Design of a Double-Crystal X-Ray Vacuum Spectrometer

By LYMAN G. PARRATT
Ryerson Physical Laboratory, University of Chicago

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A vacuum double-crystal x-ray ionization spectrometer has been constructed and the design of the instrument is explained in some detail. A tank, of inside diameter 50 cm, formed from sheets of rolled steel and welded together, rests on a thick steel bed-plate, the contact areas, generously wide, sealed with stop-cock grease, and the chamber evacuated with a megavac oil pump. This chamber is used as the fore-vacuum for the Hg condensation pump working on a metal x-ray tube. The slits, the first and second crystals, and the ionization chamber are contained within the tank. The axes of the two crystals are rigidly fixed with respect to each other, and, to allow change of wave-length, the x-ray tube, clamped to the side of the tank, is swung around, the tank sliding on the stop-cock grease, until the proper position is reached. Final alignment of the focal spot with the slits is effected while the tube is in operation by an adjustable target made possible by a sylphon connection in the tube. A method is explained for obtaining thermal contact with uranium for use as the target. The angular controls of the crystal *B* and the ionization chamber are made by having long, concentric, tapered bearings extend through the bed-plate of the instrument, lubricated and held air tight with stop-cock grease, and their positons read to within one minute of arc with verniers on a fixed circle. Fine motion of crystal *B* is accomplished by the usual lever arm and tangent micrometer screw. The insulated lead from a relatively small ionization chamber, 6.7 cubic inches in volume, which is constructed to function as a two-atmosphere pressure chamber, is brought out of the tank through the hollow center of the central steel shaft forming the axis of crystal *B*.

INTRODUCTION

THE development of double-crystal x-ray spectrometry[1] has provided the researcher with an invaluable tool in making more precise measurements of x-ray diffraction. The many and wide-spread applications of the two-crystal instrument in investigations of contemporary importance have been considered sufficient justification for the design and construction of a double-crystal ionization spectrometer for operation in a vacuum, which would allow this branch of study to be extended to include the soft x-rays of wave-lengths greater than two angstroms.

TYPE OF INSTRUMENT

The double-crystal spectrometer, whose assembly details are described in

[1] The double-crystal spectrometer was first used by A. H. Compton, Phys. Rev. **10,** 95 (1917). Bergen Davis and his collaborators at Columbia developed the instrument for use in the parallel positions, Phys. Rev. **17,** 608 (1921); **27,** 18 (1926); **32,** 331 (1928); and the high resolving power of the anti-parallel positions was first exploited simultaneously and independently by Davis and Purks, Proc. Nat. Acad. Sci. **13,** 419 (1927), and by Ehrenberg , Mark, and Susich, Zeits. f. Physik **42,** 807, 823 (1927). The theoretical possibilities of the two-crystal method were very thoroughly explored by M. M. Schwarzschild, Phys. Rev. **32,** 162 (1928), and the experimental side has been investigated by S. K. Allison, Phys. Rev. **34,** 176 (1929), **35,** 1476 (1930), and by others.

this article, is essentially of the usual design[2] permitting the $(n_A, \pm n_B)$, or all combinations of the parallel and antiparallel, positions, with each of the two crystals capable of rotation about a vertical axis lying in its reflecting face. The collimating slits, the two crystals, and the ionization chamber are supported on a circular steel bed-plate, and a metal cylindrical cap which can be lowered to the plate encloses them in an air-tight compartment. When this chamber is exhausted, soft x-rays, which would be absorbed in air at atmospheric pressure, can be studied. The instrument is designed to accommodate glancing angles from 0° to 57°, which, with calcite crystals, correspond to a wave-length region of 0 to 5 angstroms.

Principle of Operation

A diagrammatic sketch of the spectrometer is shown in Fig. 1. The axes of the crystals are fixed with respect to each other, the first, axis A, mounted at the center of the large bed-plate, and the second, axis B, mounted 12 cm from A. The slits rotate about axis A and the ionization chamber about B. The x-ray tube is rigidly clamped to the side of the cylindrical tank (Fig. 2) which is lowered by a large screw to its position on the bed-plate.

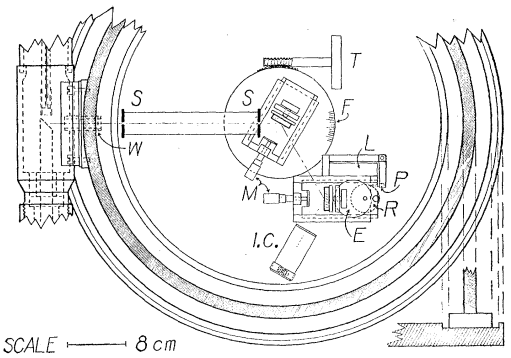

Fig. 1. Diagrammatic representation of the double-crystal spectrometer showing to scale the relative spacings of the x-ray tube, slits, first and second crystals, and the ionization chamber.

Before lowering the tank, crystal A is set for the proper glancing angle by means of the worm screw T as a vernier adjustment to 15 seconds of arc on the scale F. The proper position is determined by the condition that the reflected ray must pass directly through the axis of crystal B. The slits SS must then be placed in position as read on the same scale F and the tank lowered to the bed-plate and turned until the window W, between the tube

[2] A double-crystal spectrometer of usual design refers to one which is based upon two single-crystal spectrometers placed in proper alignment. Such an instrument can be placed alternately in the $(n_A, +n_B)$ and $(n_A, -n_B)$ positions by rotating the second crystal, B, and the ionization chamber. For descriptions of two different examples of this type of instrument see A. H. Compton, Rev. Sci. Inst. **2,** 365 (1931) and S. K. Allison, Phys. Rev. **41,** 1 (1932). Instruments of special design have been constructed by Du Mond and Hoyt, Phys. Rev. **36,** 1702 (1930), and by Spencer, Phys. Rev. **38,** 618 (1931), having certain positive advantages in treating particular problems, but which are limited in operation to the $(n, +n)$ positions.

and tank, is in line with the slits. This position has been previously allocated on a scale on the edge of the bed-plate and is now read by an indicator fastened to the tank. The tank is evacuated with a megavac oil pump and is used as the fore-vacuum for a mercury condensation pump working on the x-ray tube. This Hg pump is mounted on the top of the tank and thus is part of a rigid system.

The final adjustment of the focal spot-slits alignment is effected by a sylphon connection in the x-ray tube itself which allows independent motion of the target with respect to the tube and tank. The special features of this tube are perhaps worthy of discussion and will be briefly treated later in this paper.

The Tank

The tank itself is constructed of rolled steel $\frac{5}{8}$ of an inch thick. The curved section is shaped from a sheet bent into a cylindrical form, 50 cm inside diameter, and the ends carefully welded. The top piece is another sheet of the same material welded on to the cylinder, and a wide flange, shown in Fig. 1, furnishing large contact area with the bed-plate is similarly welded in place and turned in the lathe for a smooth surface. In the welding process

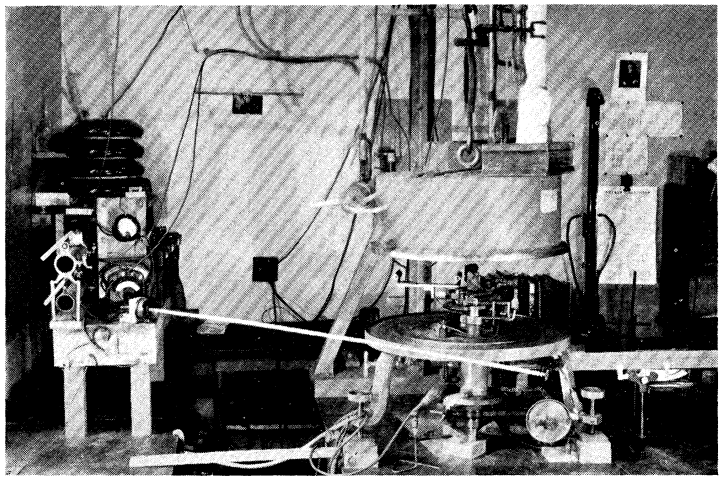

Fig. 2. Photograph of the assembly with the tank in an elevated position. On the left is seen the insulated battery stand. The electrometer is located beneath the far side of the bed-plate.

caution was taken to work the material well and to use an excess of rod to eliminate possible pin holes. The bed-plate is made of thicker steel than the tank to avoid undue warping. It was desired that the axes of the two crystals remain parallel to within one minute of arc, and calculation from structural formulae shows this condition is fulfilled by a bed-plate one inch thick. It is actually made $1\frac{5}{16}$ inches thick. An air-tight seal between the flange of the tank and the plate is made with stop-cock grease. These contact areas are of a generous width to allow a good seal and to supply additional strength.

A preliminary test for leaks was made in the following way: The tank was clamped to the bed-plate and the outside covered with soap solution. Air pressure of about 30 cm of Hg above atmospheric was maintained inside the tank and a search made for swelling soap bubbles. One small bubble was found. After a generous painting with Duco brush lacquer no bubbles were formed. The tank now holds a vacuum of less than 6 cm of Hg for about one week.

Axis of Crystal B

Following in general the design of Siegbahn's vacuum spectrograph,[3] long, concentric, tapered bearings, lubricated and held air tight by stop-cock grease are extended through the bed-plate BP as shown in Fig. 3. The crystal table C is rigidly clamped to the inner steel shaft S, and the support for the ioniza-

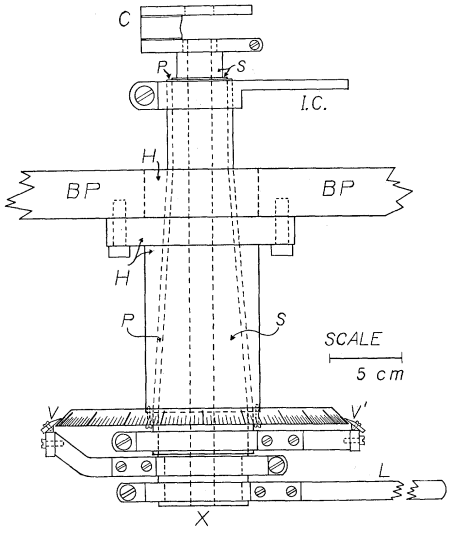

Fig. 3. Showing the tapered bearings S and P passing through the bed-plate BP and by means of which outside control of the second crystal and the ionization chamber is effected. Drawn to scale.

tion chamber, IC, is fastened to a conical shell P of phosphor-bronze, which in turn bears on the steel housing, H, screwed to the bed-plate. On the lower ends of these bearings S and P are clamped arms carrying verniers V and V', respectively, which read the angular positions of crystal B and the ionization chamber to one minute of arc on the graduated circle attached to the housing H. Crystal B must be set, of course, with an accuracy much greater than one minute of arc, but this serves as a first rough adjustment. After this setting is made, fine motion of the crystal is effected by a micrometer screw, bearing, at a distance of 20.63 cm from the B axis, on the lever arm L which is clamped to the inner steel shaft S. Fixed to the micrometer head is a large five-inch drum with 500 graduations, so that each division on the drum corresponds to an angular motion of the crystal of one second of arc, the pitch of the micrometer screw being one-half millimeter.

[3] Siegbahn, *Spektroskopie der Roentgenstrahlen*, Second Edition, page 114, 1931.

Atmospheric pressure is sufficient to keep the bearings tight and Lubriseal between them allows fairly free motion. However if the elements have not been turned for several days the Lubriseal becomes stiff and the motion is sluggish, and a work-out is required to loosen things up. To be sure that the motion as read on the drum is accurately transmitted to the crystal, and that possible slight variations in the thickness of the stop-cock grease during an operation would not be serious, a preliminary run with Cu K_α radiation was made and the observed separation of α_1 and α_2 checked with that calculated from Siegbahn's tables of wave-lengths. The observed values were consistent among themselves and also with the calculated values to within two seconds of arc. The rocking curves obtained in the $(n,+n)$ and $(n,-n)$ positions by rotating crystal B were also consistently regular and smooth.

The center of the steel shaft S is drilled out, as indicated in Fig. 3, and through this space passes the insulated lead from the ionization chamber to

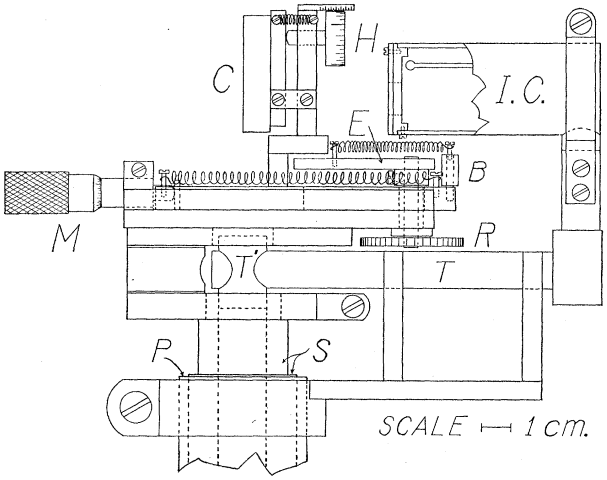

Fig. 4. Showing details, drawn to scale, of the mount of crystal B and the ionization chamber. The method of moving the crystal in and out of the x-ray beam is indicated, but the electromagnet is omitted for the sake of clarity.

the electrometer which is, of course, placed outside the tank. The air-tight seal is made at the bottom of the shaft at X where a flanged amber plug is waxed into place. The lead is unavoidably long and in consequence the capacity of the electrical system is relatively large, being 80 cm. The electrometer is of the Compton type and operates at a voltage sensitivity of 4000 mm (scale divisions) per volt per meter distance between mirror and scale.

Mounting of Crystal B

Both crystals are mounted on carefully made slides. The mount for crystal B, together with the ionization chamber, is shown in Fig. 4. The usual adjustments in aligning the crystal, labelled C, are made by the short micrometer M and the screw H. Constant pressure on the ends of these screws is maintained by springs. In many problems it is desirable to measure the in-

tensity of the x-ray beam reflected from crystal A only, and this requires that the second crystal be removed temporarily from its usual position so that it will not intercept the beam. To accomplish this purpose an electromagnet, L in Fig. 1, is clamped on the side of the crystal mount. This electromagnet, controlled by a tapping key outside the tank, activates a cog wheel R (Figs. 1 and 4) by means of a pall and ratchet arrangement (P Fig. 1). An eccentric wheel E (Figs. 1 and 4) is fastened to the axle of the cog wheel, and, as it rotates, one cog for each tap of the key, the eccentric presses against a roller bearing B (in Fig. 4) which causes the slide, carrying the crystal, to move back a maximum distance of some one and one-quarter inches, which is sufficient, for all glancing angles up to 57°, to allow the x-ray beam to pass. As the key is further tapped the eccentric moves around to its previous rest position, and the springs draw the slide after it until the motion of the slide is arrested by the micrometer screw, and the crystal is again in its usual rest position. This action is found to work very satisfactorily when a compensating spring of proper tension is attached between the eccentric and the bearing B, as in Fig. 4.

The method of alignment of the crystals and slits which was employed on this instrument has already been described by Allison[4] and by Tu.[5]

Ionization Chamber

The ionization chamber is constructed from a solid cylinder of copper $1\frac{1}{4}$ inches in diameter and $2\frac{3}{4}$ inches in length, drilled from one end to a depth of $2\frac{3}{16}$ inches, leaving the other end intact except for the introduction of the insulated electrode. The inside volume is relatively small, 6.7 cubic inches, but sufficient for the satisfactory absorption of the soft x-rays for which it is intended. The chamber is filled with one atmosphere of argon.

Because of the atmospheric pressure inside, and zero pressure outside, the chamber must actually function as a pressure chamber. A tapered amber insulator, in its grounded guard shield and Bakelite case, is carefully waxed leak-tight with a mixture of beeswax and resin and the unit firmly bound in place by two metal straps which pass over the top of the chamber (see Figs. 1 and 4). These straps are insulated from the chamber by strips of fiber since the voltage (about 70 volts) is placed on the chamber and the collecting electrode operates at earth potential, plus or minus the minute effects of the charges accumulated from the ionized gas.

The window on the ionization chamber is 3 mm by 10 mm and is cut from a piece of wrapping cellophane obtained from cigarette packages. This is the same type of window as used between the x-ray tube and tank.

The extended guard shield to which the straps are fastened, serves to support the ionization chamber as shown in Fig. 4. The chamber and the shield tube T must turn through an angle of 4 times 57° in the case of the maximum glancing angle in order to take care of both parallel and antiparallel positions. To allow this motion the table top supporting the crystal slide

[4] Allison, Phys. Rev. **41**, 1 (1932).
[5] Tu, Phys. Rev. **40**, 662 (1932).

must be hollowed out considerably. As the IC is turned the short tube T' slides inside the top of the steel shaft S supporting the crystal.

X-RAY TUBE

In constructing an adjustable target in the x-ray tube many technical difficulties in the alignment of the spectrometer were solved. Fig. 5 presents a cross-sectional view of this tube. The shell is turned from a three-inch cylinder of brass with a bearing left at B to guide and allow the target T but one degree of freedom. The sylphon S (flexible copper tubing) is soldered to this shell and also to the conical support of the target at the right end of the tube. Lubriseal makes the conical joint air tight and also allows the target to be easily removed for frequent cleaning, which is found necessary when working with soft radiation. Atmospheric pressure serves to keep a tension on the ball-bearings R, and, by screwing the cylindrical shell H in or out, the flexible sylphon contracts or expands and the target T slides on bearing B until the center of the focal spot is in line with the slits. Since the target is at earth potential this adjustment can be made while the tube is in operation.

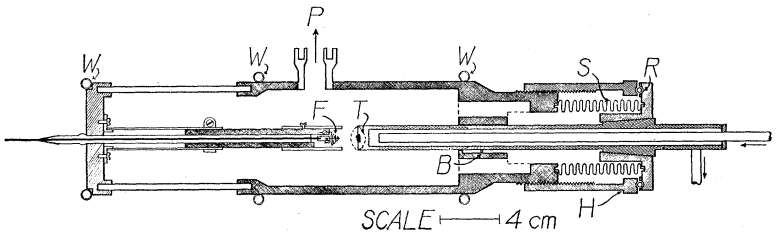

Fig. 5. Cross-sectional diagram of the x-ray tube showing the adjustable target. Drawn to scale.

Due to the drag in the ball-bearings when H is turned, a torque is transmitted to the target and an undesirable stress applied to the sylphon tube. This is eliminated by having a pin attached to the target carriage sliding in a slot in the bearing B. The pin and slot are shown in the diagram.

The filament F is easily wound by hand and is extremely simple to insert after loosening the screw and sliding back the focusing cup. The heavy nickel lead is introduced in a glass tube for mechanical support and electrical insulation and is spot-welded to tungsten to which a glass seal is made at the high-potential end of the tube. In order to keep the wax seals between the glass and metal from softening, water is circulated through the copper coils W soldered on the tube as indicated in the diagram.

The window W (see Fig. 1) to be placed between the tube and tank must be chosen to have a low absorption for wave-lengths up to 5 angstroms. The pressure differential on the two sides of the window is quite small, about 10^{-3} mm of Hg, and the mechanical strength of the window is of little importance unless it be desired to maintain a vacuum in the tube while the tank is raised for, say, a change of wave-length. Wrapping cellophane of thickness of one thousandth of an inch, similar to the window of the ionization chamber, is found to be satisfactory if the area is not too large. A window of 4 mm by 10 mm is near the limit for withholding atmospheric pressure. Such a window

ruptures frequently (about 3 hours) during operation if the tube is more than one-half kilowatt of power. This is somewhat overcome by inserting an extremely thin aluminum foil to act as an electrical conductor adjacent to and on the tube side of the window. With the tube operating at one-fifth kilowatt or less the window lasts indefinitely. However, in any case, the reduction in intensity due to absorption by the deposit of tungsten sputtered upon it may necessitate a replacement before rupture occurs.

The volume of the tube is made rather large so that the relative effect of the sudden release of gas from a gas pocket during operation would be reduced.

In a study of the reflecting powers of calcite from 2 to 5 angstroms which the author has just completed, the M series of uranium was used, and a special process of obtaining thermal contact between the uranium and the water-cooled copper part of the target was developed. The piece of uranium, whose lower surface has been freshly cleaned, is placed on a flat coil filament of 12 mil tungsten wire mounted under a bell-jar, into which are introduced two water-cooled electrodes, and the bell-jar evacuated to a pressure of approximately 10^{-4} mm of Hg. Current is passed through the filament after the desired pressure has been reached and the uranium heated. The process must be carried out in a vacuum to prevent oxidation of the uranium. Since the melting point of uranium is below that of tungsten, with a gradual increase in current, the uranium will melt at the points of contact with the filament and flow around the tungsten wire. With practice filaments can be wound so that, when heated, and the sag has taken place, they offer quite a large surface for fusion with the uranium. Repetition of the process gives a good base of tungsten on the piece of uranium to which one can either silver-solder or spot-weld without further difficulty.

Electrical Controls

The current for the filament of the x-ray tube is supplied by a series of three 6-volt A batteries, each of 150 ampere-hours capacity. A sliding contact resistance of 6 ohms is used to adjust the current through the filament, and the voltage across the tube is maintained constant during operation by a vernier control of the filament current. This vernier is another variable contact resistance of 150 ohms placed in parallel with the first, and manipulated by turning the long wooden rod extending to the observers seat from the high potential battery stand, seen at the left in the photograph, Fig. 2.

The equipment for generating the high voltage has been described by Allison and Andrew.[6] When working with low voltages, 0 to 15 k.v., a good microammeter in series with an accurately calibrated high resistance is used instead of the large electrostatic voltmeter described by them. Voltages could be read on the microammeter with an error of less than 30 volts.

The author expresses with pleasure his indebtedness to Professor S. K. Allison who proposed the construction of this spectrometer and who suggested many of the main features incorporated in its design.

[6] Allison and Andrew, Phys. Rev. **38**, 441 (1931).

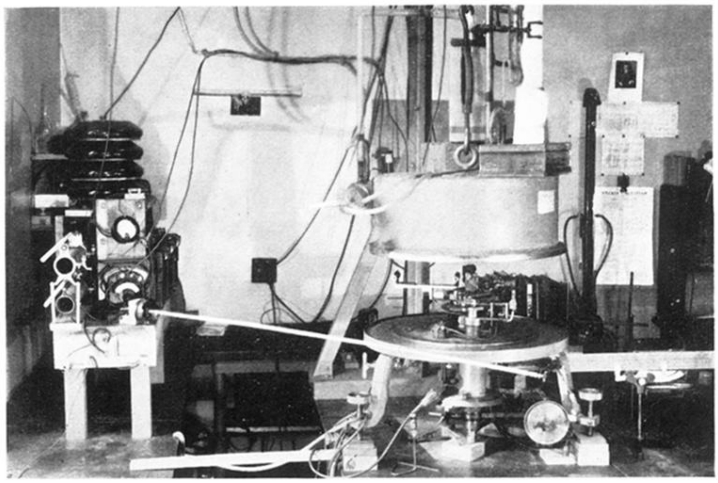

Fig. 2. Photograph of the assembly with the tank in an elevated position. On the left is seen the insulated battery stand. The electrometer is located beneath the far side of the bed-plate.