Hills-McCanna packless diaphragm valves would be quite satisfactory, but they were abandoned after finding many leaks only a few cycles of operation after they had been rendered tight. It was, in fact, more economical of time to wait for the diffusion pump to warm up than to search for the many leaks that were found in the pipe threads and diaphragms of these valves. The Sylphon bellows type of valve has proven so satisfactory in other vacuum applications that these would probably be practical, but their high initial cost prevented their use here.

Among other fittings tried for vacuum service in this project were the soldered copper fittings, and refrigeration flare fittings. The first proved excellent, the second, though better than pipe thread joints, were not very successful.

## Radioactive Source Experiment

The first experiment tried was the experiment on diffraction of electrons from a radioactive source. Throughout this experiment, the sources used were evaporated from solutions of potassium iodide obtained from Oak Ridge National Laboratory. The radioactive component in these solutions was nearly pure Iodine 131. The main reason for using this was its ready availability, and its high specific activity.

One difficulty with the use of Iodine 131 was the low percentage of radiation useful for electron diffraction. As can be seen from Figure 1,only six percent of the material disintegrates in such a way as to produce a gamma ray capable of ejecting a conversion electron in the eighty kilovolt range. Of this, eight-tenths of the gammas are effective in actually producing conversion electrons. Furthermore, because of the multiple steps in the different branches of the disintegration scheme, only six out of two hundred and six disintegrations are of the desired gamma ray type, so that the percentage of useful emissions is only 2.33.

The analysis of the solution as shipped listed less than nine-tenths of one percent of Iodine 133. The radiochemical purity stated was 99.9 percent.

There was, however, another serious drawback to this solution, which was that fact that it had been buffered

# FIGURE I. IODINE131 DECAY SCHEME ACCORDING TO METZGER & DEUTSCH (15)

GAMMA RAYS OF I					
Y RAY ENERGY	PER 100 COUNTS	Ne/Ny	N <sub>K</sub> /NL		
80±1	6	.8 <del>*</del> . 5	5.5 ± 2.5		
283±3	6	.05 ± .02	>2		
363 ± 3	79	.019 ±.005	5.2 ± 1.5		
638±5	15	<.005	·		



with sodium bisulphite to obtain a pH of 11.0. This alkalinity was necessary in order to prevent the dangerous loss of the iodine from solution as a gas. If the hydrogen ion concentration in the solution was not kept below a certain level some hydriodic acid would form and iodine would escape as hydrogen iodide gas.

So much sodium bisulphite was thereforein solution, that the potassium iodide represented only one-millionth, by weight, of the evaporated residue.

It was originally thought that a source of approximately 100 millicuries would be adequate for the experiment, but this proved too large from both the standpoint of self absorption and unsafe intensity of radiation. Two sources of one millicurie, and one of two millicuries were tried, with exposures of up to two weeks.

The diffraction camera was mounted on a magnesium bar which could be slipped bodily into the vacuum system. The source, and first slit, consisted of one unit. See Figures 2 and 3. It was mainly another magnesium block bored out so as to be almost entirely hollow, open at one end, and possessing only a very small hole at the other. The hollow space was cast full of lead, except for the exact center, which was occupied by an aluminum tube. Cross-holes were bored into the tube to provide for evacuating the central channel. The open end was bored out in steps of different drill sizes in order to form a baffle with the



FIGURE 2. DIFFRACTION SYSTEM USING RADIOACTIVE SOURCE

source holder proper.

This source holder was made by taking a cast of the baffle steps with some plaster of paris, making a mold of the casting, and casting a lead plug into the mold. See Chapter 15 in Strong (21).

A steel pin which fitted inside the aluminum channel in the source holder mount was imbedded in the lead when it was cast into the mold. The pin was bent so that it would be well anchored in the lead. The idea was, that the pin would provide a point source by itself. The pin would be coated by evaporation with the radioactive residue, and when viewed end-on would appear as a small point.

A second variation of this idea was used for the mounting of the source in subsequent trials. In these the plug was made as above, but instead of a pin, a small tube was used as a cup. In the last stage of making the plug a short piece of the aluminum tubing was set in the corresponding hole in the mold. The free end of the tube was flared so that it would be firmly held by the lead that was to be cast around it. The lead was poured in, and a lead plug,with steps matching those in the mount, was completed. The imbedded tube was identical with the liner in the source mount holder; the ends of the two tubes resting against each other. Boring out a small bit of lead that had flowed into the aluminum tube provided a small cup in which to evaporate the solution.



SOURCE HOLDER AND MOUNT



SECOND PINHOLE AND DIFFRACTION FOIL

FIGURE 3

In order to define the beam, a lead sheet with a pinhole in it was fastened over the exit channel of the source holder mount.

The diffraction foil and second collimating pinhole mount was a short, hollow magnesium block filled with lead and bored out. In this case, however, the pinhole was bored in the lead cast into the block. The gold diffracting foil was deposited in a vacuum upon a collodion film which had been picked up by a small bit of copper screen. See Harnwell and Livingood (8). The screen was clamped to the magnesium face of the block.

The film holder was likewise a block of magnesium with a slot planed and filed into the front face to receive the film. A half-inch hole was bored through the center in order to facilitate lining up of the system by eye previous to making an exposure.

The photographic film used in these experiments was Eastman No-Screen X-ray film on a safety cellulose acetate base. This was used because of its great sensitivity as measured by Marton (14). The film was developed in the recommended Eastman X-ray Developer. A water rinse was used as shortstop and the recommended fixer and washing period was likewise employed. When this proved not sensitive enough, a commercial high-speed developer, Von-L 35, was tried. This was prepared from the concentrate, with boiled distilled water in carefully cleaned flasks. This

showed far greater negative contrast for a given background fog and was employed in all further work on radioactive sources.

#### The High-Voltage Power Supply

The high-voltage power supply was constructed from an old X-ray transformer, a rectifier tube, and a filter condenser made of glass plates and tinfoil immersed in oil. This power supply had a large number of difficulties. On damp days the surface of the rectifier tube and its mount would be covered with corona discharges. The filter condenser arced over at less than the necessary twenty-thousand volts. Even the transformer sparked between terminals, along the terminal board surface, and from the windings to the case.

First the rectifier was replaced with a type KR-3 which has a much longer leakage path. Several other condensers were tried, but none proved satisfactory until a bank of twenty-five eight-thousand-volt, one-hundredthmicrofarad mica condensers were hooked up, five in parallel, in series with four similar parallel sets. This made a total capacity of one-hundredth of a microfarad, with a nominal voltage rating of forty-thousand volts. See figures 4 and 5. The transformer was filled full of transformer oil, it's terminal board was carefully cleaned, and one unused terminal was enclosed in a glass beaker for insulation. Mounting the rectifier proved to be a problem, but this was temporarily overcome by several layers of glass plate.





CONDENSER BANK, BLEEDER, RECTIFIER, AND FIRST CATHODE ASSEMBLY

FIGURE 5

At this point, the necessity of moving the equipment to a new location precipitated several much needed reforms in the equipment. The first was a remounting of the entire power supply so that most of the stray corona leakage could be eliminated. The condenser bank was fastened together with sheet metal bent so as to present only rounded surfaces to the outside. This sheet metal served as interconnection and support both, so that only those condensers which were at low potential were near any grounded object.

A bleeder to equalize the voltage drops across the condensers was made with xylene resistors. These proved, however, to be not too successful and one made of twenty, 5.6-megohm resistors in series was substituted. See Figure 5.

The condenser bank was then supported from a braced shelf, and the bank was used to furnish an insulated support to the rectifier as well. The other end of the rectifier was directly connected to the transformer so that the only insulation that had to bear the high voltage was in the transformer itself and across the well-distributed voltage-drop in the condenser bank.

Technique for Conventional Diffraction

The production of a well-collimated beam of electrons from a heated cathode is perhaps not a very difficult problem, requiring chiefly careful attention to the previous efforts along this line as described in the literature. For example see Bachman (1), Coslett (3), Harnwell and Livingood (8), Hillier and Ellis (10), and Zworykin and others (24). The high intensities possible from a well designed beam-forming system are a little deceptive, in that one might imagine that only the most casual sort of cathode, anode, and slit system would provide adequate intensity for most purposes. See Harnwell and Livingood (8). This assumption is unwarranted.

The first attempt made, used a tungsten filament borrowed from the spares of a Hilger X-ray unit which was remounted on a flanged red brass pipe. The mounting was designed with a projection extending towards the anode beyond the filament itself, so that any possible gaseous discharge between cathode and anode would strike that, rather than the filament. This was thought to be adequate protection for the filament.

The cathode structure was sealed to a glass tube with hard wax. This tube was waxed in turn to the anode, and served as supporting insulator and passage for the electron beam. The rest of the diffraction camera consisted of a

brass tube in which were mounted two collimating slits and a removable photographic film holder. The anode plate was used as the mounting for the entire diffraction system, including the supporting insulator for the cathode. See Figure 5.

The anode plate was fitted with a groove for an "O" ring, and was mounted in place of the end flange of the main vacuum chamber. In this way, the entire diffraction system could be aligned as a unit outside the vacuum system, and then evacuated without disturbing the alignment.

The cathode was heated gently with a step-down transformer and a Variac, and tested for emission. Several milliamperes were emitted with only 90 volts applied between cathode and anode, and the filament was thought to be adequate. The original purpose of these filaments was to emit as high as 60 milliamperes in the X-ray unit.

Having eliminated most of the troubles in the high voltage power supply, a diffraction pattern was attempted.

A foil of gold was prepared as in the radioactive source experiment, and mounted on the second slit. When the high voltage was applied, there were frequent arcs to the projection on the cathode assembly. Numerous trials proved unsuccessful. Although a beam did pass through the system it was not intense enough and did not produce any visible diffraction pattern. Exposures of as long as two hours were tried at several hundred microamperes of

measured emission current.

This current was measured by the means of a virtually burnout-proof moving iron meter which had heavy conductors surrounding a small pivoted magnet. The use of a shortfocus telescope for viewing the meter face partially compensated for the very low sensitivity.

In order to get more information about the electron beam, the vacuum chamber was rebuilt with a fluorescent screen at one end. In addition, a highly effective cathode assembly was borrowed from an R.C.A. electron microscope through the courtesy of Professor H. M. Bendler.

This electron gun incorporated a filament enclosed by a grid cap which had a small central hole for the passage of the electrons. When the grid cap was biased about one hundred volts negative, with respect to the filament, an electrostatic lens was formed. See Hillier and Ellis (10). This lens had a broad optimum adjustment at bias greater than required for maximum intensity.

The entire cathode assembly was mounted on a glass cylinder waxed to a shaped anode in a manner similar to the R.C.A. electron microscope. The anode mounting was given an extra degree of freedom by means of an "O" ring which faced onto a flat plate rather than a groove. This permitted the entire cathode and anode assembly to be moved laterally, and also to be tilted a little by compression of the "O" ring.



# DIFFRACTION OF THERMIONIC ELECTRONS DOTTED LINES SHOW BEAM PATH WITHOUT STOPS

This new cathode assembly was tried first with the rebuilt vacuum system which had a fluorescent screen to display the electron beam. The screen was cut off from a Dumont 3GPl cathode ray tube, and waxed in place on a brass plate. The plate was the end plate of a chamber which was used to hold the photographic lantern slide for recording.

This slide was mounted on a plate which could be rotated into place through the rotation of an "O" ring joint. The fluorescent screen was then blocked from the beam. By sliding the shaft holding the photographic plate, the plate could be locked in the down position and disengaged from the shaft. Then the shaft could be rotated back,removing the cover from the film, and thus exposing the emulsion to the electron beam. This system could be reversed in order to remove the film from the beam, which would then strike the screen once more. The utility of such an arrangement is obvious. The pattern can be observed while adjustments are made, and an exposure can then be made without changing any of the parameters affecting the beam.

The amount of information that is to be had from a fluorescent screen is so great that such a screen is virtually indispensable to any research using an electron beam. This can be illustrated by some information found in short order. Electrons were being reflected from the



HIGH VOLTAGE SUPPLY, ELECTRON GUN AND FOCUSING COIL



TILTING ANODE MOUNT, ELECTRON GUN, AND MAGNETIC SHIELDING WITH BRONZE GAP

# FIGURE 7

walls of the vacuum tube. This could be seen by the fact that there was a brass pipe with large cutouts in the system, and a badly distorted shadow of this pipe could be seen. When apertures were placed in the beam it was possible, for instance, to see from the screen whether or not there was insulating dirt in the aperture. The effects of insufficient shielding of stray magnetic fields thus were detected. The remedy was the use of smaller iron pipe whose diameter-to-wall-thickness ratio was smaller.

In order to get a well collimated beam, it was decided to try magnetic focusing. This was applied by a short solenoid of 550 turns concentric with a gap in the shielding. The new shielding, which was internal to the vacuum system, was made out of ordinary iron pipe, and the magnetic gap was a short section of bronze pipe coupling and two bushings to make up the desired length. The coil was external to the vacuum system, and so could be adjusted both as to position and direction. In this way the focused beam could be aimed during operation.

Optimum adjustment of coil current rendered the beam nearly parallel. This condition was reached at a current of 1.1 amperes or 610 ampere turns. The beam was then stopped down by a hole six-tenths of a millimeter in diameter. This hole was drilled by hammering a steel phonograph needle into a sheet of phosphor bronze, and sanding the other side of the sheet until a hole was formed. This hole was

subsequently reamed with a sewing needle to result in a nearly round hole of desired size. No ellipticity could be detected by microscopic examination. The size of the hole was not critical, but depended mostly on intensity requirements; the beam collimation not being so precise as to warrant such a small hole.

The diffracting foils were mounted on the same holder as the beam stop and, were held in place by a simple clamp. To keep the sample cool the foils were mounted on the side of the aperture away from the source of electrons. There was no first pinhole in the strict sense of the word, but instead, a rather extreme lens stop located concentrically with the lens coil in the bronze magnetic gap. For conventional diffraction this stop was three sixty-fourths of an inch in diameter. The size of this stop was not critical except for the beam intensity which could also be adjusted otherwise. This could be controlled by variation of filament heating current, lens coil position, or electron gun blas.

For safety, the cathode structure was surrounded by a grounded metal enclosure. The high voltage lead was made of polished pipe with friction fittings intended for just such service in older X-ray installations. The intensity of soft X-rays was investigated, and found to be below tolerance limits. Nevertheless the rectifier tube was shielded by sheet aluminum which was sufficient to

stop all detectable radiation. No radiation was found coming from the anode in the diffraction unit, and this is presumed to be due to the use of magnesium in its construction. Most radiation emitted from a material of such a low atomic number under twenty thousand electronvolt bombardment must have been too soft to penetrate even the glass vacuum wall.

## Single Electron Diffraction Technique

Conditions for single electron diffraction were somewhat more difficult to achieve than the conditions required by conventional diffraction. This is because of the very small currents permissible in the electron beam.

The second pinhole presumably is not small enough to cause diffraction by itself, and a beam of collimated electrons issues from it. Without the diffracting foil only a spot is seen on the screen or plate; with it one can see a diffraction pattern. The intention here is to show that there is no interaction necessary between different electrons in order to produce that diffraction pattern. This can be accomplished by allowing only one electron to pass through the system at a time.

In the present experiment it was not possible to limit the beam to only one electron between cathode and emulsion at a time. It was possible, however, to limit the majority of the electrons passing between the second pinhole and the plate to such a low number.

The distance to be covered here is 46.6 centimeters. At the accelerating potential of 22,400 volts used, the speed of an electron is greater than 8.5 x  $10^9$  centimeters per second. This means that only  $5.5 \times 10^{-9}$  seconds is required to travel between the second pinhole and the

photographic plate.

The random statistical relation between successive electrons is affected by the cathode flicker effect. According to Johnson (11) the spectrum of this noise is chiefly prominent near ten cycles per second, the lower limit of measurement. Schottky (19) claimed this was due to changes in the surface of the cathode, and that it was a maximum at zero frequency. It is estimated that this noise is small enough not to interfere with the statistics of the electron emission. The lowest frequency noise could be seen as a slow wandering about of the thermally limited emission current, which was measured with a Leeds and Northrup wall galvanometer. Its sensitivity was approximately  $10^{-8}$  amperes per millimeter.

Protection was supplied by a 1/200 ampere fuse which blew out at irregular intervals of, perhaps, five minutes average duration. These current bursts were not sufficient to cause any noticeable deflection of the galvonometer. See Johnson Op.cit. In order to prevent the loss of measurement by the blowing of a fuse during a long exposure, it was decided to build an integrating circuit to smooth out the current surges. See Figures 4 and 8.

This consisted of a neon bulb shunting the galvanometer lead followed by a 5,000 ohm series resistance, a 4Mfd. shunt condenser, a choke of 720 henries, in series, another 4 Mfd. shunt condenser, and two more sections of RC



## FIGURE 8. INTERGRATING CIRCUIT



## FIGURE 9. BEAM CURRENT MEASUREMENT

integration using 5,000 ohms each and 4Mfd. each. The last condenser was followed by a 5,000 ohm resistor in series with a 1/100 amp fuse.

This fuse did not burn out; nor did the galvanometer show any fluctuations; and the neon bulb never fired. It is therefore assumed that the current pulses were not significant in their effect on the total exposure of the photographic plate. The cathode current was measured simultaneously with the current to the photographic plate or fluorescent screen. See figure 9. These were found to be in a constant ratio for unchanging high voltage and fixed focus coil current and position.

For the particular experiment carried out here, the ratio of emission current to that arriving at the final section of the vacuum system was twenty thousand to one. Therefore, with a measured cathode emission current of less than  $1.6 \times 10^{-7}$  amperes, the total current reaching the photographic plate must have been less than  $8 \times 10^{-12}$  amperes. This is equivalent to  $5 \times 10^{7}$  electrons per second. The average time between electrons must have been  $2 \times 10^{-8}$  seconds. The time of flight from the second pinhole to the slide was less than  $5.5 \times 10^{-9}$  seconds.

If the electrons are assumed to arrive in a completely random fashion, then some must be bunched together and others widely separated. In order to get a meaningful experiment it is necessary to determine what proportion

of the electrons do not arrive within  $5.5 \times 10^{-9}$  seconds of another electron.

A perfectly random emission of electrons is assumed, with no dependence of emission of any electron upon any other. Electrons are thought to obey Fermi-Dirac statistics, however, in which the possibility of two electrons in the same eigenstate is rejected. This would tend to militate against simultaneous emission of electrons from any one part of the cathode, so that the use of statistics which apply to a completely random situation can only strengthen the argument.

Assuming then, that the random situation holds in this case, one must consider a period of time before and after the arrival of each electron. The arrival of one or more other electrons within this interval will constitute an event rejecting that electron from consideration as a "single" electron. These periods, of length 1.1x10<sup>-8</sup> seconds, are scattered at random throughout the exposure, just as the electrons are.

Supposing each electron to be statistically independent of all others, a condition required by true randomness; then the arrival of electrons is not only random with respect to a fixed and densely packed set of time intervals, but also with respect to the intervals which are themselves randomly distributed. Therefore, one can use these intervals statistically in the same fashion as a densely

packed set of intervals.

In this situation one has to use the Poisson distribution which is the distribution for an infinite number of Bernoulli trials. The use of Bernouilli trials is made possible through the consideration of no electron as no event, and one <u>or more</u> electrons as <u>an</u> event. See pages 115-118 in Feller (6).

For such a distribution the probability of no event in a unit time interval is just  $e^{-\lambda}$ , where  $\lambda$  is just the average number of events per unit time. Here the unit of time is  $1.1 \times 10^{-8}$  seconds and the average number of events per second is just  $5 \times 10^{7}$ . Thus the average number of events per unit time interval is .55. Evaluating the exponential yields .577.

For large numbers of intervals, the proportion of intervals with no event will closely approximate the probability of no event in a unit time interval. This means that .577 of the electrons will arrive at the photographic plate, having been the only one between the second pinhole and the photographic plate during flight.

In order to obtain the low intensities previously discussed, several items in the diffraction system had to be altered. The most important was the installation of a smaller lens stop to realize a sufficiently high enough ratio of emission current to beam current through the second pinhole. The smaller lens stop was made by the

, • same means as the pinhole previously described, its diameter being .013 inches or .033 centimeters. The same beam stop was used, though a smaller one might have served adequately. The main difficulty with reducing the intensity further by means of stops is that the ratio of emission current to diffraction current becomes hard to measure.

In order to determine this, one has to make certain that the ratio is constant over a reasonable range before extrapolating the ratio to a lower current range. This is because the ratio will not be constant if the beam density is too high. See Gabor (7). It is safe therefore, to carry the extrapolation below a region of constant ratio; but it must be determined that the ratio is constant.

The current reaching the photographic plate was determined at higher values by a delicate galvanometer having a sensitivity of 5x10<sup>-11</sup> amperes per millimeter deflection at one meter from the mirrer. The beam current ratie was determined to be constant over a range of ferty te one; and the extrapolation was carried less than a factor of ten below the lowest direct beam current measurement. The photographic plate and fluerescent screen were in a part of the system which was insulated from the grounded main part by an "0" ring gasket, making the beam current measurement very simple. See figure 9.

III Results

## Results from Radioactive Sources

The use of a radioactive source for the electrons in a diffraction apparatus was not successful. Several reasons which contributed to this can all be summarized as insufficient intensity. In all the exposures one could see a central spot. The spot was small enough to have resolved at least the first two rings in the diffraction pattern of gold as a single broad ring.

Only the vaguest suggestion of such a ring could be observed on one photographic film, and then, only by some observers. Attempts to reproduce this negative on very high contrast photographic paper failed. A meaningful measurement of the ring that was thought to be on the negative was not possible.

There were two observable central spots on one negative which seemed to confirm that the radiation consisted of at least two varieties. It was assumed that the darker spot was caused by the gamma-rays and the energetic beta rays; with the lighter spot being the result of the 80 kilovolt conversion electrons. The displacement of the two spots from each other was the correct amount to have resulted from a stray magnetic field of the order of the Earth's field, but the orientation of the apparatus was such as to preclude the Earth's field from being a direct agent. The building frame or the accidental nearby presence of a



permanent magnet may have been the cause.

The conclusion is, that with a source of greater specific activity than was used here, diffraction might be a possibility. The use of Geiger counters would improve the sensitivity as compared to photographic detection. See Lennander (12).

### Conclusions on Conventional Diffraction

Conventional diffraction was attempted in this thesis for two reasons. One was to support the inconclusive results arrived at with the radioactive source; and the other was to build a system for use both as a measuring equipment, and as a demonstration and practice device for use with laboratory courses.

That conventional diffraction was attained with several materials should make reasonable a presumption that the work on the radioactive source was valid.

The second objective, the machine itself, presents more tangible evidence; though the merits of the equipment are matters of degree and have to be examined more closely. The accuracy of the measurements can be examined through the sharpness of the rings on the photographic slides. The use of glass plates supplies dimensional permanence to the recorded diffraction patterns. Thus, all that has to be ascertained in the determination of the useful resolution, is the production of these patterns, and the measurements on the slide itself.

The rings are sufficiently sharp to measure within two-tenths of one percent of the diameter. The distance from the diffracting foil to the photographic plate is large enough to get this accuracy with even the inner parts of many diffraction patterns. This is desirable in identifying a

# FIGURE 10

DIFFRACTING PATTERNS OF MAGNESIUM OXIDE AND MICA



particular substance, as the possible combinations of atoms forming a Bragg reflection plane become so great that identification of a substance by other than the inner part of the pattern is often a dubious process.

Determination of the ellipticity of the diffraction rings of powdered samples gives a good idea of the effect of the first derivative of the transverse stray magnetic field in the electron path. As measured on a pattern for magnesium oxide, this must not be more than two-tenths of one percent.

The longitudinal magnetic field inside the iron shield is probably negligible beyond the diffracting foil. However, the fact that there is no complete iron return path for the focusing field means that there is probably some longitudinal magnetic field between the open end of the iron shield pipe and the photographic plate. See figure 9. The consequence is a slight distortion of the diffraction pattern, especially at the outer edges. Distortion is actually observed in the outer parts of the mica pattern,which is a triangular array of equally-spaced dots. However, if such a pattern is used as a standard of comparison of grating constants, the distortion will be the same for both known and unknown in the same region of the pattern; and the error in the determination of lattice constants, can then be reduced below one percent.

Due to the fact that the high voltage represents the most uncertain value entering into the determination of

atom spacing in a crystal under diffraction, no absolute measurement has been attempted with this instrument. For this reason a sample calculation here of a lattice constant of mica is not intended to show any great accuracy. Rather it merely demonstrates that the instrument gives plausible results. The theory of diffraction is so well covered elsewhere, for instance in Lipson & Cochran (13), Pinsker (17), Semat (20), and Thomson & Cochran (22); that it will not be discussed here.

The Bragg equation is:

$$n\lambda = 2d \sin \theta$$

where:

n is the order of diffraction,

 $\lambda$  is the DeBroglie wavelength for electrons,

d is the spacing between successive planes in the crystal, and

 $\Theta$  is the angle through which the beam is diffracted. The DeBroglie wavelength  $\lambda$  is defined by:

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

where:

h is Plank's constant,

m is the relativistic mass of the electron, and

v is the velocity of the electron.

But,

$$v^{-} = \sqrt{\frac{2 Ve}{m}}$$

where:

V is the accelerating potential in E.S.U.

e is the charge on an electron in E.S.U., and

m is the relativistic electron mass.

Substitution of the various values into the formulas yields d=3.023 Angstrom units. But this has to be multiplied by the square root of three because the lattice constant of mica represents the smallest distance in which the unit hexagon is repeated, while the shortest effective distance in diffraction is the side of the unit hexagon. (It should be noted that mica has a pseudo-hexagonal structure.) The first is the distance between opposite sides, and the second is the length of one side. Their ratio is the square root of three. Multiplication by this factor yields 5.24 A<sup>o</sup> which is 1.4 percent high compared to the accepted value of 5.17 Angstroms. See Thomson & Cochran (22). This error is most probably due to uncertainty in the high voltage.

In any precise determination of the lattice constants of a substance virtually all errors, except those of measurement of the photographic plate itself, would be eliminated by the simultaneous diffraction of a material of accurately-known lattice constants. The parameters of the diffraction machine would not affect the accuracy except as they diminish the sharpness of the pattern.

The equipment is not especially refined, and a certain amount of technique is required to operate it without trouble. It would therefore supply a challenge to a student in an advanced laboratory course without being extremely difficult or time-consuming to operate. An ambitious student could add refinements, such as an R. F. power supply, or do reasonably decent measurement of lattice constants of some unknown substance. Conclusions on Single Electron Diffraction

Diffraction of single electrons that was carried out here was partly successful. The pattern produced by the five-hour exposure is as clear as any from a short exposure. The cathode current was monitored at least every five minutes during this period. The maximum values of emission current were not exceeded at any observation and the variation of readings gave no reason for concern. The average reading over the entire period was about six-tenths of the maximum to provide a large margin. Calibration of the exposure by the shorter exposures confirmed the calculated intensity. The proportion of single electrons that exposed the photographic plate was only .57; and these were single only in so far as they had passed the second pinhole. Only a minute fraction of those emitted from the cathode could have been alone in passage through the entire system. It is quite possible that, given sufficient time, much better statistics could be attained with this equipment.

In this connection, it is interesting to note that Biberman and others (2), who have done the first and probably the most statistically extreme experiment on single electron diffraction, showed only that those electrons that landed on the photographic plate, on the average, were separated from each other. This is because they cut down the beam by means of defocusing, so that there were many electrons

# FIGURE II

DIFFRACTION PATTERN OF MICA BY "SINGLE" ELECTRONS



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stopped by apertures. These electrons could conceivably have affected the formation of a diffraction pattern. A better experiment would limit the total emission of electrons sufficiently, so that all electrons coming from the source would be virtually isolated. Such a definitive experiment as Dempster's (5) remains to be made on electrons.

One of the more interesting of the various electron diffraction experiments carried out, has been by S. Lennander (12), using Geiger counters for detection of the electron beam. He probably achieved considerably better statistics than obtained here, but was not interested in the single electron diffraction problem, and made no comment on it. The use of Geiger counters is, in itself, interesting since a diffraction pattern is shown by a series of single events whose statistical frequency requires a wave theory. See Darwin (4).

As a last comment, one should point out the importance of this experiment as a necessary part of quantum mechanics. The theory is so well established that no information is to be expected from this work; but the result of the experiment is intellectually satisfying as the closure of a gap in quantum mechanics.

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