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ARGONNE DUAL NEUTRON DIFFRACTOMETERS USING A SINGLE PRIMARY BEAM

by

Melvin H. Mueller, LeRoy Heaton,
S. S. Sidhu, and John Terandy

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TABLE OF CONTENTS

LIST OF FIGURES

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ABSTRACT

Dual neutron diffractometers have been designed and built for use at the CP-5 reactor with a number of special features:

(1) Two monochromators are used in an in-line arrangement, as suggested by Levy and Peterson, utilizing the same primary beam with two independent diffractometers, one a horizontal instrument and the other a vertical unit.

(2) The two monochromating crystals may be used in transmission or reflection, and each crystal may be accurately positioned by external controls.

(3) Considerable variations in the wavelength of the primary beam may be made for both diffractometers by changing the take-off angle from the monochromator crystals by means of interchangeable pie-shaped segments.

(4) The monochromating crystals are located inside of a relatively small, heavily shielded housing which consists of lead, steel, Boral, and depleted uranium walls which have slotted holes for the exit of two beams, one for the horizontal and the other for the vertical unit.

(5) The monochromator housing is surrounded with a large moderating shield which consists of stacked 2-in.-thick Masonite die stock forming a 4-ft cube with a minimum thickness of 2 ft from the inner housing.

(6) The detector arm on the horizontal diffractometer pivots about the center of the diffractometer table or may also be made to pivot about a point approximately 2 ft from the center of the table. This arrangement permits the use of the horizontal unit as a diffraction instrument and also as a spectrometer for crystal dynamics studies involving changes of neutron energy.

(7) The horizontal table which has optional coupled θ-2θ motion is constructed so as to support massive accessories, such as cryostat, furnace, and magnet.

(8) The vertical unit consists of a Picker diffractometer to which is attached the shielded BF_3 detector and a counterbalance. Various specimen supports may be attached including a single crystal device which permits 360° motion in both chi (χ) and phi (ϕ).

(9) The movement of the detector arm for both horizontal and vertical instruments may be continuous or incremental by means of the stepping device recently described.

(10) Provision is made to read visually and to move three angles independently for both horizontal and vertical units by means of this stepping motor. The intensity and angle are recorded by an x-y recorder and in addition, the intensity versus angle is printed automatically with an IBM typewriter.

INTRODUCTION

Prior to 1953 neutron diffraction investigations at Argonne National Laboratory were carried out at the CP-3 reactor with a modified Zinn spectrometer. With the construction of the CP-5 reactor a new instrument was designed, built, and placed in operation in 1954. This instrument, called Spectrometer I, has some of the features of instruments previously used at Oak Ridge. The shielding was of the rotary type with the monochromator located at the center and θ-2θ table rigidly attached to this rotary tub.

This spectrometer was in use only for a short while when it became apparent that another neutron diffraction instrument was essential. Since suitable experimental neutron beam holes were at a premium, it was thought economical to design a dual instrument which would make use of the same primary beam, somewhat similarly to that planned at Oak Ridge National Laboratory and reported by Peterson and Levy.[1] This would provide some economy of neutrons. Based on the success of Masonite layer-type shielding used by Bacon[2] at Harwell and Shull[3] at MIT, it was believed that this type of shielding would permit flexibility in assembly and disassembly of the proposed instrument.

All during the design stages of this instrument it was kept in mind that there should be maximum flexibility, so that various types of diffraction studies could be carried out. A large horizontal instrument was desired which would be suitable for powder studies under various conditions necessitating use of magnet, cryostat, furnace, etc., as well as providing an instrument for the study of energy changes in the diffracted beam. It was fully realized that, if an in-line arrangement of the monochromators was used, there would be some attenuation of the beam from the first crystal, and therefore the intensity for the second instrument would suffer to some extent. However, it was believed that this could be tolerated if the beam from the second crystal were used primarily for single-crystal studies.

Although this description of the instrument could have been made shortly after assembly, there has been some advantage to waiting more than $1\frac{1}{2}$ yr. Thus, it has been possible to insert suggested changes which either have been found necessary to carry out already, would be desirable in the future, or would be recommended for the construction of another similar instrument.

In the following report this dual instrument is described, including the main shielding section which houses the two monochromators, the horizontal Spectrometer II, the vertical Diffractometer I, and the electronics for both instruments.

The CS numbers referred to in this report are from the Argonne Central Shop drawings. The more detailed descriptions of these instruments have been made in terms of the individual item numbers which appear on these drawings.

MONOCHROMATIC BEAM SOURCE AND MAIN SHIELDING

A useful neutron beam for diffraction purposes is obtained from the reactor proper by passing it through an inner pile collimator into the primary crystal housing. At this point the heterogeneous collimated beam is sorted into two monoenergetic, collimated neutron beams. The remainder is absorbed by the main biological shielding which consists of the primary crystal housing and Masonite sheets supported by a steel framework. Provision is made for the adjustment to the desired neutron energy by angle selectors for both the horizontal and vertical instruments. In the following section, a detailed description will be given of these various parts.

Inner Pile Collimation

The beam emerging from the CP-5 reactor core used for this instrument passes through an initial collimator (CS-1907-8), 85 in. in length, which has a rectangular opening $2\frac{1}{2}$ in. high by $1\frac{1}{2}$ in. wide.

This rectangular opening is defined by a stainless steel center tube which is surrounded by lead, steel, and heavy concrete shielding throughout the biological shield of the reactor, and by graphite encased in aluminum in the D_2O tank. The inner end of the collimator extends approximately 20 in. into the D_2O tank of the reactor, and the outer end terminates behind a rotary shutter which is a part of the CP-5 reactor shielding. This rotary shutter is approximately 18 in. in thickness by 3 ft in diameter, with an attached sprocket gear around its circumference for a chain drive for rotating the shutter. The shutter has a $4\frac{1}{8}$-in.-diameter hole eccentrically located at one side for exit of the beam. Suitable collimating slits can then be inserted in this $4\frac{1}{8}$-in.-diameter hole and, by means of stops and micro-switches, this hole can be lined up with the inner pile collimator described above, thus opening the beam hole. When desired, this beam hole may be closed by means of the motor-driven chain drive which merely rotates the shutter, thus moving this eccentric $4\frac{1}{8}$-in.-diameter hole from the in-line position.

Primary Crystal Housing

The beam emerging through the rotary shutter described above is 42 in. above the main floor of the reactor, as indicated in Figure 1. It now enters the primary crystal housing (CS-2397-1), which is a shielded box surrounding the monochromating crystals, as shown in the cross-sectional view of Figure 2. This primary crystal housing is supported and held in position by a heavy steel plate and rod framework. The main walls of this housing are 3 in. thick and were fabricated by welding $\frac{1}{4}$ in. steel sheet together to form a hollow shell which was then filled with lead. In direct line with the beam there is a pie-shaped, steel-jacketed, depleted uranium block which is 7 in. in thickness. There is also an inside wall lining of

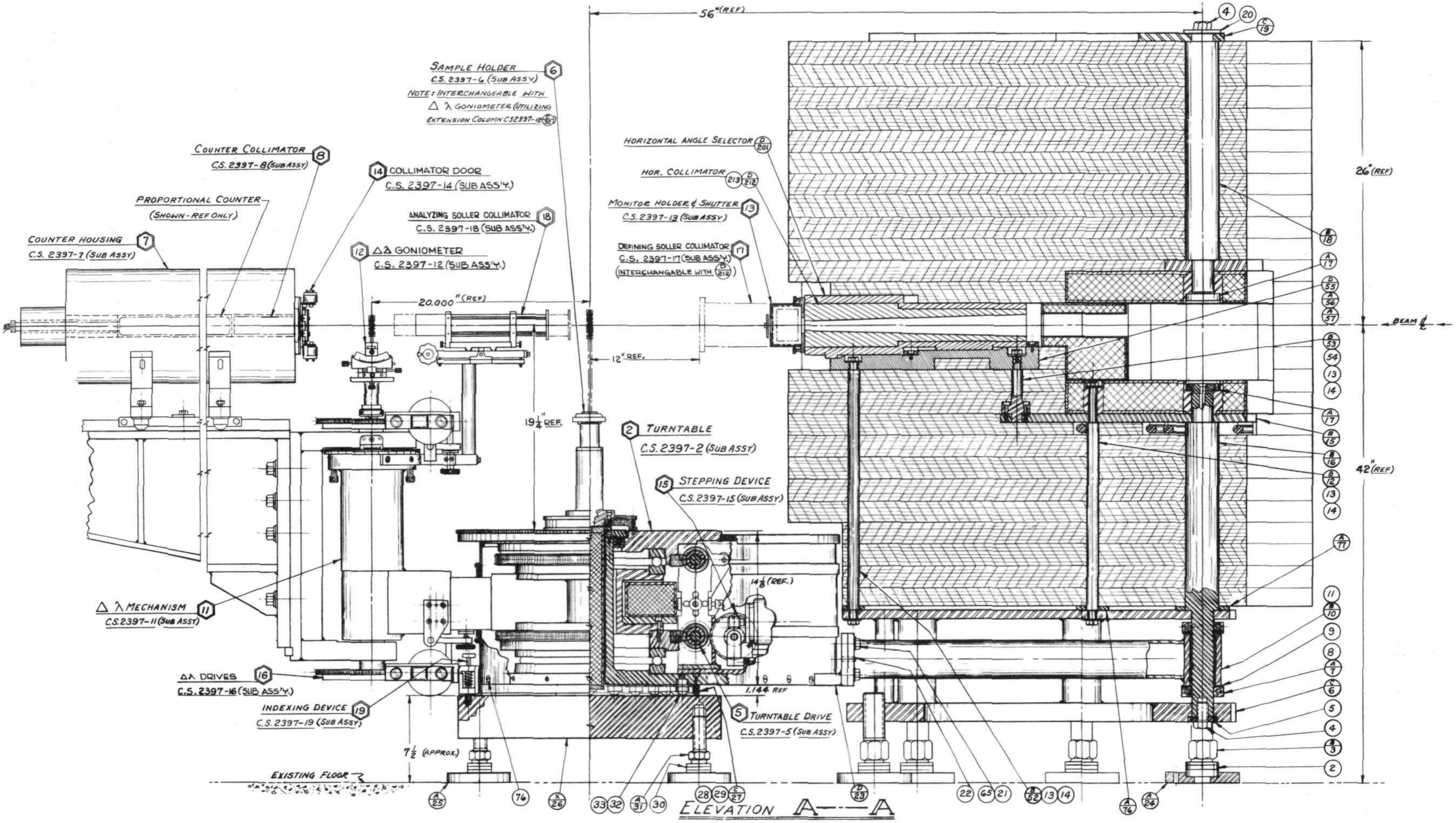

Figure 1. Cross-sectional View of Neutron Spectrometer II in Plane of Horizontal Beam

Boral, approximately $\frac{1}{4}$ in. in thickness, which, together with the uranium gamma shield, assists in cutting down the fast-neutron flux. In addition to the entrance opening for the beam next to the pile face, there are two exit openings provided: one for the horizontal monochromatic beam and the other for the vertical beam. The holders for the crystal monochromators are described later.

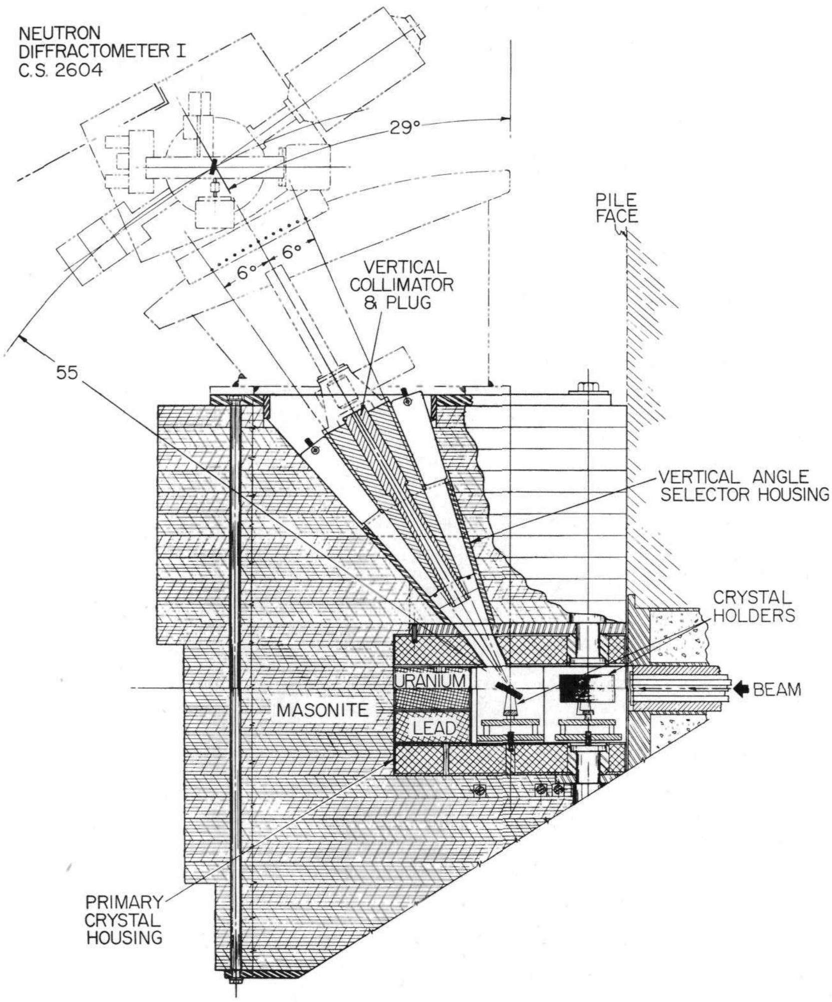

Figure 2

Cross-sectional View of Neutron Spectrometer II in Plane of Vertical Beam

Shielding Framework

From the initial stages, it was planned that this framework serve as a rigid support for the various components and hold them together to the desired accuracy. This was accomplished by use of relatively small, precisely machined parts which were bolted together, rather than by the use of massive shielding sections. This framework (CS-2397) within the external shield serves to: (1) support the primary crystal housing, (2) support the monochromator crystal holders, (3) support and hold together the Masonite shield plates, (4) provide alignment and controlled movement of the horizontal and vertical defining collimators, and (5) establish a pivot point for the θ-2θ diffractometer table. The framework base, C-6, in Figure 1 supported by adjustable jack screws, B-3, consists of two 36-in.-diameter circular plates held apart by five $7\frac{1}{2}$-in. lengths of 3-in.-diameter iron pipe. This assembly was stress relieved after welding, and the surfaces were finished square, parallel, and concentric to each other. The primary crystal housing is supported on this base by means of a 3-in-diameter center post, B-16, turned down at both ends to a 2-in. diameter with square shoulders and two 1-in.-diameter support rods, B-12, turned and shouldered to $\frac{3}{4}$-in. diameter. All ends were drilled and tapped for bolting securely to the base and primary crystal housing. This arrangement positions the crystal housing, the selector tie bar, B-15, and the base accurately and parallel to each other (see Figure 1).

The angle selector base, D-55, is held rigid to the previous assembly by means of two support rods, B-52, extending to the base, the selector pivot post, B-53, and to the selector tie bar, B-15. This base, D-55, serves as the positioning plate for the horizontal angle selector, D-201, and has a number of radial slots spaced 1° apart. This permits the 2θ take-off angle from the monochromating crystal to be changed over the range from 16° to 40°.

The vertical unit also has a means of positioning a collimator by means of the vertical angle selector housing (CS-2397-3), shown in Figure 2. Its outer shell is of steel and rigidly pinned and bolted to the top of the primary crystal housing (CS-2397-1), thus positioning it properly for the emerging vertical beam. It, like the horizontal unit, has a means of adjusting the take-off angle to 60° ± 6° from the vertical. Holes, spaced 1° apart, are drilled in the sides of the steel shell for accurate positioning of the vertical angle selector, D-301.

Angle Selectors and Associated Shielding

The horizontal angle selector is constructed of Masonite and steel, with offsets to avoid radiation leaks, together with a smaller insert horizontal collimator, D-212. This insert defines the beam, and may be easily removed and replaced with different collimation. The insert collimator used at present on the horizontal unit has a rectangular opening, 1 in. high by $\frac{1}{2}$ in. wide, at the sample end, which enlarges to 2 in. by $1\frac{1}{2}$ in. at the monochromator end. A horizontal and vertical cross section of this collimator can be seen in Figures 1 and 3. At the bottom of the angle selector there is a projecting key which slides into the radial grooves previously described in the angle selector base, D-55 (see Figure 1).

At the front of the horizontal collimator, provision is made for holding a fission monitor and a shutter assembly (CS-2397-13). The fission counter consists of an aluminum chamber, $3\frac{5}{8}$ in. in diameter by $2\frac{9}{16}$ in. thick, with front and back windows of aluminum, approximately 0.015 in. thick, and three parallel aluminum plates with each of the four inside surfaces coated with 0.1 mg/cm^2 of U^{235}. A metal box, approximately $2\frac{1}{2}$ in. x $2\frac{1}{2}$ in. x 8 in. long, houses a preamp and is attached to this counter. Attenuation is approximately 2.3%.

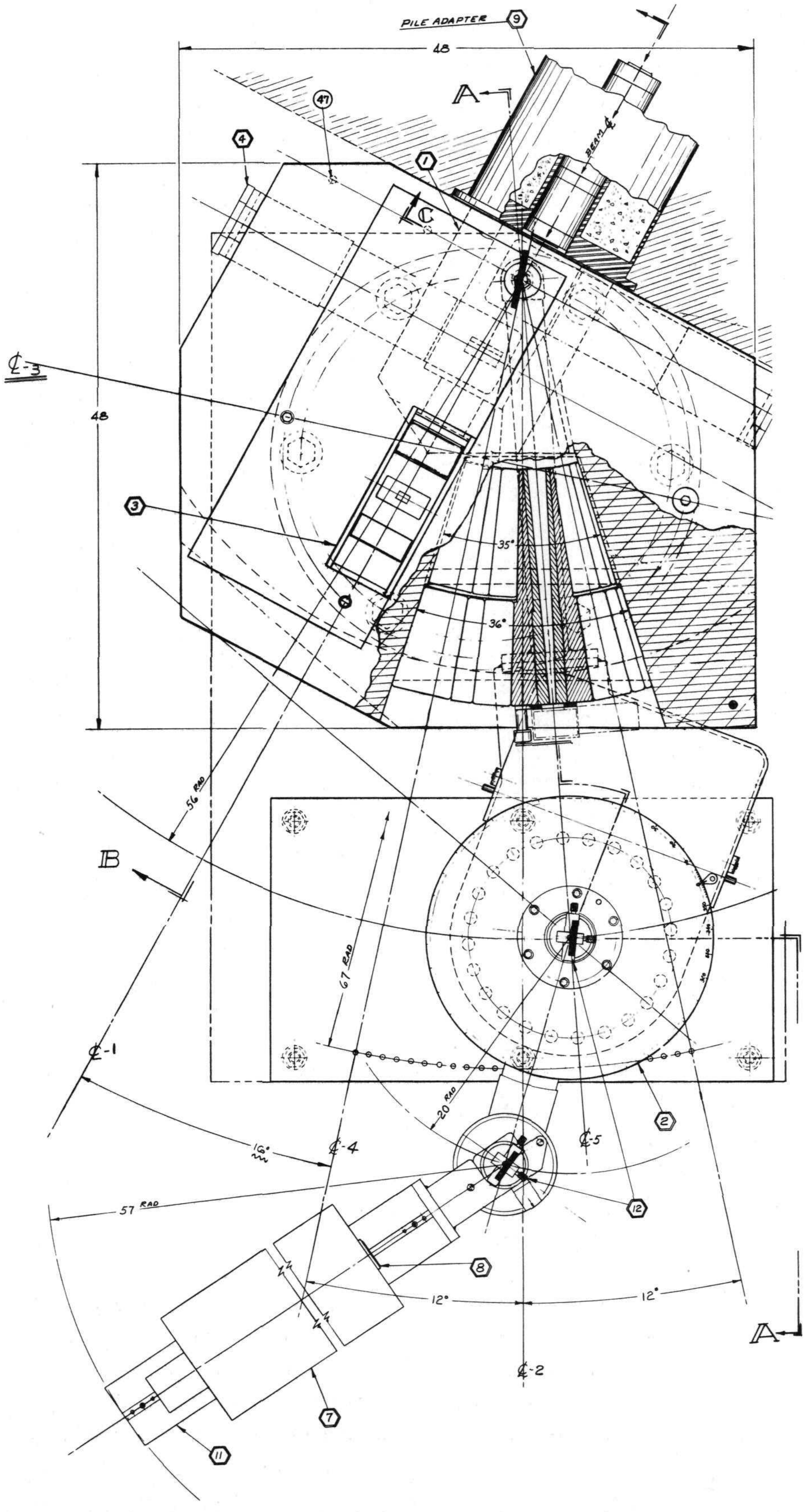

Figure 3

Top View of Neutron Spectrometer II

The shutter part of this assembly consists of a small rotary Leedex solenoid whose shaft is attached to a small paddle-shaped arm. This arm, by rotating up and down with the solenoid, can be made to intercept the beam. The aluminum arm with a cadmium insert for stopping the thermal beam provides a convenient way to cut out the monoenergetic beam without rotating the pile shutter.

The space between the sides of the angle selector and the angle-selector housing is filled in with two stages of shielding, the inner shield and outer shield. These pie-shaped sections have a steel frame with Masonite inserts. Two such rows of sections are used in order to provide offsets in the shielding with no in-line leakage. Both rows consist of sections having various angular incremental widths permitting an interchange of the sections from side to side and still filling the gap.

The vertical angle selector, D-301, is constructed similar to the horizontal one described above and has a collimator, C-302, with a 1 in. x $\frac{1}{2}$ in. wide opening at its end near the face of the shielding. This housing also has provision for attaching a fission monitor and shutter assembly at this point. In addition, this angle selector is provided with an extension (CS-2604-5, see Figure 9) which carries the collimation closer to the sample. There are two such removable extensions, one with a $\frac{1}{2}$ in. x $\frac{1}{2}$ in. square opening, and the other with a rectangular opening, $\frac{1}{2}$ in. by 1 in. In addition, provision has been made for conveniently reducing the hole size further by means of a cadmium mask in front of these extensions.

The pie-shaped shielding for this vertical angle selector consists of a single piece on each side with built-in offsets. At the present, this provides three positions for the vertical collimator, with an angular difference of 12°. Intermediate positions could be obtained by making different pie-shaped shielding sections.

Masonite Outer Shielding

As mentioned above it was decided to use Masonite sheets which not only serve as a good fast-neutron moderator because of their high hydrogen content, but make disassembly of the shielding easy. Stock sheets of Benelex (high-density Masonite), 4 ft square and 2 in. in thickness, were used. These sheets were cut to size with proper holes for the support rods and were stacked around the inner shield and within the shielding framework - thus, the Masonite forms an overcoat-type shield. The total gap between the shoulders of the support rods was filled in with several thin sheets of steel and Micarta. These thin sheets may be removed in the future to provide space for interleaving thin foils of either cadmium or indium in order to reduce radiation leakage at higher fluxes.

Monochromator Holders

In order to provide neutron beams for these two instruments, it is necessary to have two large single crystals which serve as monochromators and are located within the primary crystal housing (see Figures 2 and 3). These monochromator holders are described as goniometers (CS-2397-4).

The purposes of these holders are to support the monochromator crystals, to provide an external means of alignment, and to make it possible to readily remove and replace these crystals from the main shielding assembly. The external adjustment of both holders provides four translations, two linear and two rotary. In addition, the holder for the crystal for the top unit, Diffractometer I, has an additional rotary motion added for a monochromator crystal used in transmission. These holders are designed to accept crystals $2\frac{1}{2}$ in. x 5 in. and $2\frac{1}{2}$ in. x $2\frac{1}{2}$ in. to be used for Spectrometer II and Diffractometer I, respectively. In practice, crystals of copper, lead, and beryllium have been used to obtain neutron wavelengths from 0.8 to 1.6 Å. The holder assembly (CS-2397-4) consists of the framework for holding the monochromator crystals, a flexible cable remote drive arrangement for the crystal translations indicated above, the associated movable shielding, and the sliding drawer arrangement for removing the entire assembly. One such complete assembly is used for each monochromator with provision in the main shielding for inserting one assembly on each of the two sides of the shield. These monochromator holders have not been entirely satisfactory, and it would be a recommendation that they be redesigned with less backlash in the crystal adjustments and, in some cases, more built-in translations for the monochromators.

THE HORIZONTAL INSTRUMENT - DIFFRACTION SPECTROMETER II - (CS-2397)

The horizontal instrument has been used for the collection of both powder and single-crystal diffraction data, and has an added feature of a third axis for spectral analysis of the diffracted beam. The diffraction-spectrometer table is capable of supporting a ton of thrust; consequently, auxiliary equipment, such as cryostat, furnace, and electromagnet, can be mounted directly on the diffractometer table. The basic instrument consists of a turntable, specimen mount, counter arm, counter, collimator doors, and $\Delta\lambda$ mechanism. In the following paragraphs a brief description will be made of the various sections together with alignment procedure.

Turntable

The turntable (CS-2397-2) is supported on a heavy steel platform, 23 in. by 42 in., X-26, which is 4 in. thick and supported by six jack screws, A-31, as indicated in the side view in Figure 1 and in phantom in Figure 3. These jack screws support this platform so the top surface is $7\frac{1}{2}$ in. above the floor. Although the platform is not anchored to the floor, it was initially accurately located so that the arc formed by the holes in this platform, as shown in Figure 3, would lie on a 67-in. radius from the pivot point of the lower center post, B-16, of this instrument. This base was positioned so that these holes lie on radial lines passing from the center post along the individual slots in the angle-selector base to the corresponding holes in the base. The indexing device (CS-2397-19) fits into any one of the base platform holes and positions the turntable axis such that it intersects the corresponding radial line. Provision was also made to shift the turntable slightly for fine adjustment of this alignment.

The carriage frame, D-23, is attached to the pivot, B-10, which pivots on a bearing, A-7, at the lower center post (see Figure 1). This frame is supported on twenty-four 1-in. ball casters, Item 32, which make it easy to slide the turntable for changing the take-off angle.

The turntable assembly consists of the carriage frame, post, two ball-bearing races, two drive worm gears, scanning arm stub, tapered roller bearing, and turntable top. The arrangement of the turntable drive (CS-2397-5) which allows the sample to rotate with one-half the speed of the counter arm is shown in Figure 1. It can be noted in this figure that the table top is $19\frac{1}{4}$ in. below the centerline of the beam, which permits auxiliary instrumentation to be built on the table top without interfering with the beam.

Each large drive gear has one hundred teeth with a 1-in. face, and the θ-2θ motion is accomplished by using a single thread worm for the θ motion and a double thread for the 2θ motion (2 to 1 pitch difference between the worm drives). In order to couple the two worm shafts together, each shaft has a 3-in.-diameter gear attached. These gears are separated sufficiently so that a similar gear, $4\frac{1}{2}$ in. in diameter, mounted as a spring-loaded idler can be used to couple the two shafts. This provides an easy means of coupling and decoupling the θ-2θ drive.

Two types of drives were originally supplied for the 2θ table: one continuous and the other a stepping motion. In order to provide these two different motions, a clutch arrangement engaged by means of a screw knob made it possible to couple and uncouple the continuous drive Bodine speed-reducer motor (Type NSY-12RG, 1800 rpm with reducer to 3 rpm, $\frac{1}{75}$ hp). By replacing one gear in this drive, the following 2θ speeds could be obtained: 24, 12, 6, 3, and $1\frac{1}{2}$ degrees per hour. The stepping device (CS-2397-15) makes use of a 250-oz-in. torque Slo-Syn motor (type SS250) made by the Superior Electric Co. The motor is coupled through suitable gears to the 2θ worm shaft so that 1 revolution of the motor shaft resulted in a 1° change in 2θ. With this type of motor therefore, 1 DC pulse would provide a 2θ motion of 0.01°. The use of these motors for diffraction instruments has been described.(4) This motor and gear assembly is mounted on a slide arrangement with a screw drive which makes it possible to disengage this stepping motor gear from the main 2θ worm.

With both the continuous-drive clutch and stepping motor drive disengaged, a crank can be attached on either the extended θ worm shaft or the 2θ worm shaft. Thus, it is possible to move each independently or coupled with the hand crank. When the step motor is in use, however, hand operation is not necessary, since this motor can be used as an AC continuous motor which turns 72 rpm and serves as a rapid means of moving the 2θ arm. This same hand crank is also used for disengaging the idling gear which couples the θ-2θ drive.

A small set of reducing antibacklash gears is also coupled onto both ends of the θ and 2θ worm shafts. This provides a small shaft for an indexing drum which is rotated one revolution per one degree either θ or 2θ, depending upon the shaft to which it is coupled. These drums are indexed in 100 divisions and provide a means of reading the angle to hundredths of a degree. In addition, a mechanical counter attached to each shaft gives four digit reading of degrees (xxx.x). Therefore, with the combination of drum and counter, it is possible to read either the θ or 2θ angle to five digits (xxx.xx). A Coleman five-place digitizer (Model No. AP5DUT19) is attached to the opposite end of the 2θ worm shaft, and provision is made for a similar arrangement on the θ shaft.

This 2θ arrangement provides a convenient method for continuously checking if the electric and mechanical angle read out are in agreement.

In order to indicate the 2θ position on the x-y recorder, a 40-turn potentiometer (Helipot SE 404 10K) is coupled by a 45° bevel-gear arrangement onto the same shaft as used for the mechanical counter discussed in the previous paragraph. Although the potentiometer had the largest number of turns readily available, it was necessary to use a 6 to 1 reducer in the coupling between the bevel gear and potentiometer in order to cover the full range of 2θ. This reducer was an antibacklash Metron type (Series 9A with a ratio of 6 to 1).

This entire worm-and-gear assembly, together with the turntable drive, is enclosed. The enclosure consists of an aluminum case with a removable cover over the drive mechanism and a Teflon belt, approximately 6 in. wide, attached to the 2θ arm. This belt slips around in a small grooved track as the counter arm is turned.

Based on experience with the instrument, we would recommend that the large 100-tooth drive gears be replaced with 360-tooth gears. This arrangement would make it possible to eliminate a large number of small gears in the read-out system and probably improve the accuracy of the θ-2θ coupling. This would also make it possible to couple a stepping motor directly to each of the worm shafts for both θ and 2θ. Then, by a suitable electronic device, a 1 to 2 DC pulsing system would provide a θ-2θ coupling without mechanical gearing. This is the scheme used on the α-2α motion on the counter arm pivot point used for the $\Delta\lambda$ mechanism, which is explained later in this report.

Sample Holder

The top of the turntable is located $19\frac{1}{4}$ in. below the centerline of the beam. This makes considerable space available for auxiliary instrumentation in order to vary the physical environment of the sample. The table top has a central hub with accurately machined OD and ID reference surfaces, and is pinned and bolted concentric with the turntable top. The sample holder (CS-2397-6) for ordinary powder samples consists of a mounting arbor for supporting a Brown and Sharpe collet chuck. This arbor has a finished flange and shaft which matches reference surfaces, and is bolted and pinned to the hub. In order that the arbor axis coincide with the axis of the turntable, the powder sample (which may be contained in a thin vanadium tube with cadmium end caps - one of which has a threaded hole) is held in the collet by means of a support rod. This rod may be of different sizes for different specimen arrangements and collets, and threaded on one end for attaching to the sample holder with the other end machined to a somewhat smaller diameter. A shoulder on this end thus permits the proper height adjustment for the sample.

As an alternative sample-holder arrangement, an extension column (CS-2397-12) is provided which, instead of a collet, has a nest or platform whose height is adjustable from $3\frac{1}{2}$ in. to 5 in. below the beam height. This platform has the same hole arrangement as the G. E. spectrogoniometer base, and, consequently, the single-crystal orienter, flat specimen support, etc., may be conveniently mounted. There is also a limited rotary motion provided for this base by means of a segment gear worm and indexed knob. One complete turn of this knob corresponds to 1° rotation of the platform. This platform also serves as the base for the large goniometer with two translatory motions and two orthogonal arcs. This goniometer has been designed especially for the $\Delta\lambda$ mechanism to be discussed later.

Counter Arm

The scanning-arm stub, which is a part of the turntable as explained previously, terminates at a pivot housing 20 in. from the center of this table. (The function of this pivot will be discussed under the $\Delta\lambda$ mechanism.) The counter-arm adapter yoke, which attaches at this point, supports the counter beam and, when used as a diffractometer, this beam is held in a rigid in-line position to prevent pivoting. This was accomplished by two thumb screws which lock the yoke to the flange. The beam itself has a tapered I-beam construction fabricated of aluminum for reducing the weight. Although not shown in Figure 1, the arm itself consists of two parts hinged at a distance $35\frac{1}{2}$ in. from the pivot, which permits the arm to fold back on itself. It is generally used in the shortened position to prevent interference with neighboring instrumental set-ups at CP-5, especially when the arm is at high 2θ positions. The top of the counter beam has a $\frac{3}{4}$-in. by 1-in. steel rail down the center for guiding the back-and-forth motion of the counter.

Counter Housing

The counter housing (CS-2397-7) is supported on the counter arm with two aluminum cradles which rest on 1-in. steel ball casters. These casters are the same as used on the turntable and provide for an easy back-and-forth motion of the counter. The motion of the counter housing is confined by the central guide rail and two parallel positioning bars. One cap screw at the end of each bar provides angular adjustment of the counter housing and securely locks the housing in the back-and-forth position. The counter housing is a long cylinder which is built of alternate layers of 2-in.-thick discs of Masonite and 0.010-in.-thick cadmium discs held together by long, threaded rods inserted through steel end plates. A 2-in. hole through the length of the cylinder is lined with 0.010-in.-thick cadmium and provides space for the BF_3 counter tube, the associated preamp, and slit system. These parts are evident in Figure 1.

In the operation of the instrument, some difficulty has been encountered in the stability of the preamp, and its design has been changed. Instead of the 1-in. round type previously used, the preamp is mounted in a small box attached to the back of the counter housing with the high-voltage lead projecting into the housing. This then serves to hold the BF_3 counter in a fixed position within the housing. There is no shielding surrounding the new type of preamp.

One type of counter collimator (CS-2397-8) consists of a cylindrical insert, 7 in. in length, which contains a stacked arrangement of rectangular steel tubes, which form a Soller slit. These tubes are spaced by means of aluminum end plates in such a way as to occupy alternate positions. This checker-board arrangement provides a minimum of blanked-out cross-sectional area. The end plates of aluminum cause no particular difficulty because of the very high transmission of aluminum for thermal neutrons. Other desired collimators may be easily interchanged for the above.

BF_3 Counter Detector

The BF_3 counter (Nancy Wood Counter Laboratories) has a 2-in.-diameter brass case and a small-diameter central wire with a 20-in. active length. The BF_3 gas (96% B^{10} enrichment) is at a pressure of 60 mm, the operating voltage is approximately 2500 v.

Collimator Door Assembly

When the instrument was put in use, a method similar to that described by Furnas[5] was established for alignment of samples, especially single crystals. To use with the neutron beams, cadmium masks were made which permitted looking separately at top, bottom, left, and right portions of the sample. These masks were held and positioned in front of the counter tube by a rigid frame. Later, it was decided that this could be done much more rapidly by remote operation. This was accomplished by constructing an accurately positionable frame in front of the detector (CS-2397-14). Four cadmium plates were mounted within this framework and hinged on the sides, dividing the face of the counter tube into equal quadrants. Each one of these four plates or shutters was attached to separate rotary solenoids made by the Leedex Corp. (Types H-2259-031 and H-2419-031). The normal position for the shutters is the open position as held by springs on the solenoids. As soon as the solenoid is engaged, a rotary motion is imparted to the hinge pin and the shutter is closed (see Figure 4). A simple electronic control permitting separate motion was constructed for operating these shutters. Cadmium masks of various shapes - square, rectangular, slit, etc. - have been constructed for insertion in a recessed section just behind the shutters in order to provide a means of masking down the counter window.

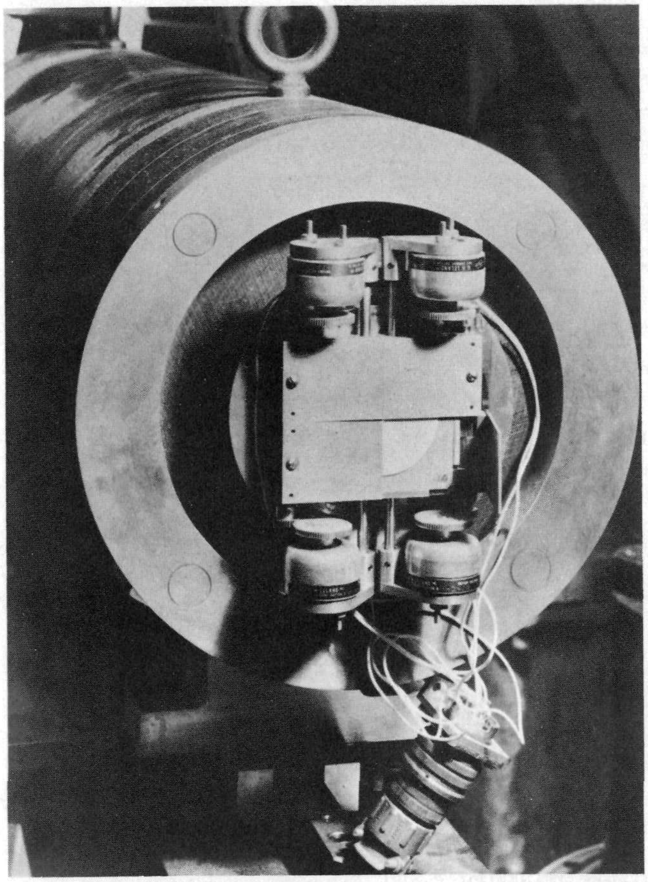

Figure 4. Collimator Door Assembly

Δλ Mechanism

As mentioned in the introduction, it was desired to make the neutron instruments as flexible as possible so that a number of different types of investigations could be carried out. Although the main purpose of the new instrument was for diffraction studies, it was possible to provide a means for studying changes in neutron energy after diffraction. In order to accomplish this, it was necessary to provide a pivot point in the counter arm about which the detector could be moved around an analyzing crystal which would be located at this pivot point. This Δλ mechanism (CS-2397-11) utilizes an α-2α coupling arrangement (CS-2397-16). Figure 5 and Figure 6, the side and top views, respectively, provide the details showing the construction of this additional pivot point. The two Δλ drive gears as shown in Figure 6 are located at the top and bottom of this pivot position. The top gear is used for rotating the central

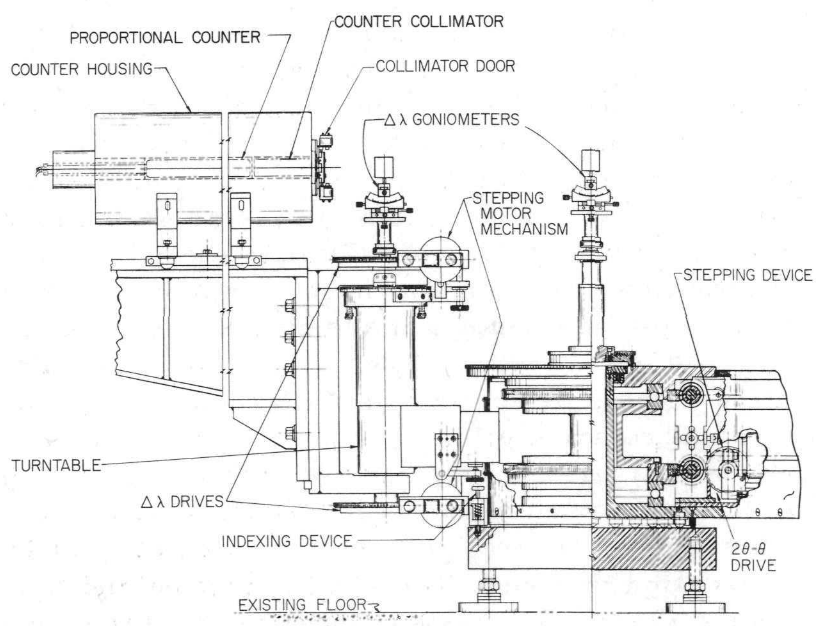

Figure 5. Cross Section of Spectrometer II Showing Δλ Mechanism.

shaft or goniometer axis (α motion), and the lower gear drives the counter arm about this axis (2α motion). These gears were purchased from Picker X-ray and are the same as the 360-tooth gears used on their diffractometer.

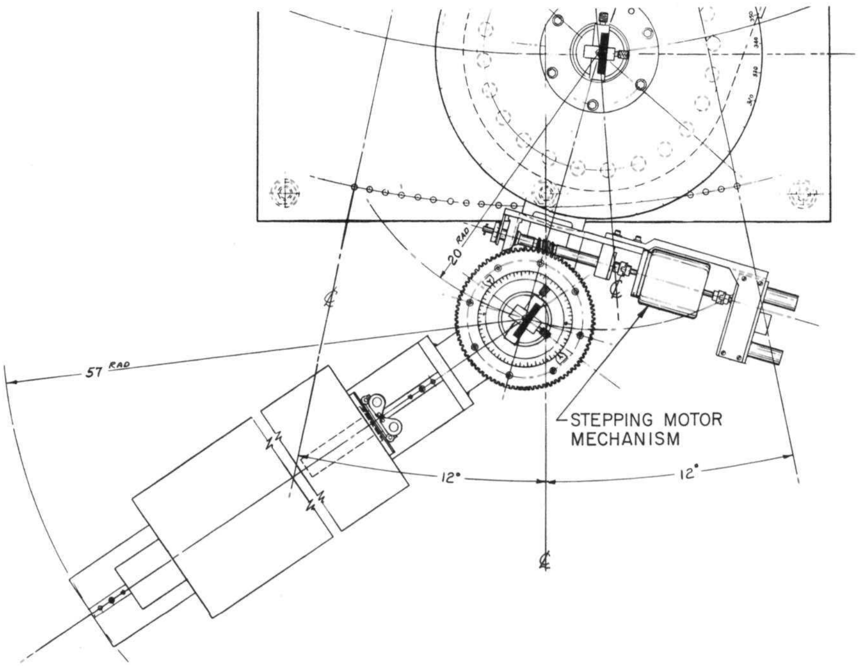

Figure 6. Top View of Spectrometer II Showing $\Delta\lambda$ Mechanism.

The details of the drive motion can be seen in Figure 6. The drive assemblies for both α and 2α motions are identical. A bracket arrangement was constructed for holding these drives in the proper plane associated with the two gears. This drive consists of the mounted worm shaft with one end attached to a 250-oz-in. Slo-Syn stepping motor, described above, with the additional feature that the motor shaft extends completely through the motor. The opposite end of the motor is then attached to a Coleman five-place digitizer which provides an electronic means of reading the angle to hundredths of a degree (xxx.xx). At the opposite end of the worm shaft, a drum calibrated in 100 divisions is attached, together with a means of coupling a hand crank for manual motion. Since 360-tooth gears were used, one revolution of the worm shaft corresponds to one degree; therefore, it is easy to indicate mechanically the angle by the drum, and an indicator pointer, was associated with the gear, which has a 360° divided circle attached to its face. It is quite evident from Figure 6 that this drive assembly is a compact unit and can be easily taken off the instrument by removing a hinge pin on the bracket supporting this drive assembly. Both the α and 2α drive assemblies are identical, except the α motor receives one pulse for each two pulses received by the 2α motor. This is accomplished by an electronic pulse divider; therefore, the 2α motion should always be stepped in even hundredths of a degree.

In order to provide additional collimation for use with the $\Delta\lambda$ mechanism, alternate collimators were constructed. One such defining collimator (CS-2397-17) was made to replace the previously described horizontal collimator, D-212. This collimator, which defines the beam between the monochromating crystal and sample, contains a rectangular opening, approximately 2 in. high by $1\frac{3}{4}$ in. wide, extending the full 28-in. length of the collimator. This opening is filled with cadmium-plated rectangular steel tubes $\frac{1}{8}$ in. x $\frac{1}{2}$ in., arranged in a checkerboard fashion as described previously for the counter collimator (CS-2397-8). Provision has been made for changing the beam size by suitable cadmium masks and also for mounting the monitor counter on the front face of this assembly.

Another Soller collimator (CS-2397-18) is used to define the diffracted beam. It contains an opening, approximately 2 in. x $1\frac{3}{4}$ in. x 12 in. long, filled with rectangular steel tubes, as described in the previous paragraph. This collimator is supported by a platform which is attached to a bracket which is a part of the $\Delta\lambda$ drive. This collimator may be adjusted for radial distance by means of this platform and is a collimator for the $\Delta\lambda$ mechanism as well as for high resolution when operating the instrument as a diffractometer.

In order to support the sample and analyzing crystal for the $\Delta\lambda$ mechanism, two goniometers together with suitable supports were designed and constructed. The goniometers (CS-2397-12) were scaled up by a factor of two from typical x-ray diffraction goniometers which have two arcs and an x-y translation. The goniometer used to hold the specimen for the $\Delta\lambda$ studies is supported at the center of the turntable by the extension column and platform previously described (CS-2397-12). A similar goniometer is used to support the analyzing crystal at the $\Delta\lambda$ pivot point. The goniometer rests on a platform or nest which is the same as previously described except that there is no worm-gear rotation provided. Various frame-type supports may be used to hold the analyzing and sample crystals on the goniometer heads.

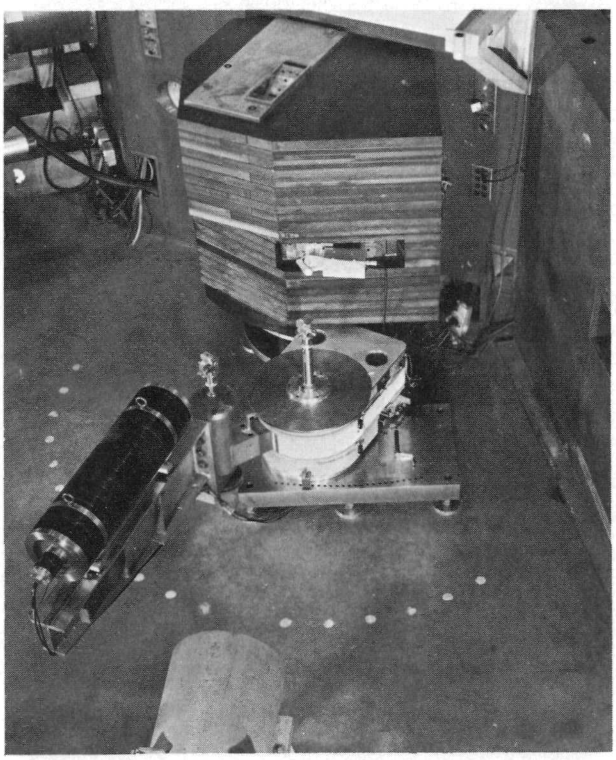

Figure 7. Photo of Spectrometer II - Top View.

Figure 7 shows an overall view of Spectrometer II illustrating the $\Delta\lambda$ pivot and goniometers, and Figure 8 shows this mechanism as pinned.

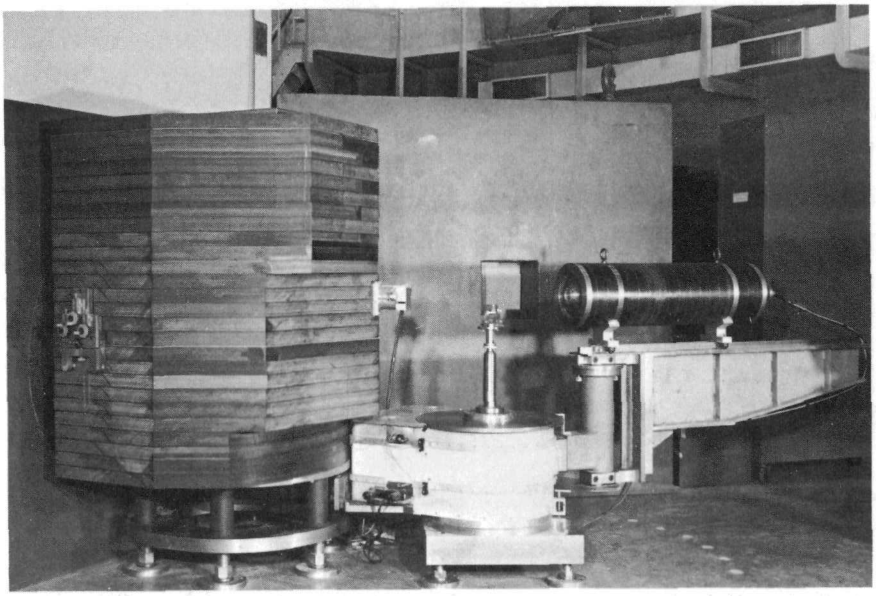

Figure 8. Photo of Spectrometer II - Side View.

Alignment Procedure

The initial stepwise alignment of this horizontal diffraction spectrometer was carried out according to the drawings during the assembly at the CP-5 reactor. In this section we shall describe briefly the operational procedure of alignment for this instrument. It is necessary that the sample, the detector, and the beam from the monochromator lie in the same plane, and that the incident beam be bisected by the sample. This is accomplished by the following stepwise procedure:

1. After choosing the proper take-off angle for the wavelength selected, a reasonably good preliminary alignment of the monochromator crystal can be obtained by observing the beam intensity on the fission counter rate meter. When the various external adjustments of the monochromator crystal have been carried out to achieve a maximum intensity, the next alignment steps can be carried out.

2. In order to establish that the beam is parallel to the floor, it has been found convenient to use a portable BF_3 counter which is shielded with a cadmium cover except for a long, 0.5 mm-wide slit extending along the length of the counter. A beam stop, some 15 ft from the reactor face, has a reference mark which is at the same height from the floor as the center of the beam hole, namely, 42 in. The portable BF_3 counter is placed at this reference mark, and the cradle motion of the monochromator holder is adjusted until the maximum beam intensity occurs at this reference point, thus assuring a horizontal beam.

3. A $\frac{7}{16}$-in.-diameter nickel powder sample is now held in the specimen mount at the center of the turntable in order to determine if the incident beam is bisected by this sample. The turntable is moved by the indexing device adjustment until the sample splits the beam. The adjustment necessary is checked with the portable BF_3 counter with the slit arrangement as described above.

4. A final check on the above sample alignment may be made with a thermal-neutron camera[6] which is available from National Radiac, Inc. (Type TNCP). This radiograph also serves as a good indication of the beam uniformity.

5. With the instrument aligned, it is now necessary to determine the true instrument zero and then to synchronize it with the mechanical and electronic angle read out. The peak positions for corresponding nickel reflections on either side of zero are determined, and the instrument zero is derived. The 2θ arm of the instrument is then set at this position, and both the mechanical and electronic angle read outs are set at zero.

6. A complete powder pattern of the nickel sample is next obtained for use in determining the wavelength λ of the monochromatic neutron beam. A least-squares technique may be used for obtaining the best value.

NEUTRON DIFFRACTOMETER I - (CS-2604)

We have chosen to call the top vertical instrument a diffractometer, since it is to be used primarily for measuring diffracted intensities and not for analyzing the spectrum as can be done with Neutron Diffraction Spectrometer II. The method for obtaining the monochromatic beam for this vertical instrument was discussed in a previous section. This included the monochromator, collimator, and fission counter. It was previously indicated that provision was made to change the take-off angle and hence the wavelength for this instrument.

Since it was originally planned to use this instrument primarily for single-crystal investigations, massive instrumentation as in Spectrometer II should not be required. Therefore, a commercial instrument (Picker Bi-plane Diffractometer) was adapted for the basic table. It provided a guaranteed load-carrying capacity, large 2θ range in both positive and negative direction, and ability to move θ independently of 2θ. The mounting of the Picker unit together with the additions made to the unit, including the detector arm, detector, detector shielding, and single-crystal goniostat, are shown in Figure 9. A complete description of the entire assembly is given in the following paragraphs.

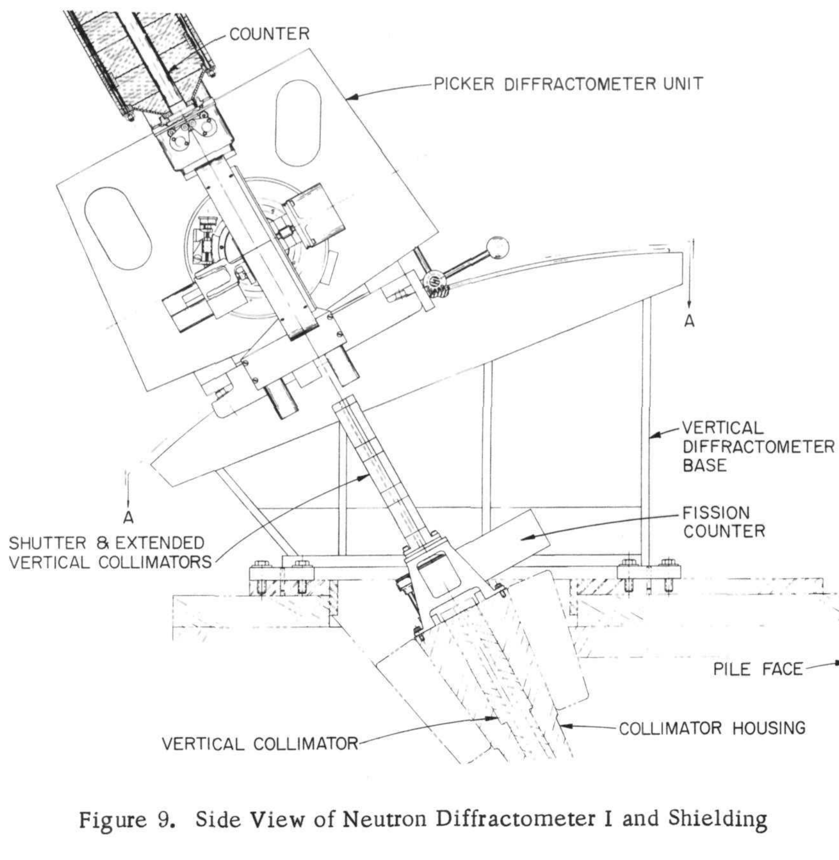

Figure 9. Side View of Neutron Diffractometer I and Shielding

Base Support

It was first necessary to design a support for the Picker unit, which would hold it rigidly in the neutron beam. This consists of several parts, namely, the base, a movable L-shaped angle bracket, and hub.

The base is bolted to the top plate, C-19 (see Figure 1), of the shielding framework. This base is a web construction of steel plates supporting an arc track which permits the change in take-off angle (see Figures 9 and 10). There are holes spaced 1° apart on this track for holding down the L-shaped bracket which serves as the mount for the Picker unit. The upright part of the bracket, just barely visible in Figure 10, contains two 2-in.-diameter holes. One hole is used for supporting the Picker unit on a column. This column has a flange and stub at one end for attaching to the side of the Picker unit, and the other end, threaded, is inserted through the hole in the L-bracket and held by a large nut. The other 2-in.-diameter hole in the L-bracket is currently being used for the support of the beam stop although as originally designed it was intended to support the Picker unit further from the shielding and thus permit the detector arm to travel to greater 2θ angles. Two aluminum wedges located between the top of the L-bracket and the case of the Picker unit prevent the rotation of this unit about its own 2θ axis.

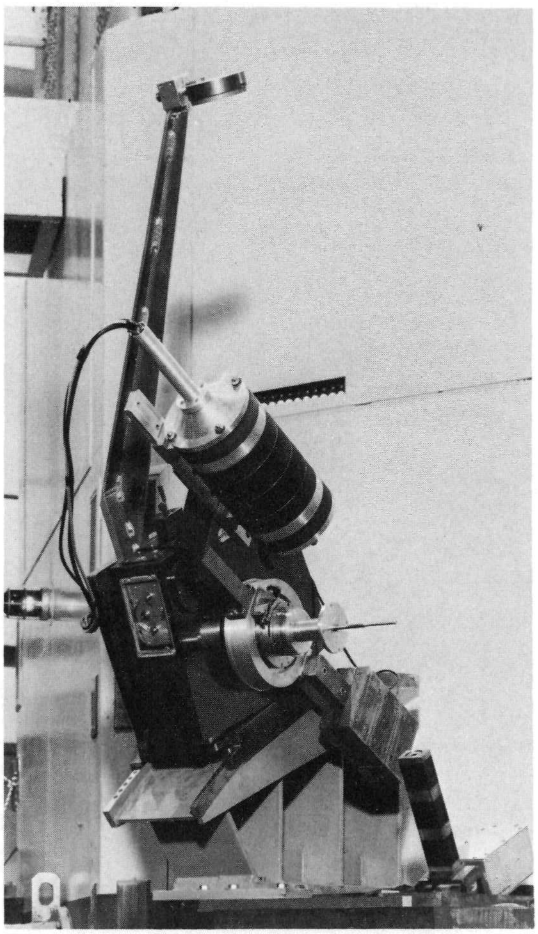

Figure 10. Photo of Diffractometer I as a Powder Unit

Beam Stop

As indicated in the previous section, the top 2-in.-diameter hole in the L-bracket is used for supporting a beam stop. It consists of a welded aluminum arm of T-shaped cross section with a cup on the top (see Figure 10). This cup is made of steel with a lead and cadmium lining, and provides adequate absorption for the monochromatic beam of thermal neutrons.

Detector and Arm

In order to support the neutron detector on the Picker unit as mounted vertically, it was necessary to use the following parts: the base ring with arms for the BF_3 detector and for the counterweight. The base ring as supplied had two flats on opposite sides, suitable for bolting on both arms, as shown in Figure 10. As noted, the arm has a built-in offset for holding the detector at the proper level. A standard type American Crystallographic Association instrument track has been used for holding the detector and thus providing an easy means of moving the detector with respect to the specimen. The counterweight arm is merely a short arm for holding some large steel blocks whose position can be adjusted by sliding back and forth and then clamping in position by tightening bolts.

The detector housing is practically the same as that used for the horizontal Spectrometer II except that the size is smaller. In order to lighten the load, some of the frame is made of aluminum; otherwise the Benelex rings, cadmium sheets and cadmium tube surrounding the detector are the same as for the larger housing except for size. It is believed that the detector counter housing could be lightened considerably by removing some of the shielding.

Shutter Assembly

A quadrant shutter or collimator door assembly has also been designed (CS-2604-4) for the front of this detector in order to make it easier to center the diffracted beam. This door assembly is a scaled-down model of the one used for Spectrometer II, which was described before.

BF_3 Detector Tube

The BF_3 detector tube currently being used is a Reuter Stokes (Type RSN-1125) with a 1-in. diameter, approximately 6-in. active length, 96% enriched B^{10}, 100-cm pressure, nickel end window, and an operating voltage of approximately 2500 v. The preamp is of the same type as used for Spectrometer II.

ω-2θ Drive Arrangement

The Picker unit was purchased with a continuous-drive motor for the 2θ motion; however, no motor was supplied for the ω drive. Since magnetic clutches are used normally for coupling these motors to their respective drives, it was an easy matter to uncouple the motor from the 2θ drive, merely by not supplying power to the clutch. This arrangement was desirable since it was intended to add a stepping Slo-Syn motor to the 2θ motion. It can be noted in Figure 10 that there are a number of stub shafts available on one side of the Picker unit. Then in Figure 11 it can be seen how this stepping motor (of 250-oz-in. torque) was coupled onto the 2θ drive. It can be noted in this same figure that there is a Coleman digitizer coupled to a through shaft of the motor. At the opposite side of

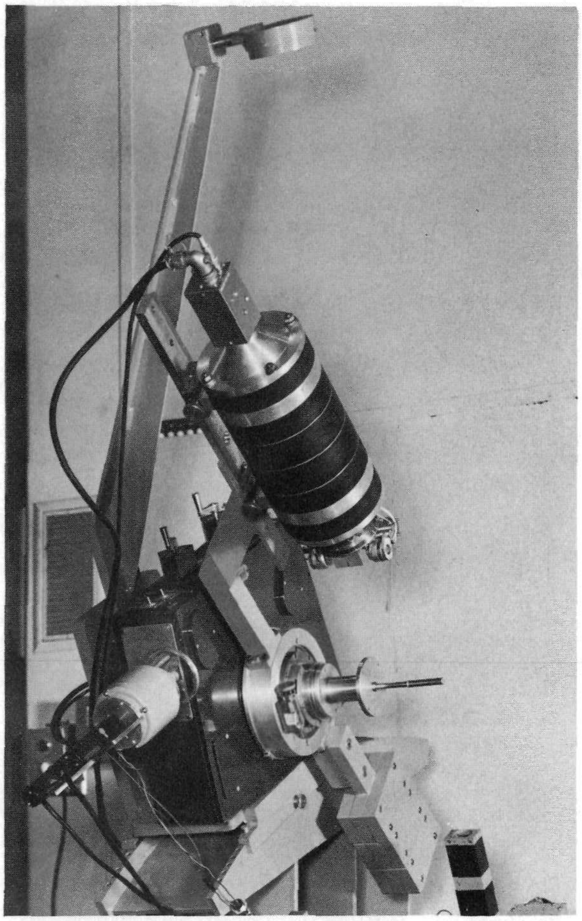

Figure 11. Photo of Diffractometer I Showing Stepping Motor and Digitizer Arrangement

the Picker, there is another 2θ shaft available which was used for connecting to a 40-turn potentiometer which couples the 2θ arm motion with the x-y recorder. This is the same arrangement as used for the lower instrument (Spectrometer II).

It is fully anticipated that in the future it will be desirable to be able to drive the ω motion independently from 2θ, such as is necessary for a fixed-counter movable-crystal technique for gathering single-crystal intensity data. Shafts are available on the Picker unit which can be used for attaching another stepping motor in order to use this technique.

Mechanical indicator read-out devices were also purchased as a part of the Picker unit. These are coupled to 45° take-off shafts from both 2θ and ω, and are mounted on the top side of the Picker unit. The handle of the 2θ indicator is just visible in Figure 10. These indicators serve a very useful purpose at present since it is possible to move the ω a known amount with respect to 2θ, and also a check can occasionally be made to see that no slippage of the couplings has occurred between the 2θ indicator and digitizer.

Powder Sample Mount

When the original design was planned for Diffractometer I, it was not certain, as indicated above, what type of commercially available diffractometer would be used. Therefore, in planning for the single crystal holder motions, a base was designed providing a limited ω manual adjustment which could be read to a hundredth of a degree on a drum (see Figure 10). Since this base was designed as a separate but complete assembly, it was convenient to make the necessary adapters for supporting powder samples. A spool-shaped piece of aluminum was made whose table surface was 4 in. from the center of the neutron beam. This made it convenient to attach X-ray diffraction auxiliary pieces which already required a 4-in. beam-to-base height, such as the G. E. single-crystal orienter. It is anticipated that this would be useful in initially aligning a crystal with X-rays and then transferring to the neutron instrument. It has also been found convenient to machine a simple base for supporting this spool without the ω adjustment. The base consists of a flange with bolt holes and a hub (see CS-2604-2 for necessary dimensions). This allows the more complex ω-adjustable base to remain attached to the goniostat described later; however, this ω-adjustable base has been found useful for other instrumental arrangements.

In order to support the ordinary powder samples, a $\frac{1}{2}$-in.-diameter hole was provided in the center of this spool-shaped table. A $\frac{1}{2}$-in.-diameter rod was then made to fit into this hole; the position of the rod in and out could be governed by a small collar with set-screws. The sample end of this rod was provided with a 5-40 threaded pin which could be screwed into the usual base end of the sample tubes. These sample tubes usually consist of vanadium tubing, $\frac{1}{4}$ in. to $\frac{7}{16}$ in. in diameter, 5-mil wall thickness, several inches in length, with cadmium end plugs.

It was also found that this spool-shaped table was convenient for alignment. A $\frac{1}{2}$-in. rod with a machined point was inserted in this $\frac{1}{2}$-in.-diameter hole. Then another pointed rod was inserted in the hole in the collimator extension. This provided a reasonable means for insuring that the beam and sample axes coincided.

Goniostat

A number of built-in design features for a goniostat which are desirable are: 360° phi (ϕ) motion, 360° chi (χ) motion, an accuracy of setting these two angles (especially the ϕ motion) to at least a hundredth of a degree, digitizer coupling and read-out for each angle, ability to program automatically the angle settings, and minimum blanked-out areas due to component parts getting in the way of the neutron beam - either diffracted or primary. The goniostat (CS-2604-2) consists of a base, a circle incorporating both ϕ and χ motions, and associated digitizers and motor drives.

The base, described in the previous section, for this goniostat is not a necessity when used with a diffractometer which has a built-in independent ω motion; however, it is a convenience even in this case. The χ ring, $12\frac{1}{2}$ in. in OD and 2 in. thick, has been constructed of a nonrustable material, light in weight yet rigid and durable. Most of the exposed parts are of an aluminum alloy.

The construction and basic operation of the instrument can be understood from Figures 12 and 13. The large ring consists of two circles, an outer, rigid one and an inner, movable circle to which the goniometer is attached. The rigid circle consists of a radial brace, C-69, and the bearing housings, C-82 and C-83. The inner movable ring consists of a 12-in.-OD

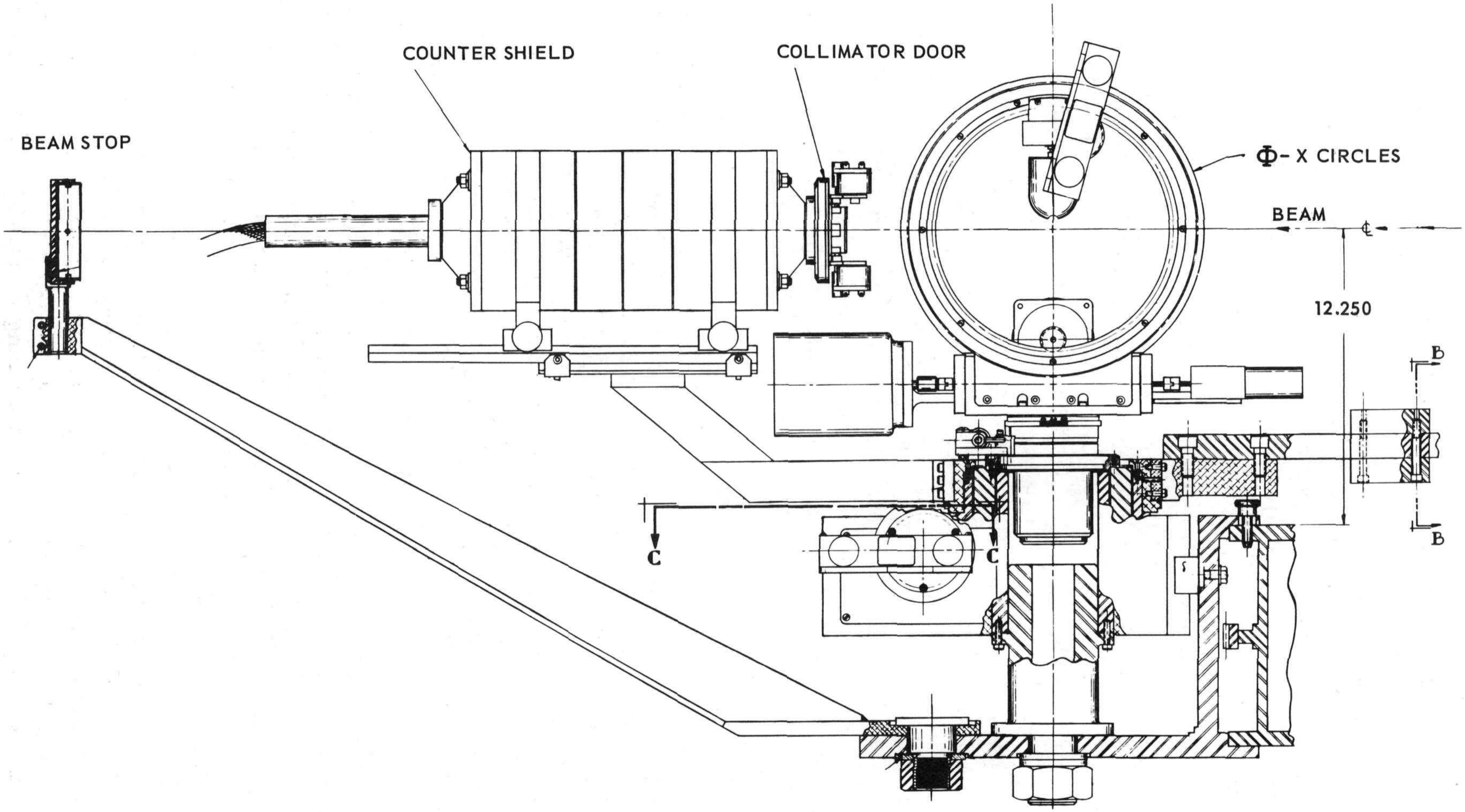

Figure 12. Random Section View of Neutron Diffractometer I with Goniostat

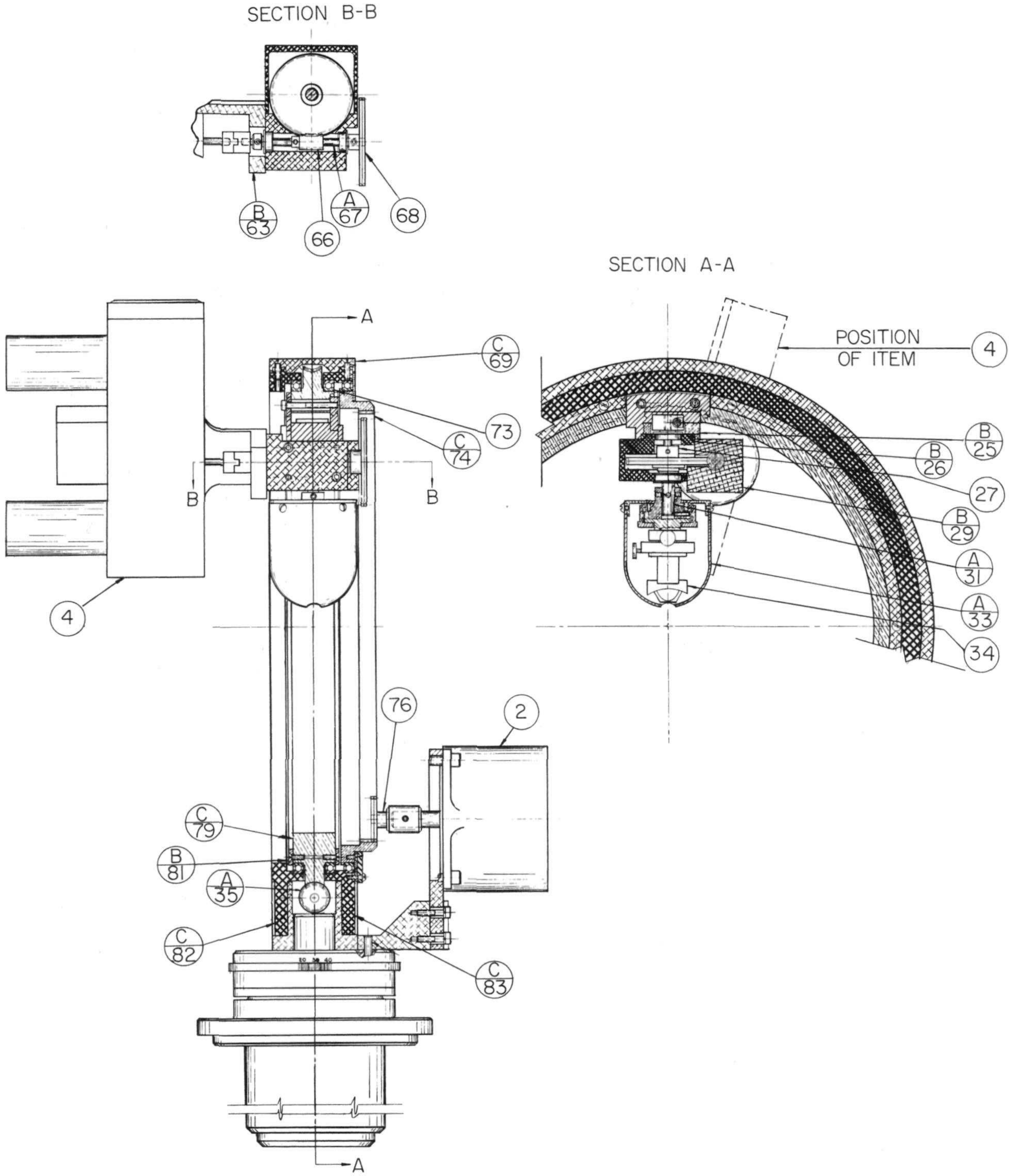

Figure 13. Cross-sectional View of Goniostat

worm gear, C-79, and χ bearing retainer, B-81, supported within the outer rigid circle by two Kaydon ball bearings (KA-110-CP $11\frac{1}{2}$-in. OD Item No. 73). By moving this inner ring, the χ angle is changed. This motion is accomplished by a worm gear, A-35, and drive located at the base of the ring, as shown in Figure 13. Although not visible in this drawing, the end of the worm shaft towards the viewer is attached to a Coleman digitizer (Model AP5 DUT19, 5-place 100 c/rev), and the other end is attached to a Slo-Syn stepping motor (Type SS, 150-oz-in. torque). Since the gear for this motion has 360 teeth, it was possible to again standardize on one revolution of the worm shaft corresponding to one degree.

The ϕ angle motion is also accomplished by a worm gear and anti-backlash worm wheel, Items 66 and 27 (Pic Q8-2 and Q14-45, respectively) located in the base of the goniometer, Section BB, which is a part of the inner ring. This base consists of the ϕ mounting bracket, B-25, the ϕ worm wheel housing, B-26, and the ϕ worm housing B-29. This gear, 27, is attached to the ϕ spindle, A-31, which upon rotation generates the ϕ motion. As can be noted in Figure 13, a ϕ worm shaft, A-67, extends through the ϕ worm housing, B-29, at one end of which is attached a gear, Item 68 (Dynaco antibacklash spur gear AB-100-96/P2) and the other end drives a Coleman digitizer, Item 4, (Model AP5 DJ, 5-place, 400 c/rev) for indicating this angle. This digitizer is attached by a bracket, B-63, to the base of the goniometer and therefore rides around the ring with the change of the ϕ motion. It can be noted in this figure that the gear, 68, on this ϕ worm shaft, A-67, engages in a large-ID, 10-in. ϕ internal spur gear, C-74, visible in Figure 13. This large idling spur gear is not attached to the inner ring and can move freely on a bearing, Item 73, (KA-110-CP, $11\frac{1}{2}$-in. OD) with respect to the rigid outer ring. This large-ID gear is moved by the spur gear, 76, on the stepping motor which is rigidly attached to the circle base and provides a means of transmitting motion to the spur gear, 68, regardless of the χ position. Although the original gear ratios provided a movement of 0.02° per DC pulse on the stepping motor, this has since been changed by using a smaller gear ($\frac{1}{2}$ the number of teeth - Dynaco Spur gear 105-24-P2, 24 teeth) on the ϕ angle stepping motor. The spur gears, 68 and 76, do not follow the same track on the large interval spur gear, C-74; hence, the 360° motion of ϕ is possible without restrictions.

Although stepping motors do impart a slightly jerky motion which may affect delicately mounted crystals, no difficulty is anticipated under normal operating conditions. In the more delicate cases it is perhaps better to substitute small DC motors which are commercially available and lend themselves to accurate positioning and also automatic programming. Figures 14 and 15 are photos of the goniostat attached to the omega adjustable base, which in turn is bolted to a large aluminum storage base.

The cover, A-33, surrounding the goniometer ($\frac{1}{2}$ attached to the goniometer and $\frac{1}{2}$ lying on the base in Figure 15) is formed from 0.020-in.-thick cadmium sheet. It is for the purpose of eliminating any diffracted intensity arising from any part of the goniometer in those cases when the beam is rather large.

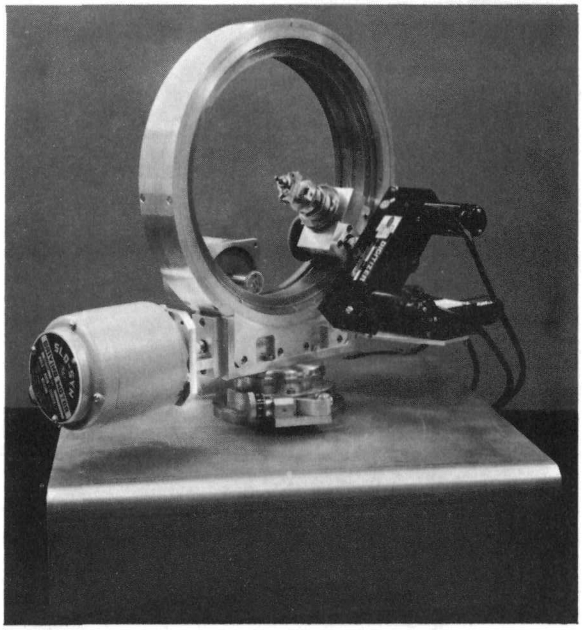

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Figure 14. Photo of Goniostat

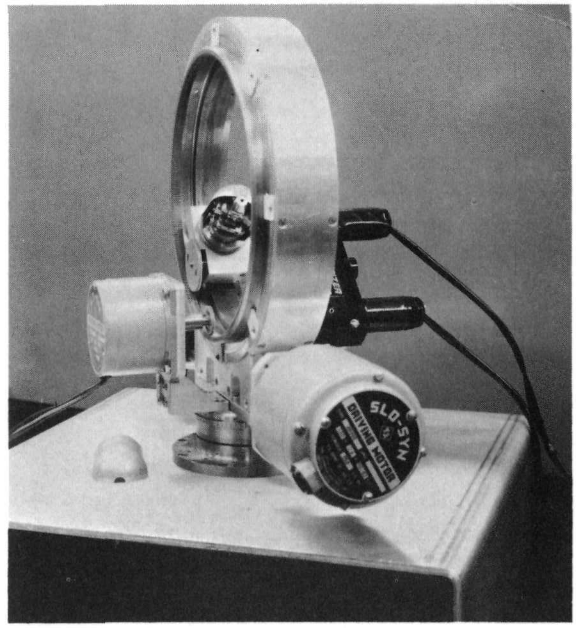

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Figure 15. Photo of Goniostat

This goniostat has been made to accommodate both the old-style goniometer head, 34, as well as the eucentric head which must be used with a commercially available goniometer adapter head in order to bring it to the proper position. Approximately $\frac{1}{8}$-in.-elevation adjustment of the goniometers is possible by sliding the goniometer adapter, A-14, up and down on the ϕ spindle, A-31, and locking it by two 5-40 set screws.

Figure 16 is a photo of the goniostat on Diffractometer I, and Figure 17 is a photo showing both neutron instruments, Spectrometer II and Diffractometer I, as installed at the CP-5 reactor.

The design and construction of this instrument were carried out with the idea of built-in accuracy rather than of providing for a number of adjustments to be made later. For example, it was specified in the construction drawings that the axis of the ω adjustment and the axis of the χ motion be perpendicular, and that the ϕ motion lie in the χ plane. All three axes were to intersect within 0.002 in. for all values of ω, χ, and ϕ.

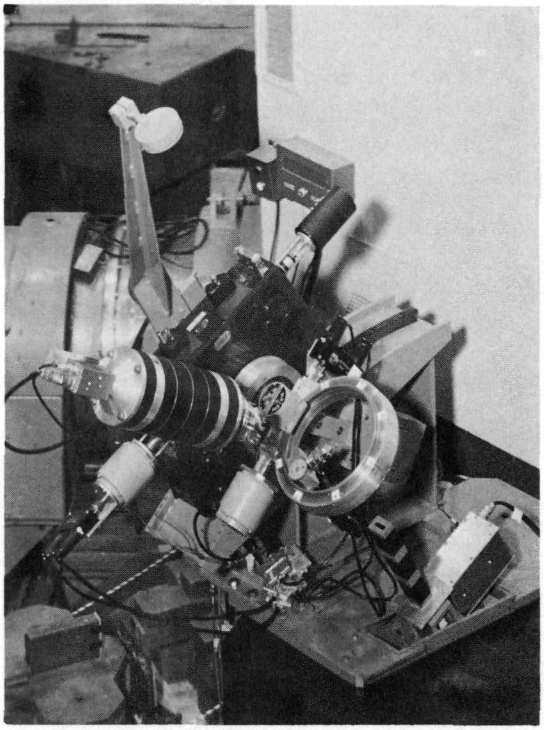

Figure 16. Photo of Diffractometer I with Goniostat

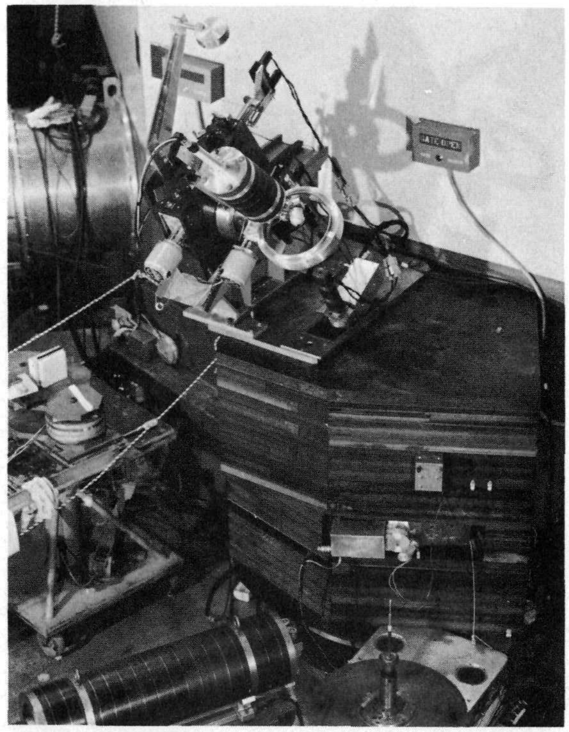

Figure 17. Photo of Dual Instrument at CP-5 Reactor

In order to check on the built-in accuracy of the instrument after assembly and for the construction of alignment gage blocks, it was submitted to the Inspection Department. A spherical steel ball, $\frac{1}{2}$ in. in diameter, was supported on an $\frac{1}{8}$-in. pin as a sample crystal on the goniometer. Dial indicators were then used to determine the displacement of the ball over the complete ϕ and χ motions of 360°. It was found that the ball remained stationary within ±0.001 in. with both goniometer arcs set at 0^0. The goniostat was then mounted on the Picker unit as assembled at CP-5, and the ω motion was used to rotate the ring. The indicator on the ball again showed no greater deviation than ±0.001 in. Consequently, a gage block for accurate location of the intersection of the three above-named axes, which is the correct position for the sample, was constructed.

Since considerable variation of the location of the focal point has been observed in the ordinary, commercially available goniometer heads, it is necessary to check out each one and construct the proper gage block for locating the goniometer head-height adjustment. This was accomplished by moving each arc of the goniometer its full ±20° travel and locating the height so that the focal point of the arcs lies within ±0.001 in. of the intersection of the three axes. Suitable gage blocks, such as shown in Figure 18, were then constructed for the two goniometer heads currently in use - one the old style and one eucentric type. This latter test and the following tests were performed with the goniostat in the Inspection Department with the instrument removed from the Picker unit.

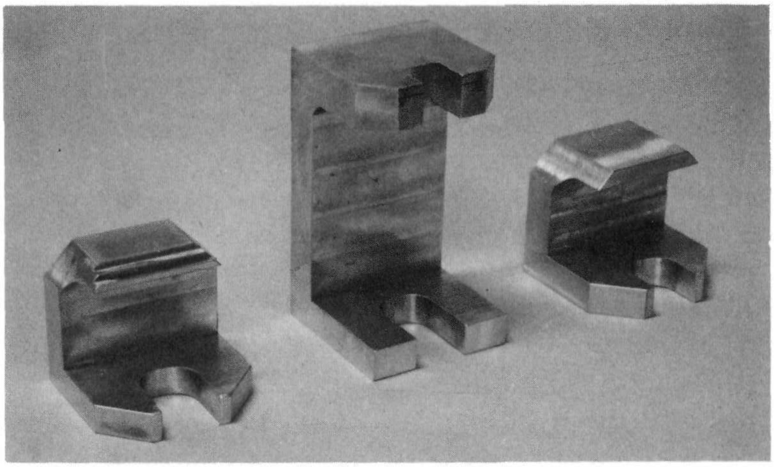

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Figure 18. Photo of Gage Blocks Used for Aligning Crystal and Goniometers on Goniostat

In order to determine the accuracy of the ϕ settings, a 30° optical polygon was positioned accurately in place of the goniometer head in order to check the location every 30° about the ϕ axis. The ϕ axis was rotated in both directions with the stepping motor drive. The coincidence of the optical 30° positions and the digitizer read out was then noted. It was found that there was agreement within ±0.01° in all cases.

A check was also made to insure that the instrument axis (ω-2θ) and the ϕ axis lie in the χ plane. This was carried out in the following way: The goniostat together with its ω base assembly was positioned on its side (the usual position on Diffractometer I) with the cylindrical part of the base supported in a V block. For this check, the large $\frac{5}{8}$-in.-diameter post previously used for supporting the optical polygon was carefully checked for eccentricity about the ϕ axis. This post was then indicated along its $1\frac{3}{8}$-in. length for being parallel to the Inspection Department plate used for performing this check. It was found to be parallel to within 0.0005 in. The entire orienter was then flipped over 180° and the same indicator check made. Readings now checked within 0.0015 in. of the previous parallel readings.

Alignment Procedure

For alignment purposes, Diffractometer I was assembled as a powder unit, as shown in Figure 10. It was required that the beam, the sample, and detector arc lie on the same plane, and the axis of the instrument be perpendicular to this plane and bisect the incident beam.

It was found necessary in the initial assembly to add a special plate in order to tie the top plate, C-19 of Figure 1, to the vertical angle-selector housing (CS-2397-3) to maintain the instrument axis perpendicular and rigid with respect to the scattering plane. This also maintains the reference distance of 12.250 in. between the frame (CS-2604-1) and the centerline of the beam, as shown in Figure 12. The following operational alignment procedure was carried out:

1. A straight edge extended from the collimator extension (CS-2604-5) was used in order to establish the correct position for the beam stop, (see Figure 12) along its post (CS-2604). The portable BF_3 detector described in the alignment procedure for Spectrometer II was used to establish the beam location with respect to the beam stop. Necessary adjustments were made on the monochromating crystal by rotating the external controls to center the beam with respect to this stop thus assuring that the beam lies in the scattering plane.

2. To determine if the axis of the instrument bisects the incident beam, it was found convenient to use alignment tools. These alignment tools consist of two pointed rods - one for insertion in place of a powder sample and one into the hole of the collimator extension. As these rods are moved along their respective axes, the points should meet, indicating alignment. Since there is no provision for fine adjustment on the angle position of the frame supporting the diffractometer, it was necessary to change the take-off angle of the collimator slightly. This was accomplished by inserting shims between the pie-shaped sections until the beam was bisected by the sample. It would be a convenience, although not a necessity, to add a fine angular adjustment to the frame supporting the diffractometer, similar to that on Spectrometer II.

3. As a further check on this alignment, a $\frac{1}{4}$-in. diameter nickel powder sample was inserted in position, as shown in Figure 10. A portable BF_3 counter was then used to determine the location of the beam with respect to the sample.

4. A final check on the above alignment, as well as beam uniformity, was also made with a TNCP Thermal Neutron Camera as described in the previous discussion of Spectrometer II. In the alignment of either instrument it is necessary to repeat some of the previous checks in order to gradually attain the best alignment.

5. This next step involves the same procedure as explained for Spectrometer II under step 5, which establishes the true instrument zero and the synchronization of the mechanical and electronic angle read out.

6. This step is also the same as step 6 for Spectrometer II, which establishes the wavelength λ of the monochromatic neutron beam.

7. In those cases where it is important to have the specimen, such as a single crystal, well aligned, considerable assistance in alignment can be obtained by using the collimator shutters. These shutters, as explained previously, are provided for both instruments and divide the front of the counter tube into quadrants, and make it possible to check the intensity appearing in the top, bottom, left, and right quadrants.

Electronics

The basic description of the electronic circuitry used for both of these instruments has been described by Johanson.(7) In Figure 19 we have shown a block diagram of the system. Figure 20 is a photo of electronic console as used for both instruments.

The electronic controls consist essentially of two counter systems, two data-recording systems, and a programmer which controls the sequence of events. The two counter systems consist of a BF_3 detector and a fission monitor together with their associated pre-amplifiers, linear amplifiers, and scalers. One data-recording system is digital, recording the detector scaler counts and the angle encoder readings on an IBM typewriter, and the other system is analog, recording the scaler and angle on an x-y recorder - the x voltage corresponding to the angle and y voltage corresponding to the detector scaler.

In normal operation (refer to Figure 19) with the 2θ arm in a fixed angular position, both the detector and monitor feed counts to their

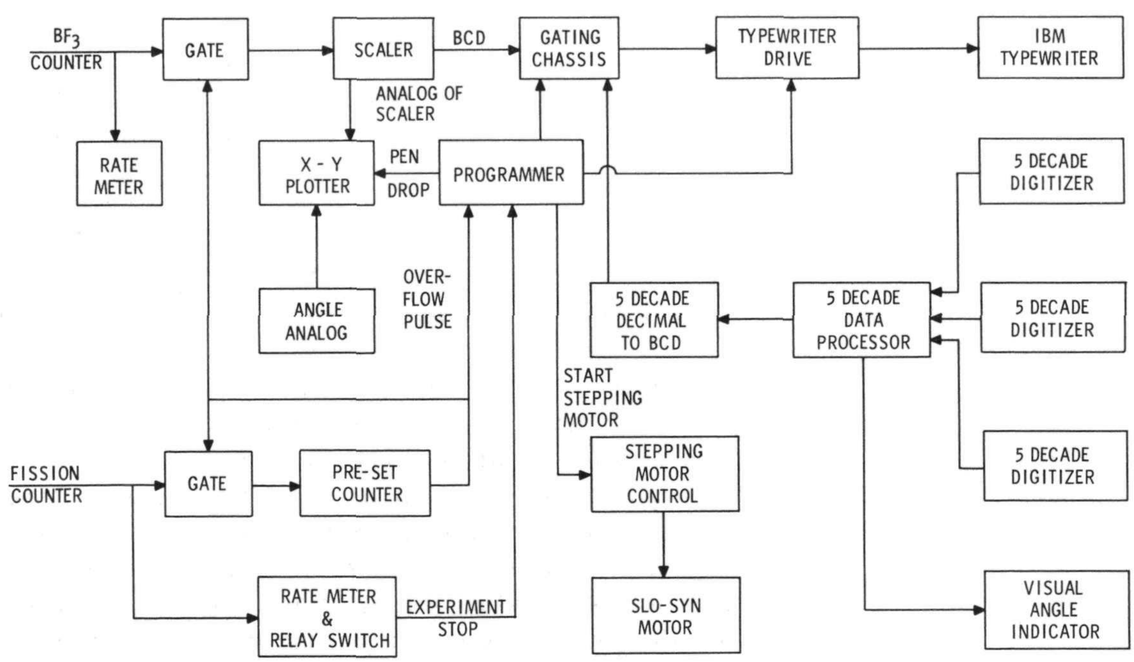

Figure 19. Block Diagram of Electronic Controls

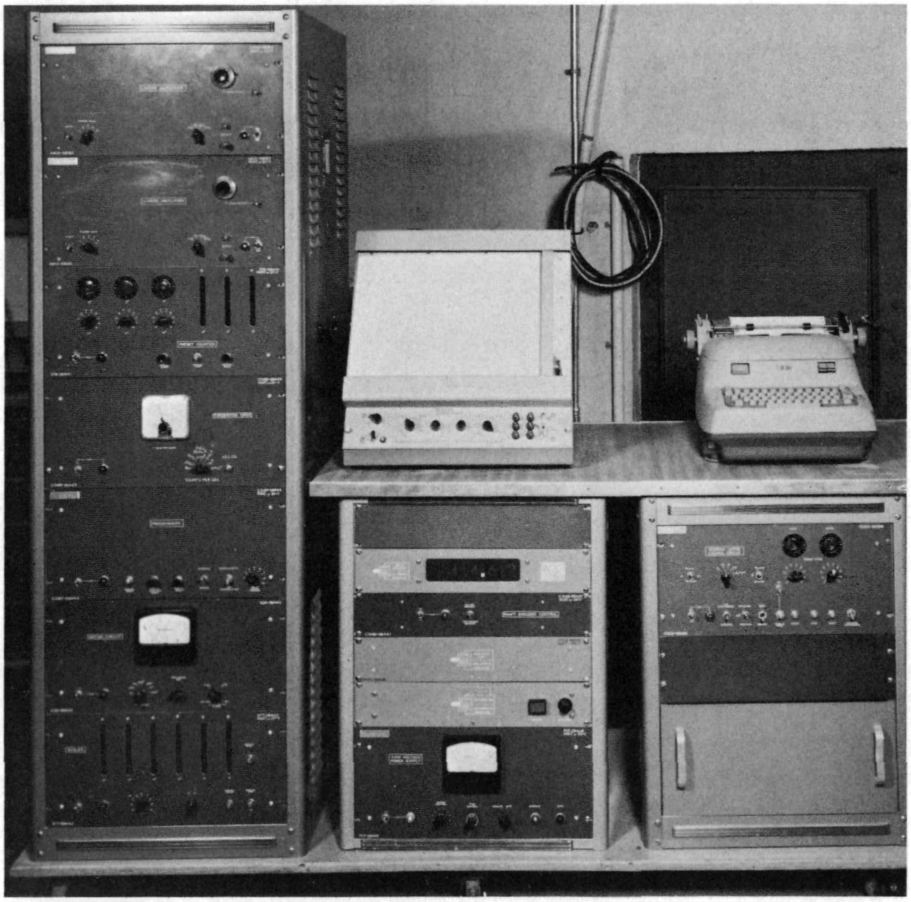

Figure 20. Photo of Electronic Console

respective scalers. At the end of a preset number of monitor counts, an output pulse is sent to the programmer and gates between the linear amplifier and scalers in the two counting systems. The programmer sends a pulse which engages the angle encoder and activates the gating chassis and the typewriter drive. In turn, the scaler counts pass through the gating chassis to the typewriter drive which records them on the IBM typewriter. The gating chassis is next switched to the angle encoder, and the angle is printed out. Then a pulse is sent to the pen-drop control on the x-y recorder which records a point on the curve of intensity versus scattering angle. The programmer then sends a pulse to the 2θ stepping-motor control which moves the 2θ arm to its new position. The programmer then resets the two scalers, opens the gates, and the cycle is repeated at the new angular position. This continues until the arm hits a stop switch which cuts off the monitor count or may be set to start reversing the 2θ arm. The monitor counting rate is visible on a rate meter. If the monitor count falls below a set value, either because of a reactor shut down or the stop switch described above, the rate meter switch sends a signal to the programmer which stops its operation. When the counting rate once again rises above the set value, the block is removed from the programmer and the cycle continues.

A sample of the output data as presently arranged is shown in Figure 21, which is the x-y recorder plot of the neutron diffraction data obtained from a nickel powder sample by means of Diffractometer I. Figure 22 shows a section from the IBM typewriter output for this same pattern.

All of the electronic circuitry has been designed with the intention of adding additional electronic equipment to provide automatic three-dimensional data collection. This can be accomplished with a very minimum modification of existing components and will make use of prepunched-card input for setting up four different angles and card output for angle verification and collection of intensity data. It is planned to add this to the goniostat on Diffractometer I; however, a similar system may be added in the future to the $\Delta\lambda$ arrangement for Spectrometer II.

C-40 February 15, 1962 Nickel Sample
Monitor Count = 121K/minute
Preset = 100K Full Scale = $10 \times 10^4 \times .1$

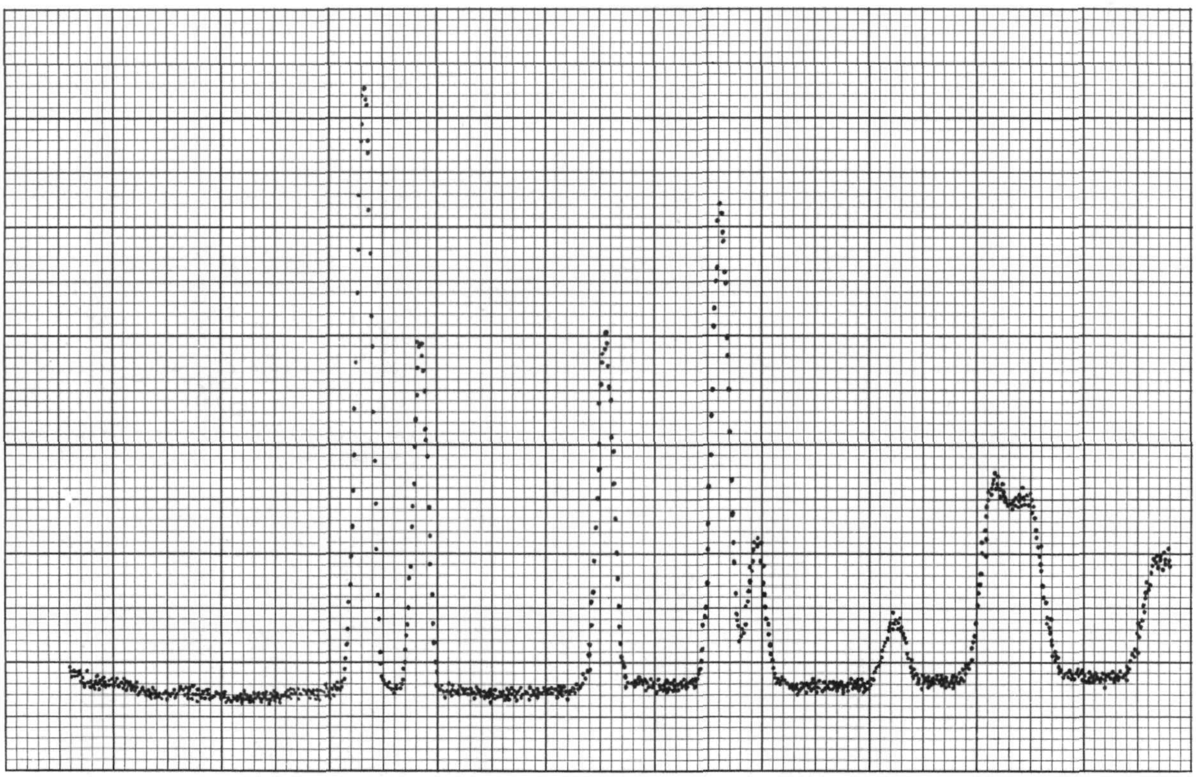

Figure 21. x-y Recorder Plot of Diffraction Pattern of Nickel Sample Obtained on Diffractometer I

THIS IS A SECTION TAKEN FROM THE FOLLOWING PATTERN.

C-40 FEBRUARY 15, 1962 NICKEL SAMPLE
MONITOR COUNT = 121K/MINUTE
PRESET = 100K FULL SCALE = 10 x 10^4 x .1

I	2θ	I	2θ	I	2θ	I	2θ
000945	05430	001081	05440	001170	05450	001341	05460
001567	05470	001663	05480	001963	05490	002246	05500
002513	05510	002784	05520	003025	05530	003394	05540
003564	05550	003770	05560	003850	05570	003889	05580
004027	05590	003941	05600	003730	05610	003542	05620
003419	05630	003085	05640	002947	05650	002261	05660
002345	05670	002029	05680	001789	05690	001521	05700
001348	05710	001173	05720	001059	05730	001011	05740
000970	05750	000880	05760	000783	05770	000822	05780
000826	05790	000804	05800	000811	05810	000800	05820
000813	05830	000795	05840	000791	05850	000811	05860
000808	05870	000850	05880	000809	05890	000821	05900
000838	05910	000843	05920	000831	05930	000840	05940
000820	05950	000754	05960	000828	05970	000775	05980
000855	05990	000845	06000	000787	06010	000821	06020
000818	06030	000771	06040	000800	06050	000746	06060
000845	06070	000756	06080	000786	06090	000763	06100
000752	06110	000792	06120	000811	06130	000744	06140
000766	06150	000812	06160	000776	06170	000810	06180
000753	06190	000800	06200	000839	06210	000768	06220
000767	06230	000754	06240	000802	06250	000807	06260
000764	06270	000811	06280	000798	06290	000836	06300
000796	06310	000788	06320	000785	06330	000802	06340
000784	06350	000837	06360	000849	06370	000842	06380
000817	06390	000781	06400	000831	06410	000858	06420
000868	06430	000912	06440	000949	06450	001029	06460
001024	06470	001134	06480	001352	06490	001425	06500
001516	06510	001746	06520	002050	06530	002292	06540
002630	06550	002916	06560	003275	06570	003518	06580
004101	06590	004271	06600	004519	06610	004659	06620
005093	06630	005247	06640	005166	06650	005151	06660
004974	06670	004882	06680	004584	06690	004496	06700
004002	06710	003823	06720	003512	06730	003130	06740
002685	06750	002363	06760	002119	06770	001926	06780
001647	06790	001450	06800	001409	06810	001356	06820
001206	06830	001248	06840	001304	06850	001279	06860
001354	06870	001357	06880	001521	06890	001566	06900
001672	06910	001752	06920	001880	06930	001865	06940
001930	06950	002059	06960	002105	06970	002091	06980
002132	06990	002082	07000	002103	07010	001933	07020
001923	07030	001902	07040	001745	07050	001635	07060
001522	07070	001404	07080	001375	07090	001275	07100
001183	07110	001098	07120	001039	07130	000962	07140
000957	07150	000906	07160	000873	07170	000852	07180
000828	07190	000809	07200	000811	07210	000838	07220
000868	07230	000843	07240	000845	07250	000846	07260
000804	07270	000813	07280	000796	07290	000777	07300
000791	07310	000768	07320	000774	07330	000783	07340
000759	07350	000799	07360	000764	07370	000700	07380
000733	07390	000757	07400	000771	07410	000811	07420
000780	07430	000803	07440	000720	07450	000791	07460

Figure 22. IBM Typewriter Output Obtained for Nickel Pattern Shown in Figure 21

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