Volume I of II

X-RAY EQUIPMENT FOR LUNAR RECEIVING LABORATORY

Final Report (Part I)

by

N. Spielberg and G. Ziedins
April 1970

Prepared by

PHILIPS LABORATORIES
A Division of North American Philips Corp.
Briarcliff Manor, New York

Prepared for

NASA Manned Spacecraft Center
Lunar Receiving Laboratory
Houston, Texas

NASA CR 108466

X-RAY EQUIPMENT FOR LUNAR RECEIVING LABORATORY

Final Report (Part I)
September 1968 - April 1970

Contract No. NAS 9-8813

Prepared by

N. Spielberg and G. Ziedins

PHILIPS LABORATORIES
A Division of North American Philips Corp.
Briarcliff Manor, New York

Prepared for

NASA Manned Spacecraft Center
Lunar Receiving Laboratory
Houston, Texas

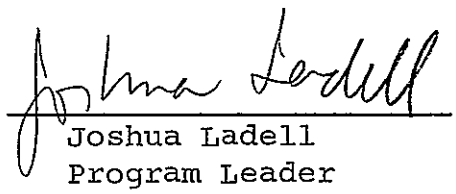
TABLE OF CONTENTS

LIST OF FIGURES

PREFACE

This report is the first part of an overall description and operating manual of the x-ray analytic instrumentation developed for the Lunar Receiving Laboratory. It deals primarily with the manually operable equipment delivered to LRL on September 15, 1969. This report has been prepared by Dr. Nathan Spielberg and Mr. Gunar Ziedins.

A second part in a separate report will be issued describing the computer interface, software, and automatic operating procedures.



Joshua Ladell
Program Leader

Philips Laboratories
Briarcliff Manor, New York
April 24, 1970

1. INTRODUCTION

1.1 Scope of Report

This report comprises a detailed description of the design, fabrication, installation and operation of an x-ray diffractometer and x-ray spectrograph system for preliminary examination of returned samples of lunar soil while these are still in quarantine within the Lunar Receiving Laboratory at the NASA Manned Spacecraft Center in Houston, Texas. The work was carried out under the provisions of NASA Contract No. NAS 9-8813 between MSC and Philips Laboratories.

The instrument heads are within the primary biological barrier at LRL and are operable by remote control from within the secondary biological barrier. The remote control may be accomplished by either a manual or computer control system. This report is concerned with the instruments themselves and the manual control system. The computer control system will be described in a separate report.

In addition to the information contained in this report, further detailed documentation of the system is contained in the sets of mechanical drawings, instruction manuals, and shipping lists delivered with the system. Some additional components, drawings and instructions were sent to MSC to permit carrying out necessary pre-installation preparation of the location of the system.

1.2 Genesis and Development of the System

During the course of NASA Contract No. NASW-1477 between NASA, Washington, D. C. and Philips Laboratories, and as a result of

discussions with personnel at the Manned Spacecraft Center, an extensive study was undertaken in December 1967 of the feasibility of developing x-ray diffraction and x-ray fluorescence analysis instrumentation for the analysis of returned lunar samples while they were still in biological quarantine in the Lunar Receiving Laboratory. This study is described in detail in the reports delivered under Contract No. NASW-1477 (Parrish, 1968). As originally envisioned, the computer control system was to be an integral part of the proposed system, and a proposal was submitted to NASA-MSC for the design and fabrication of the system. Due to various administrative considerations within NASA, it was necessary to excise the computer control system from the original proposal. A new proposal dated May 15, 1968, was submitted for a manually controlled instrument system to which a computer control could be later added. Finally contract NAS 9-8813 was awarded as of August 28, 1968, with delivery scheduled to be in time for the Apollo XI flight to the moon in July 1969. Unfortunately it was not possible to have the facilities at LRL ready to receive the x-ray instrumentation during the "operational window" in April of 1969, and thus it was necessary to postpone delivery of the system until a later date. It was possible, however, to install certain necessary feedthroughs and mounting flanges in the floors of the biological containment boxes (glove boxes) in which the instruments were to be installed.

The installation of the manual system was successfully carried out from September 15 to 19, 1969. The instruments were placed into operation, tests were made using sample materials from Apollo XI flight, and LRL personnel were instructed in the operation of the system. It was not possible to obtain release of

the test results obtained during installation, and these are not contained in this report.

On June 1, 1969, the original contract was amended to include the addition of the computer control system.

1.3 System Concepts

The instrumental concepts are discussed in detail in Section 2 below. It is desired to emphasize here that the x-ray diffractometer was considered to be the primary instrument of the system, and the design of other components was subordinated to this consideration. Thus the x-ray spectrograph was designed so as to accept specimens from the diffractometer without any further specimen preparation. This means that many of the sample preparation and calibration techniques often used for detailed quantitative elemental analysis will require the preparation of special specimen holders.

The control system has been designed in such a way that the computer control system now under construction can be added with little or no modification of the existing instruments.

1.4 Acknowledgements

The instrumental system described in this report was conceived by, and major design begun under the direction of Dr. William Parrish, now at the NASA Electronics Research Center in Cambridge, Massachusetts. The entire project has benefited in a major way from his detailed advice and cooperation in all aspects of the program from initial conception to final installation. Mechanical design construction and installation was carried out under the direction of Mr. Imre Vajda, now retired.

2. DESIGN CONSIDERATIONS

The following requirements were imposed on the general system by the operating conditions, specifications and time schedule at the Lunar Receiving Laboratory.

2.1 The diffractometer and spectrograph were to be installed within biological containment or glove boxes, and all electrical, water, gas, vacuum, etc., connections were to be made in such a way as to preserve the integrity of containment as verified by halogen leak detection procedures. Two adjacent glove boxes, of maximum internal dimensions 102 x 91 x 76 cm length, width, and height, were assigned for the x-ray instrumentation. The wall separating the two boxes was removed so as to make one large box about 205 cm long.

2.2 It was desired that the diffractometer be capable of meaningful analysis of as little as one milligram of sample, and that the spectrograph have a sensitivity of the order of 100 to 1000 ppm for elements of atomic number 13 or higher, and have some sensitivity for atomic number 12. It was intended that the spectrograph be used primarily as an adjunct to the diffractometer.

2.3 The instruments were to be so designed so that a minimum of specimen preparation would be necessary to insert and retain specimens in the instruments. In particular, the free surface of the specimen was required to be up and as near horizontal as possible, so as to minimize the possibility of the specimen falling out of the specimen holder in the absence of any pellet-

izing or adhesive procedures in specimen preparation. Moreover a minimum of vibration and shock was to be transmitted to the specimen.

2.4 Specimens were to be freely interchangeable between the two instruments, so that it would not be necessary to add further preparation procedures in order to examine the specimen with one instrument after it had been examined with the other.

2.5 The instruments and system were to be designed in such a way that they would be independently operable from outside the biological containment boxes and so that a dedicated computer control and data acquisition system could be added for time-shared operation of the two instruments, with essentially no modification of the basic manual system.

2.6 Only a short time was available for design purposes because the complete manual system was to be delivered and installed before the Apollo XI mission. Moreover the instruments were to be operable by LRL personnel who were not necessarily research oriented.

To satisfy these requirements, it was decided that the diffractometer should be designed and constructed as a new instrument, based, however, on proven design criteria and components; whereas the spectrograph should be based on a modification of existing commercial instrumentation. Because of the relative inaccessibility of the instruments within the glove box, it was felt that the designs should be carried out in such a way as to reduce the amount of realignment required when changes in the instrumental parameters are made. The electronic control system, although

based on well-known principles and proven components, should necessarily be a new design in order to satisfy the aforementioned special requirements.

3. SYSTEM DESCRIPTION

3.1 General Arrangement

Because of the physical dimensions of the various components of the instruments, only the goniometers, x-ray tubes, x-ray detectors, sample presentation devices, etc., could be placed within the biological containment boxes, while the x-ray high voltage generators, detector electronics, control systems, water cooler and circulator, etc., were necessarily outside the containment boxes.

A schematic block diagram of the instrumental system is shown in Figure 1. Items to the left of the heavy dashed line are considered to be within the biological containment boxes, while items to the right are outside of the boxes. The upper half of the diagram is devoted to the x-ray spectrograph (fluorescence analysis), and the lower half to the x-ray diffractometer (diffraction). The water recirculator and cooler is common to both instruments, as is the PDP 8/I computer and its peripheral equipment. This report is not concerned with the computer control system (enclosed in dotted rectangle in Fig. 1). This is shown on the diagram only to indicate how it links with the system.

The lower half of the block diagram (diffraction) will be discussed in some detail. The upper half is schematically the same except for minor differences which will be specifically noted.

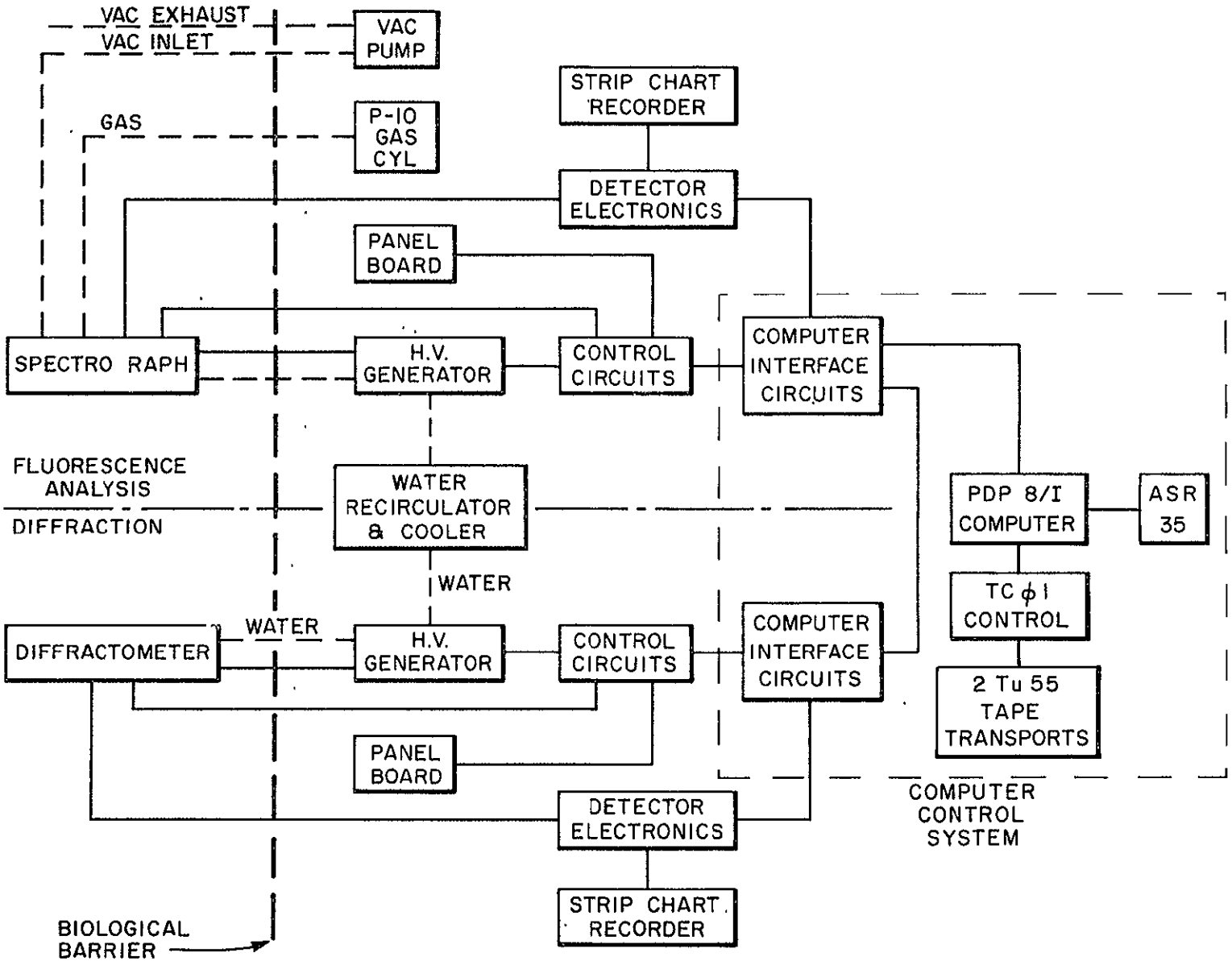


Figure 1

Block diagram of x-ray analysis system

The diffractometer within the glove box is operated from the control circuits in response to commands from the portable panel board which has its most convenient position on top of the glove box. The control circuits also monitor the diffractometer and activate the panel board display. The control circuits also carry out a limited monitor, control, and display functions with respect to the high voltage generator. The high voltage generator supplies high voltage and current and cooling water for the x-ray tube. Cooling water for the high voltage generator and x-ray tube operates in a closed circuit cycle making use of the water recirculator and cooler. The x-ray detector is activated from the detector electronics circuits and supplies signals to the detector electronics circuits. After appropriate processing of the detector signals, the output of the detector electronics circuits is permanently recorded by means of the strip chart recorder, or, in the computer control mode, the output is further processed by the computer interface circuits and the PDP 8/I computer for data reduction and recording.

For fluorescence analysis (upper half of Figure 1) there is also a source of P-10 gas required for operation of the flow proportional detector and a mechanical vacuum pump for evacuating the spectrometer. Both the P-10 gas and the vacuum pump exhaust are vented within the glove box in order to maintain integrity of biological containment.

3.2 Recommended Room Arrangement

Plan and elevation views of the recommended room layout for the system are shown in Figure 2. It will be noted that the high voltage cables, signal cables, power cables, control cables,

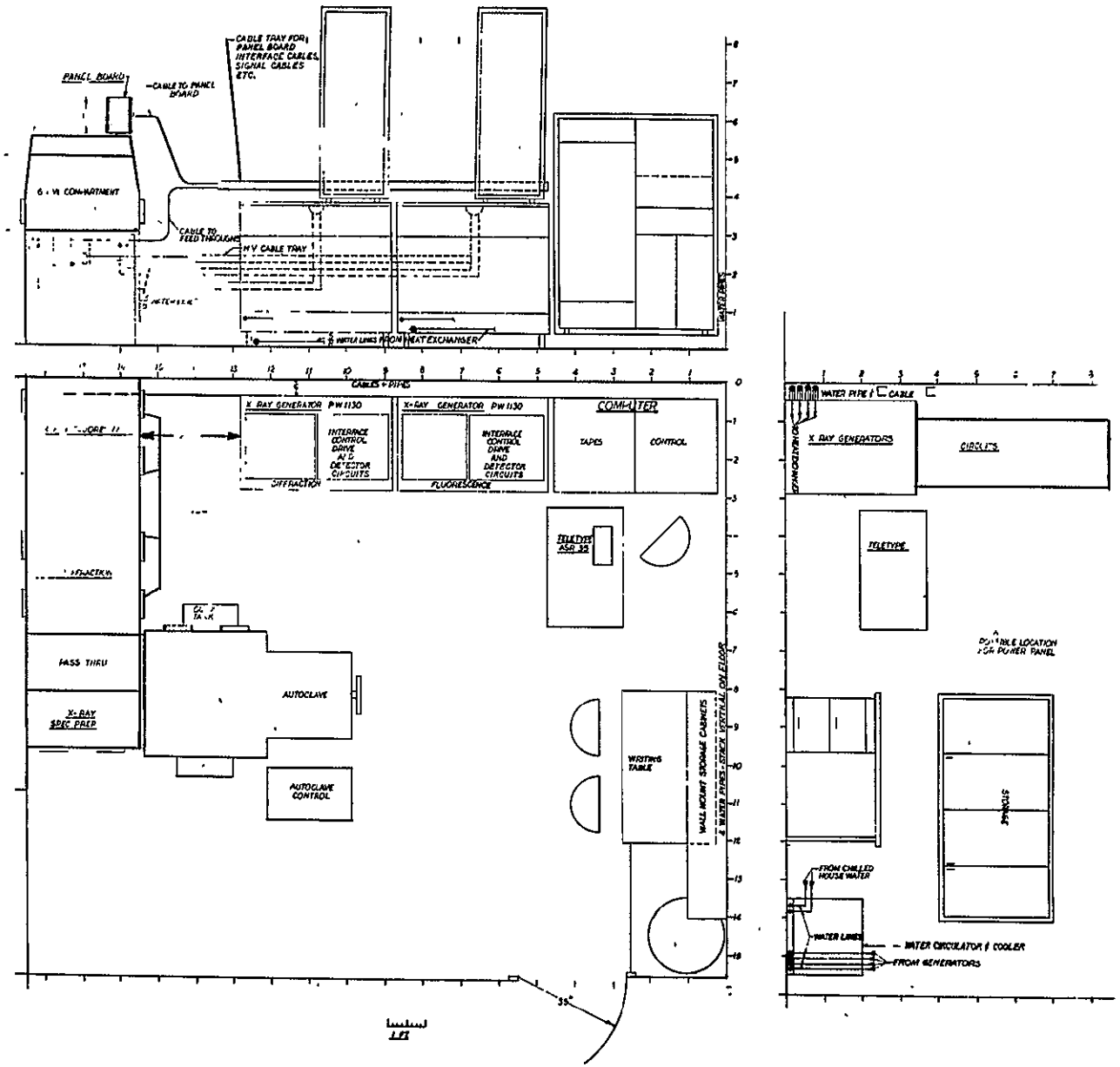


Figure 2

Recommended layout of laborat

water lines, and gas and vacuum lines (not shown in the figure) are routed along the walls from the appropriate units to the glove boxes, and fed into the glove boxes through appropriate feed-throughs in the floor of the glove boxes. The panel boards are placed on top of the glove boxes. The x-ray high voltage generators are placed along the walls as close as possible to the glove boxes to minimize the required length of high voltage cable. The cabinets housing the detector electronics, control circuits, and computer interface circuits are placed on top of the high voltage generators in order to conserve space and again to minimize the cable lengths. Short cable length from computer to interface is required to minimize transmission time in accordance with the computer manufacturer's specification. Thus the computer should be positioned as close as possible to the interface circuits. The same considerations apply to the teletype. The water circulator and cooler may be placed in any reasonable location, and it was therefore recommended that it be placed in a corner of the room out of the way. In order to facilitate the rapid examination of the data produced by the x-ray instrumentation, and to satisfy the need for preliminary study and utilization of the data to guide the course of the measurements, space should be provided for a writing table or desk close to the instrumentation. Appropriate reference data, tables, and indices should be available close at hand.

3.3 Glove Box Layout

Figure 3 shows a photograph (taken during installation) of the glove boxes with the instruments installed, and the panel boards in position atop the glove boxes. The diffraction glove box is

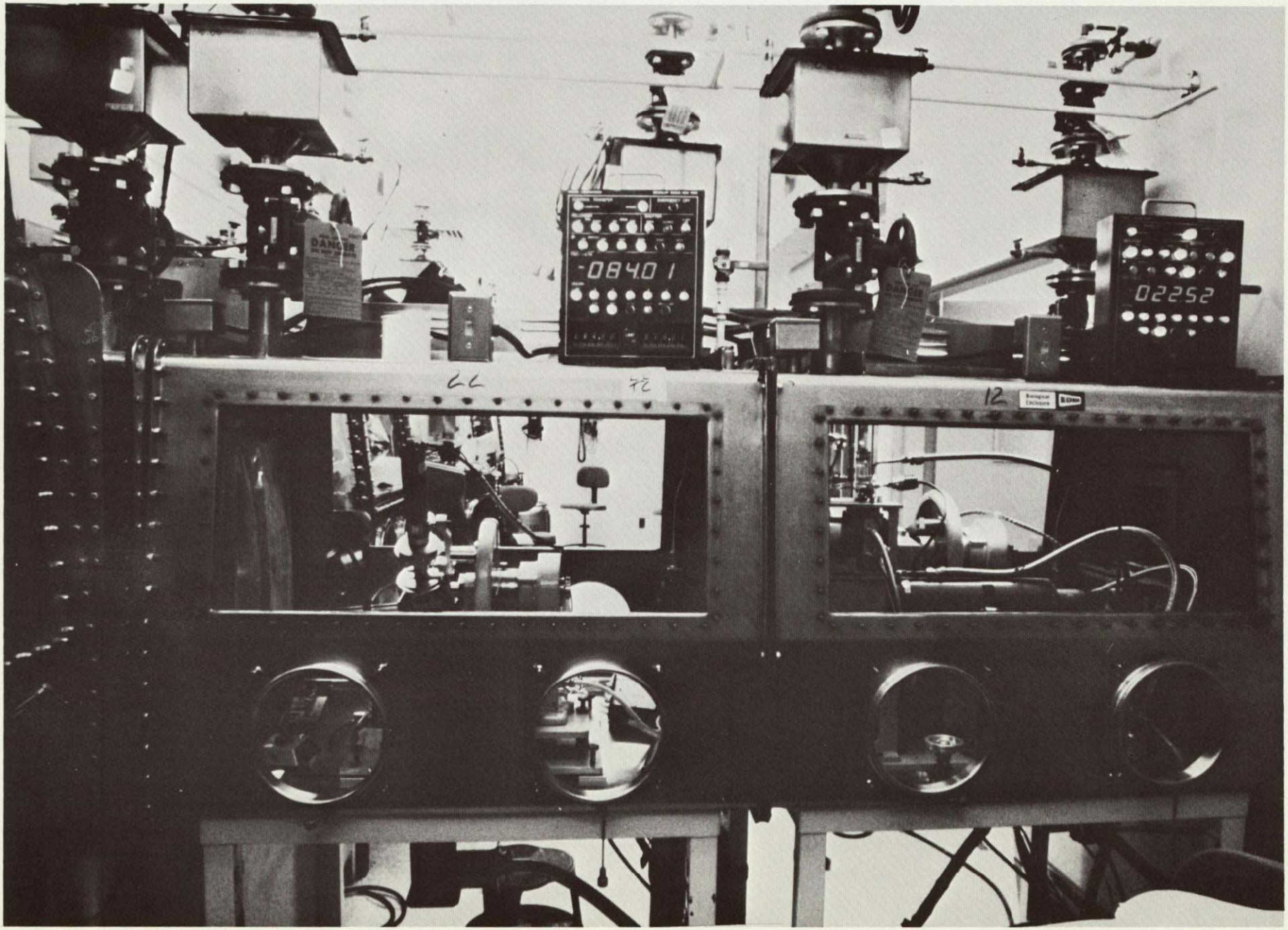


Figure 3

X-ray analysis system during installation

on the left. Figure 4 shows a plan view of the diffraction glove box on the left and a plan view of the fluorescence analysis glove box on the right. Figure 5 is a photograph of the diffractometer. Figure 6 shows an elevation view of the diffraction glove box, and indicates how the high voltage connections are brought through the floor of the glove box. In Figure 6, the base plate 4, which supports the x-ray tube tower assembly, 3 and 1, the goniometer 10, and the specimen drive motor 8, is bolted to the flange 5, which is welded into the floor of the glove box. The high voltage feedthrough assembly 6, is bolted to the bottom of the tube tower support 3, which is welded to the base plate 4. Appropriate O-ring gasket seals are provided to maintain containment integrity. The high voltage feedthrough assembly is discussed in more detail in Section 3.6. See also Figure 12.

3.4 Diffractometer

3.4.1 General Design

The design of the diffractometer was carried out according to specifications laid down by Dr. William Parrish, and is based on recent research work aimed at achieving significant increases in intensity and resolution (Parrish, 1968). Specific features are the use of $CrK\alpha$ radiation to attain higher dispersion and hence improved resolution; the use of a large angle-of-view of the specimen for increased intensity; the use of a long narrow focal line x-ray tube to enhance resolution; the use of a short radius diffractometer for increased intensity and for reduced instrument size to fit within the glove box; and the use of a new and easily changeable receiving Soller slit assembly for improved resolution.

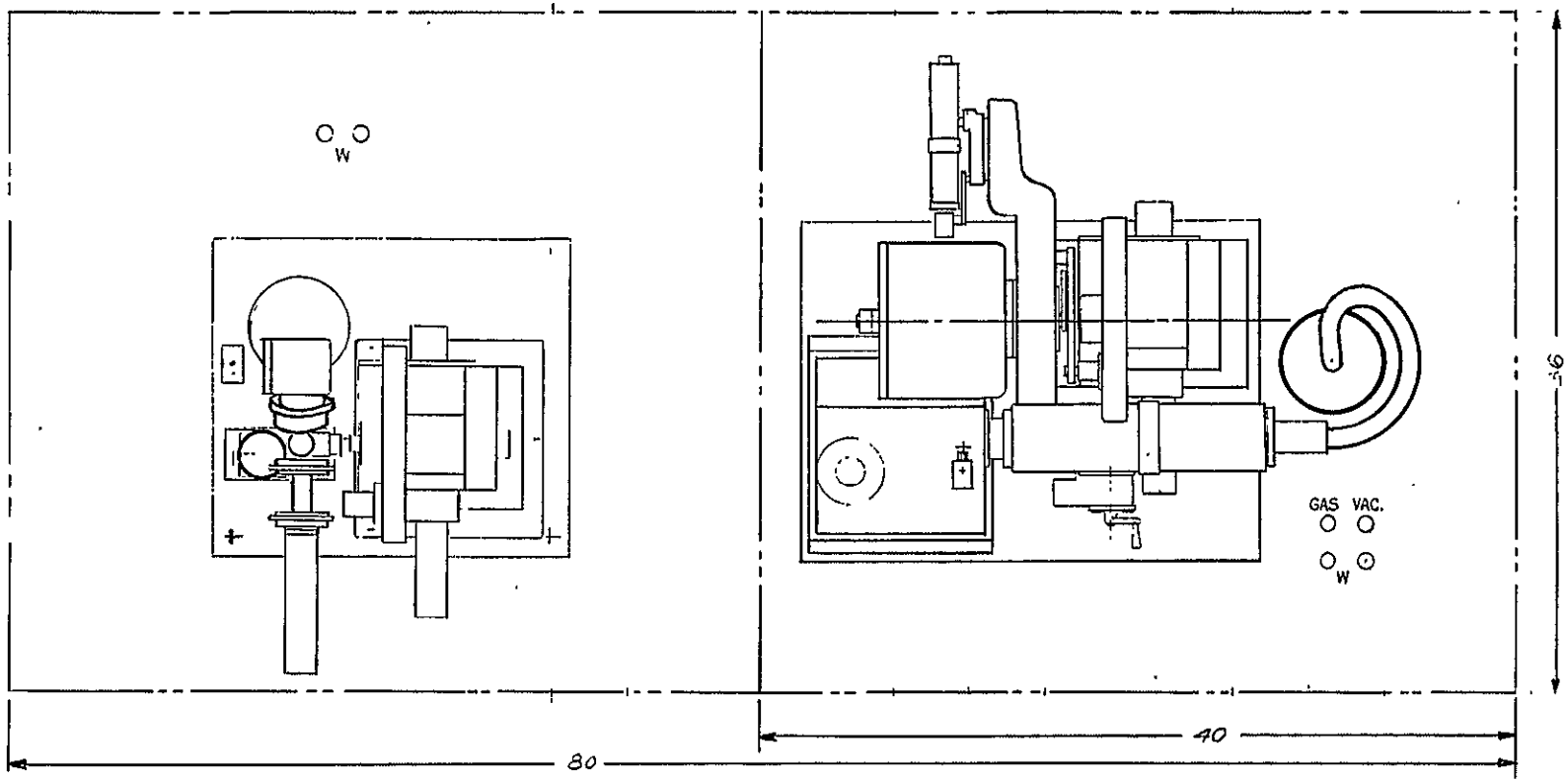


Figure 4

Plan view of diffractometer (left) and spectrograph glove boxes

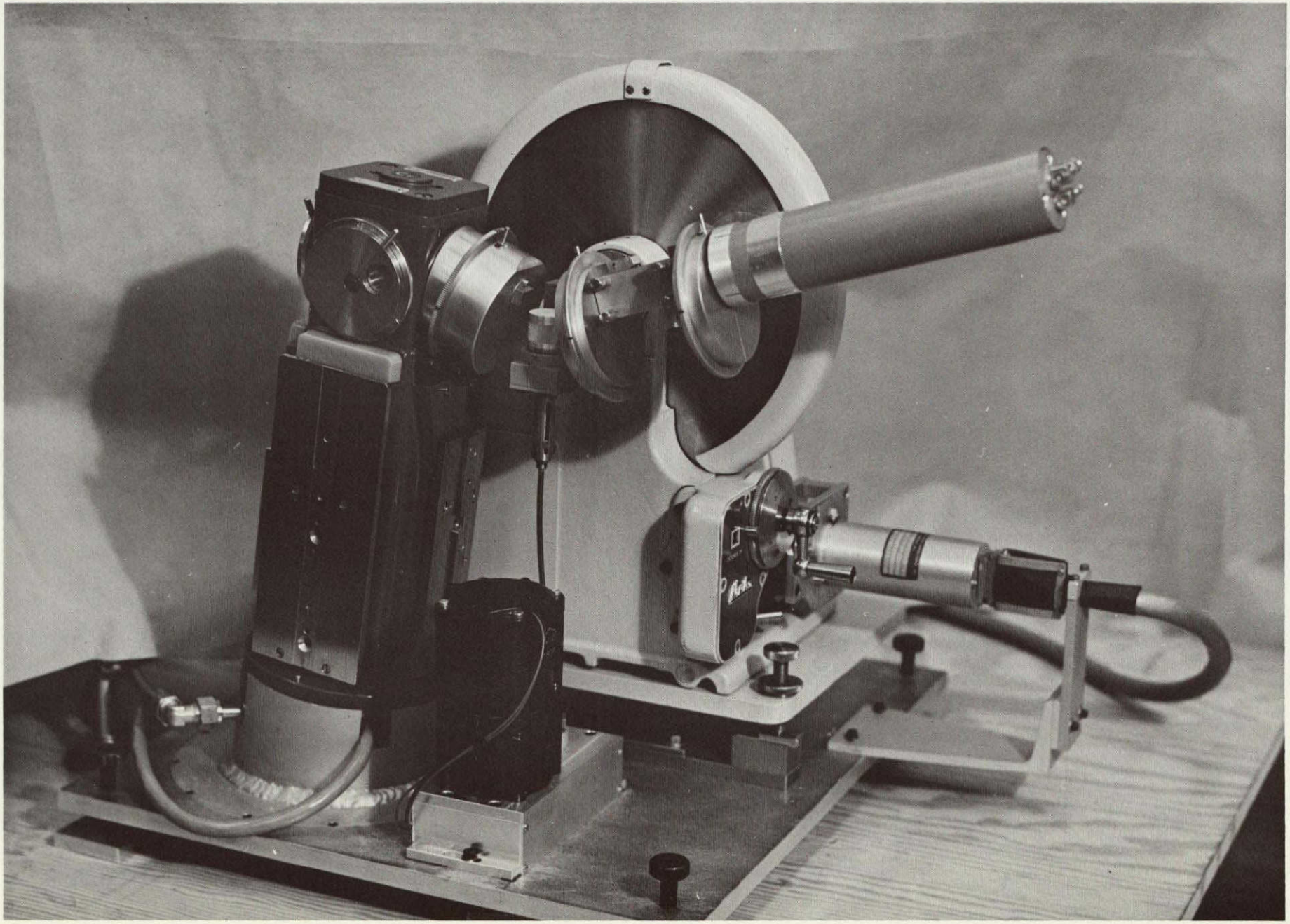


Figure 5

The x-ray diffractometer

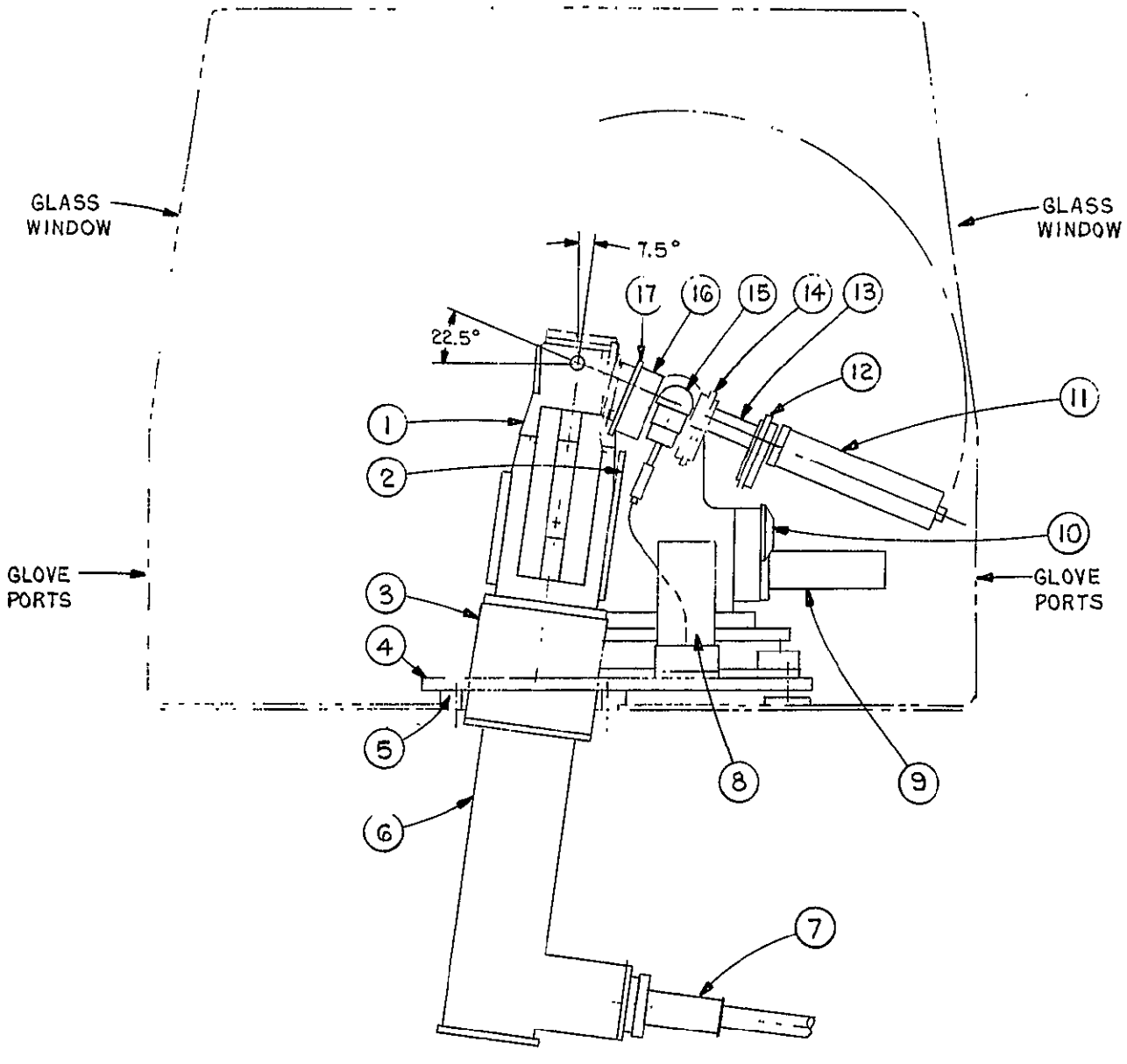


Figure 6

Diffractometer elevation

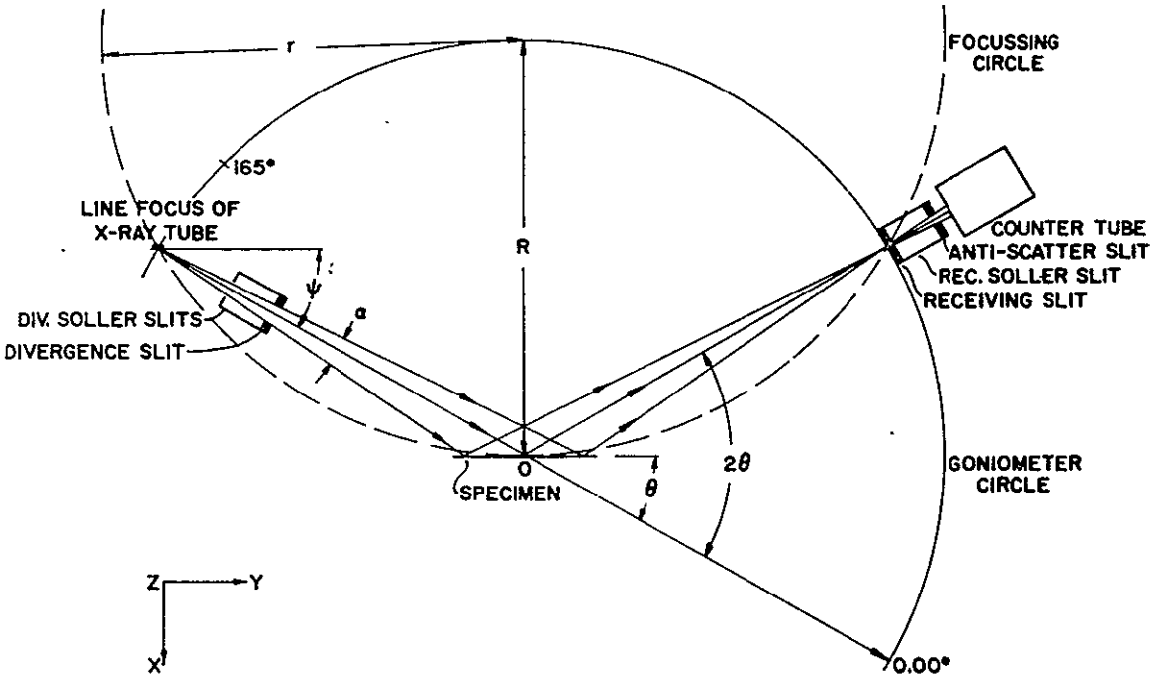
3.4.2 Optics

The various optical parameters indicated in Figure 7 are specified in terms of the nomenclature given in the published literature (Parrish and Lowitzsch, 1959). One difference should be noted between the optics used for this instrument and that usually used: the antiscatter slit in the present instrument is placed between the receiving slit and the specimen, rather than between the receiving slit and the detector. This was done in order to keep the overall size of the instrument within the constraints imposed by the dimensions of the glove box. Values of the various parameters are given in Table I.

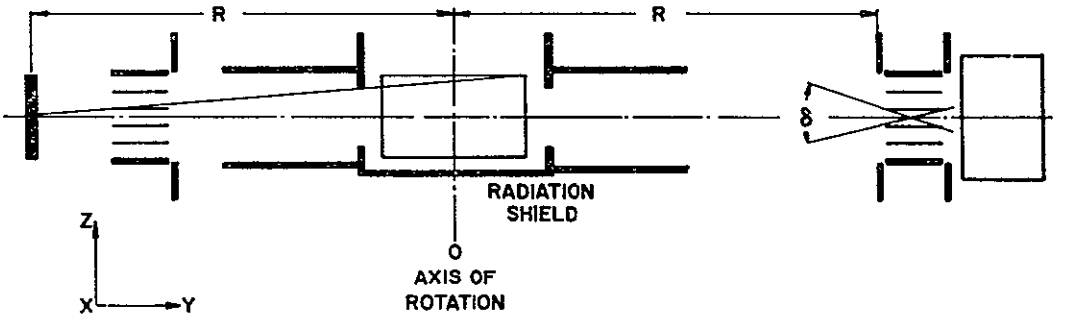
The divergence, anti-scatter and receiving slits are mounted on handwheels (17, 14, and 12 respectively in Figure 6) so that slit changes are made by turning the handwheels to appropriate indexed positions. Provision is made for aligning each slit on its handwheel, so that changes of slits do not require realignment of the instrument. The divergence Soller slit assembly is fixed in the housing 16 and is not changeable. The receiving Soller slit assembly and the β-filter are placed within the housing 13, and may be extracted and replaced after removing a cover plate from the side of the housing 13.

3.4.3 X-Ray Tube Tower Assembly

The x-ray tube tower 1 (Figure 6) is a commercial tower, Philips PW 1316, modified to permit viewing through port no. 1 the target of the x-ray tube at $\Psi = 15°$ at the maximum divergence angle of 4°. The standard filter wheel assembly was removed from this port to permit rayproof mounting of the assemblies 16 and 17



a. FOCUSSING PLANE



b. AXIAL PLANE

Figure 7

Optical parameters of diffractometer

Table I

DIFFRACTOMETER OPTICAL PARAMETERS

Ψ, angle-of-view of focal spot:			15°
Focal spot dimensions:	Actual size		0.6 x 10 mm
	Projected size		0.16 x 10 mm
R, goniometer radius:			110 mm
Divergence slits:		Width (mm)	Nominal α (degrees)
	1	4.15	4.0
	2	2.54	2.4
	3	1.71	1.6
	4	0.86	0.8
Anti-scatter slits:		Width (mm)	Nominal α (degrees)
	1	4.77	4.0
	2	3.16	2.4
	3	2.16	1.6
	4	1.31	0.8
Receiving slits:		Width (mm)	ϵ_{RS} °2θ
	1	0.419	0.20
	2	0.251	0.12
	3	0.168	0.08
	4	0.105	0.05
Divergence Soller slits:	1/2 mm foil spacing x 13 mm long δ = 2.4°		
Receiving Soller slits:	1 mm foil spacing x 48 mm long, δ = 2.4° 2 mm foil spacing x 48 mm long, δ = 4.8°*		

* This set is normally used

directly to the tube tower. Modifications were also made to the shutter circuits to permit operation and sensing from the main control circuit system. An alignment bar 2 was mounted on the tube tower to facilitate alignment of the goniometer. The tube tower support 3 is welded to the base plate 4 at an angle of 7-1/2° from the vertical. Thus the maximum deviations of the specimen from horizontal throughout the range from 0 to 90° 2θ is 22-1/2°.

The x-ray tube is based on a standard commercial design, Philips RD60, modified to permit viewing of the nominal 0.4 x 10 mm focal spot at an angle-of-view of 15° through the center of the offset window. Inherent window filtration of 0.3 mm Be. The target material is Cr, but a Cu target tube is also available. A spacer is inserted between the cooling cap of the x-ray tube and the tube tower in order to bring the target surface to the proper position relative to the exit port of the tube tower.

3.4.4 Specimen Holder and Insertion

The specimen holder is mounted within the radiation shield 15 (Figure 6). This is shown in more detail in Figure 8. The radiation shield 8 is slotted to permit entrance and exit of the incident and diffracted x-ray beams. The specimen holder 1 is retained on the rotable platform 7 by means of spring loaded pin plungers which engage a groove 6 machined on the inside wall of the specimen holder (see Figure 9 also). The platform is mounted by means of a precision ball bearing 5 in the assembly. Rotation or spinning of the specimen is accomplished by a motor driven flexible shaft 4 which engages a pin in the platform

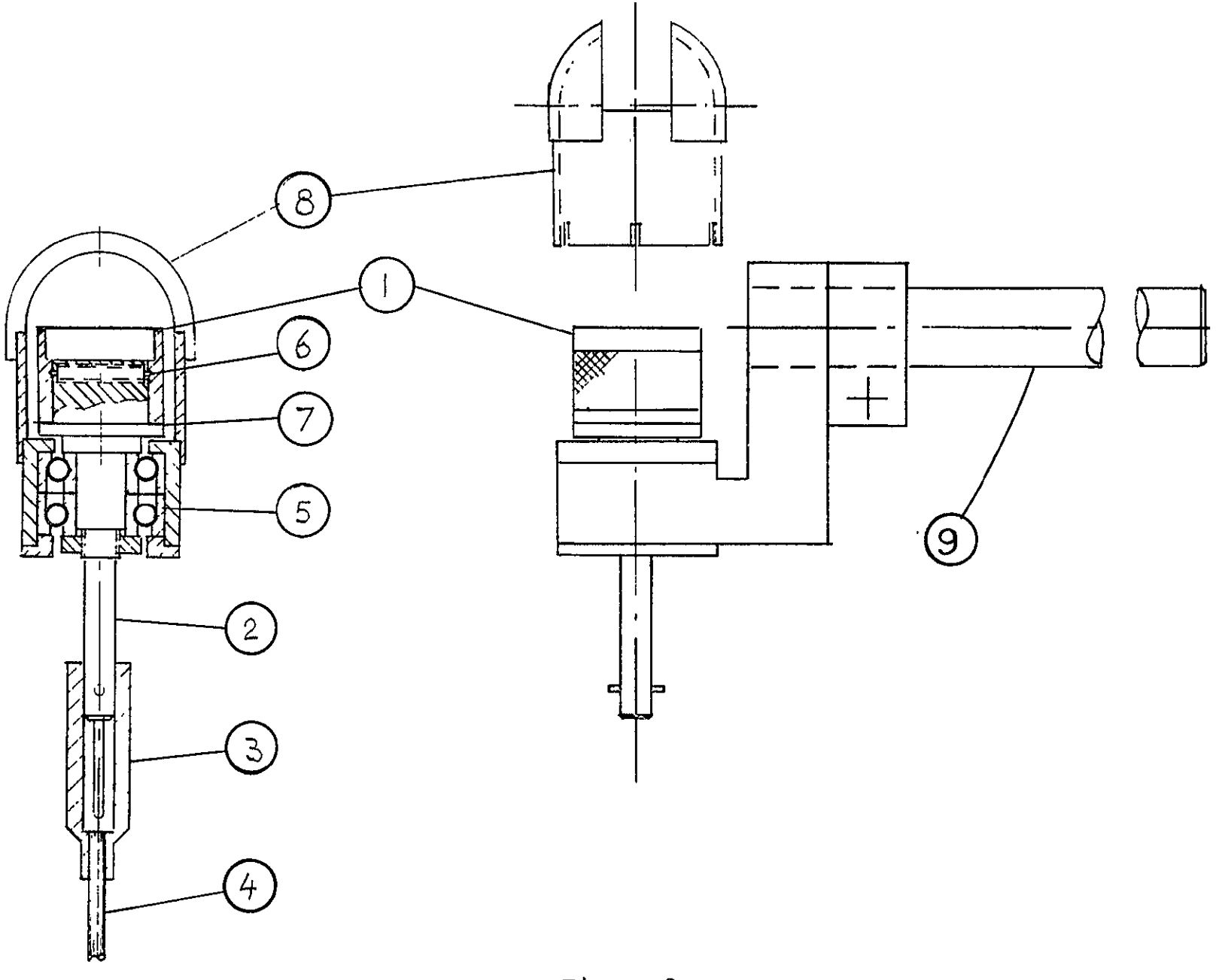


Figure 8

Diffractometer specimen assembly

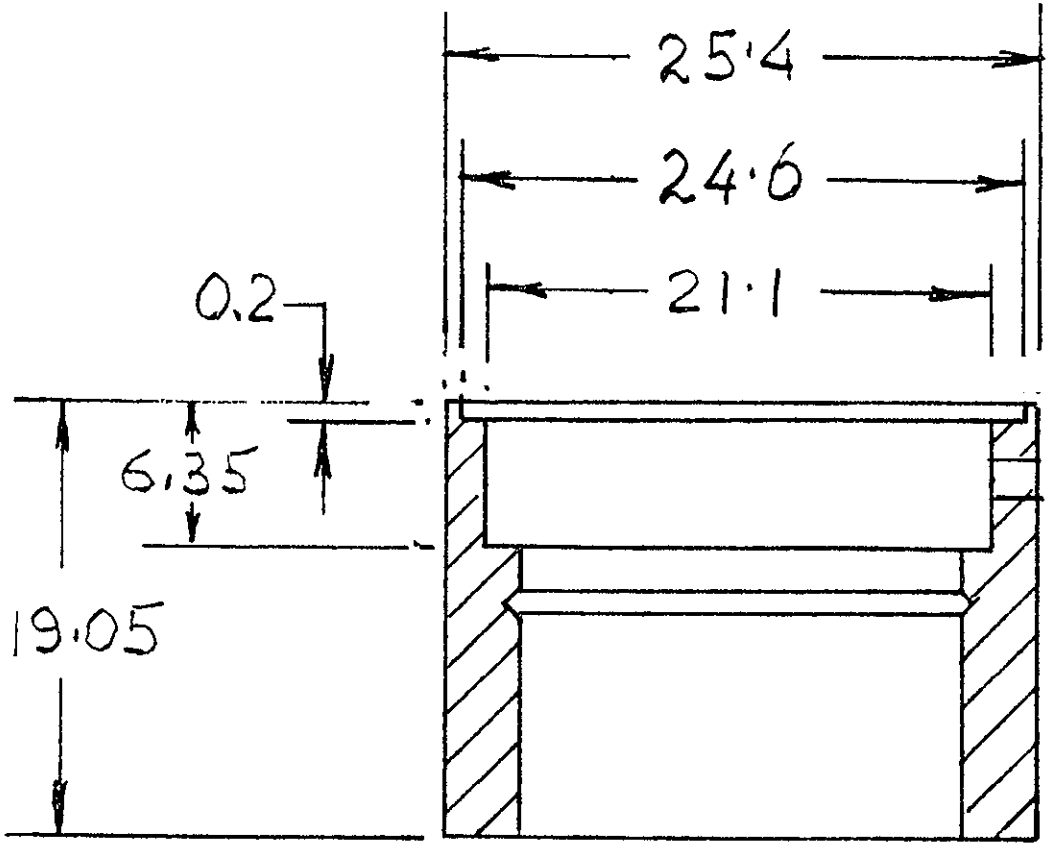


Figure 9

Diffractometer specimen holder

extension 2 by means of a slotted sleeve 3. (The motor drive, a Holtzer-Cabot motor with electrically changeable gear ratio, is shown at 8 in Figure 6.) Tolerances are such that the center line of the specimen shaft 9, which is inserted in the axis of the goniometer, lies in the top surface of the specimen holder 1, and the axis of rotation of the rotable platform intersects the center line of the specimen shaft in the center of the top surface of the specimen holder. This design was chosen to minimize the amount of vibration transmitted to the specimen by the specimen rotation drive. As shown in Figure 9, the specimen holder is essentially a hollow aluminum sleeve with an 0.2 mm recess to accept a thin (0.025 or 0.05 mm thick) beryllium foil. Thus the amount of scattered radiation from the specimen holder is minimized. The beryllium foil does cause the appearance of the diffraction line from the (002) planes of beryllium in the diffraction pattern, which may be used for calibration purposes.

3.4.5 Goniometer

A standard Philips vertical scanning goniometer was modified by replacing the normal drive motor by a stepper motor mounted on the rear panel of the goniometer. A shaft angle encoder (9, Figure 6) capable of five digit readout is mounted on the bosses normally used for the goniometer change gears, and permits readout to 0.01° 2θ. The clutch, handcrank and mechanical dial readout of the 2θ drive worm may also be used.

3.4.6 Powder Camera Mount

Provision is made for mounting a 57.3 mm radius Debye-Scherrer powder camera, which was delivered with the apparatus, at window no. 2 of the x-ray tube tower. The shutter for this window is operated from the x-ray generator control panel.

3.4.7 Alignment Devices

The alignment devices furnished were based on those described by Parrish and Lowitzsch (1959), and include a machined triangle for setting the 15° Ψ angle, slit and fluorescent screen, goniometer height fixture, pinhole assembly for zero-angle calibration, 2:1 adjustment slit, small square, and various attenuating filters.

3.5 Spectrograph

3.5.1 General Design

As already indicated, it was decided that the fluorescence analysis instrument should preferably be based on a modification of a standard commercial instrument. After careful study of a number of commercially available instruments, it became quite clear that the Norelco universal vacuum x-ray spectrograph was the only available instrument of sufficient sensitivity and resolution, capable of being modified to meet the various requirements. In particular it was the only one which fit within the glove box. To meet the requirements for specimen preparation and handling, it was necessary to invert the specimen chamber and modify the specimen retaining mechanism. In addition, the specimen chamber was modified to permit the use of a new higher

powered and more efficiently exciting x-ray tube. Photographs of the modified spectrograph are shown in Figures 10 and 11. The specimen chamber 1 is mounted on a stand 2 high enough to give sufficient clearance above the base plate for the scintillation counter 3 when approaching values of 2θ in the vicinity of 90°. The stand 5 brings the goniometer 4 to the proper height for alignment with the crystal chamber 6. The cover 7 of the crystal chamber is mounted by means of a shaft hinge 8 at its edge, and is fastened down with three hand screws to permit one-handed removal of the cover with the instrument in place of the glove box.

3.5.2 Optics and Crystals

The x-ray optics is basically that of the standard Norelco instrument, except that the optical chamber is rotated 180° about its axis. The crystal holder positioning assembly 9 has been modified to permit easier removal of the crystal slide 10. Two crystal slides have been furnished with the crystals to be preadjusted for proper 2:1 tracking, and it is planned that these can be interchanged while the instrument is within the glove box. The particular crystals furnished and their range of application are listed in Table II.

One crystal slide carries the LiF 200 crystal and the KAP 001 crystal. These two crystals should suffice for the majority of specimens to be studied. As is apparent from Table II, the LIF 200 crystal is suitable for detecting radiation in either the K or L series from all elements having atomic number 22 (Ti) or higher. This crystal may be used with either the flow proportional counter or the scintillation counter. The KAP 001 crystal

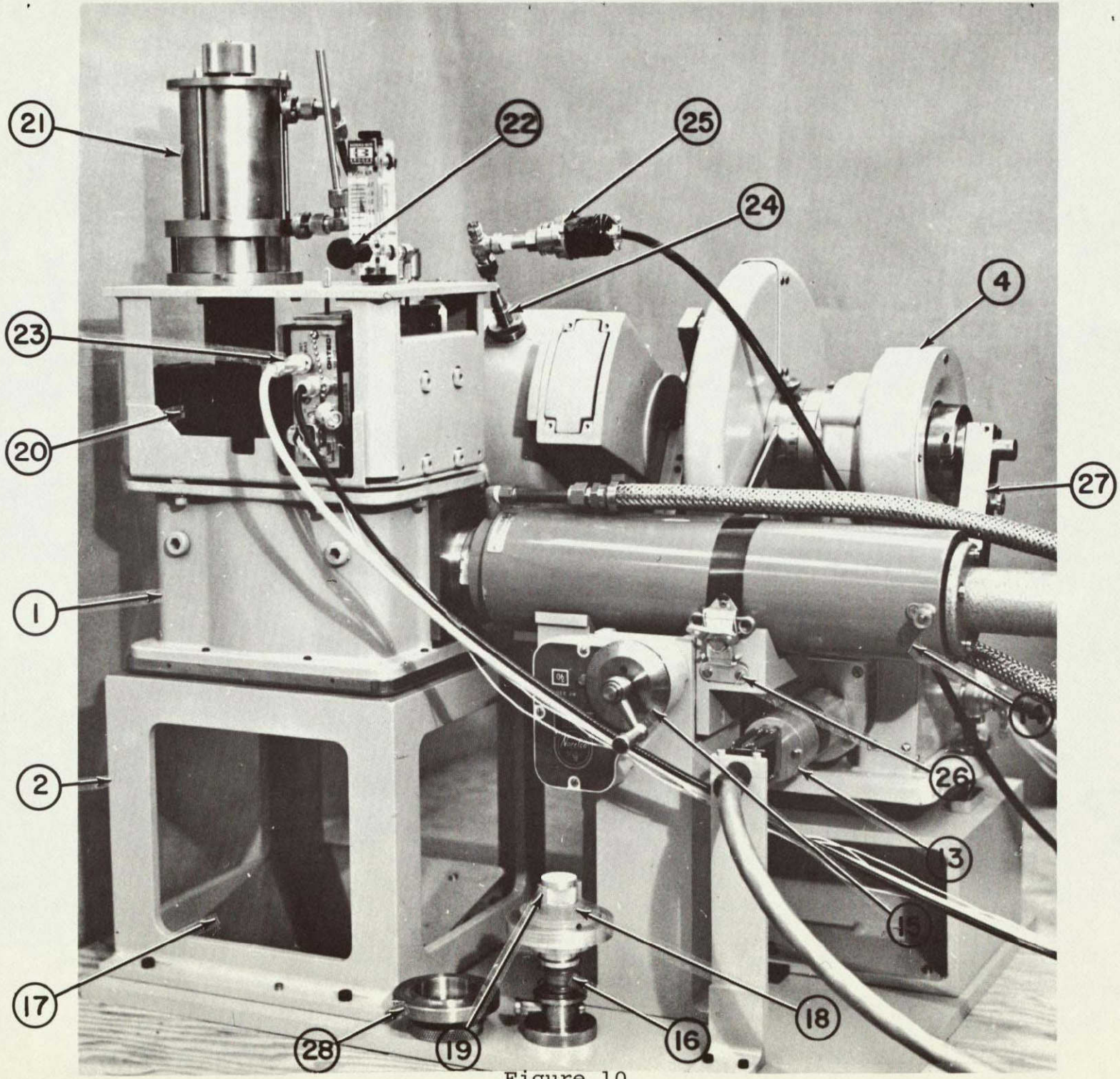


Figure 10
The x-ray spectrograph (front view)

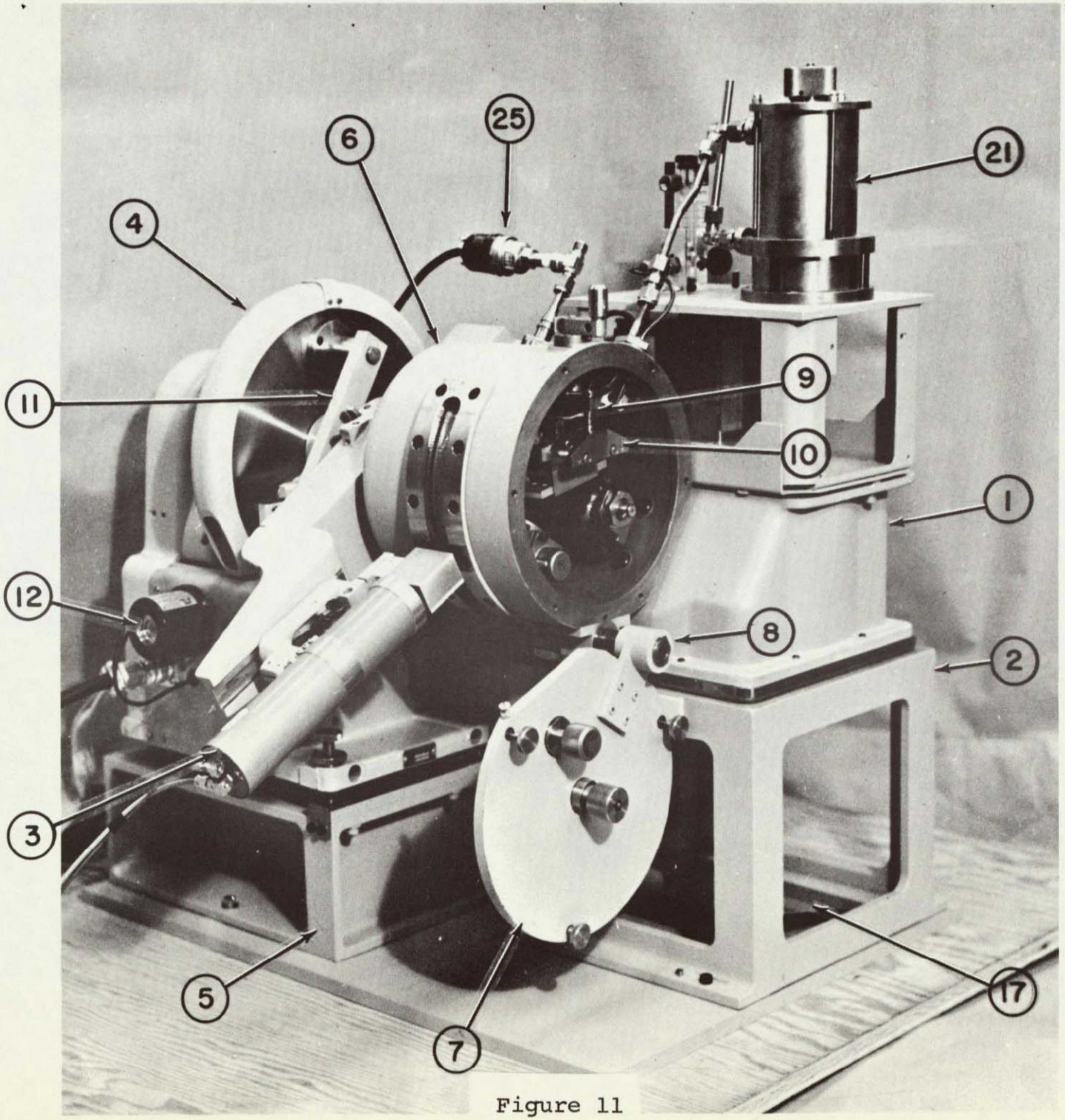


Figure 11

The x-ray spectrograph (rear view)

Table II

CRYSTALS FOR SPECTROGRAPH

Crystal & diffracting planes	2d (A)	λ_{max} (A)	Recommended Range		
			λ(A)	Atomic Number (Z) K Series	L series
LiF 200	4.03	2.8[a]	0.5 - 2.8	22 - 50	58 - 92
KAP 001	26.6	12.[b]	2.5 - 12.	12 - 22	
Quartz 203[d]	2.74	1.9[a]	0.2 - 1.9	27 - 72	66 - 92
PET 002[d]	8.75	7.5[c]	2.7 - 8.	14 - 22	

[a] Assuming $2\theta_{max} = 90°$ to prevent scintillation counter from striking base plate.

[b] Limited by excitation conditions.

[c] Assuming $2\theta_{max} = 120°$ with flow proportional counter in advanced position.

[d] These crystals are used primarily for higher resolution. The intensities obtained will be considerably less than fo: the other two crystals.

is suitable for elements having atomic number between 12 (Mg) and 22 (Ti), and is used primarily with the flow proportional counter. (Actually this crystal may be used to detect elements as light as O or F, but these are weakly excited in conventional x-ray instruments.

The length of these two crystals is 76 mm, which is the maximum permissible without mechanical interference with free motion of the flow proportional counter, and gives substantially more intensity than shorter crystals for small values of 2θ. With the 76 mm long crystal, the full beam of radiation from the specimen is intercepted and diffracted down to about 19° 2θ, whereas for a crystal 51 mm long, the full beam would not be intercepted between 29° 2θ.

For some analyses it may be desirable to have higher resolution than is attainable with the LiF 200 and KAP 001 crystals. For such analyses, the Quartz 203 and PET 002 crystals give greater resolution (but with considerably lower intensity) because of their higher dispersion. These crystals have been mounted on the second crystal slide. In using the PET crystal, it should be kept in mind that it is in the "advanced" position, as described in section 4.2.2 below and the flow proportional counter must therefore also be in the "advanced" position. (The scintillation counter cannot be used with this crystal.) The true value of 2θ in this case is obtained by adding 30° to the value indicated by the goniometer 2θ readout. This is described in the Norelco instruction manual for the universal vacuum spectrograph. These two crystals were not obtainable in the 76 mm length, but it is anticipated that they will receive their

maximum application at values of 2θ high enough that they can intercept the entire beam from the specimen.

3.5.3 X-Ray Tubes

The FA60/2.5 x-ray tube normally used with the Norelco spectrograph has been replaced by a newer tube specified as FA100/3, and it was necessary to modify the specimen chamber to accept this improved tube. This tube is distinguished by its higher power rating and the relatively small distance between its focal spot and the exit window for the x-rays, thus giving increased intensity for small specimens (Spielberg, Parrish and Lowitzsch, 1959; Spielberg and Mack, 1958). Two tubes have been furnished with the instrument, one having a Cr target and one having a W target, both tubes are rated at 2.7 kW target loading. The W target tube is recommended for general usage, although the Cr target may be preferred if the primary emphasis in sensitivity is to be given to light element excitation. No effort was made to furnish or include provisions for special demountable x-ray tubes for enhanced sensitivity for light element excitation (Spielberg, 1959, 1962, Henke, 1963) because it was felt that such tubes would be almost impossible to maintain and operate in the apparatus under the special constraints imposed by the biological integrity requirements and the limited space available.

Because the maximum specimen area is considerably smaller than the maximum area irradiated by the x-ray tube, it is necessary to place a 6.3 mm diam. lead diaphragm over the window of the x-ray tube in order to minimize the radiation coming from the

specimen holder and other parts of the specimen chamber. This necessarily reduces the intensity obtained from the specimen.

The inversion of the specimen chamber also dictated the construction of a new support stand 26 and clamping arrangement for the x-ray tube.

3.5.4 Detector Selection

The spectrograph offers the operator a choice of detectors: either the scintillation counter or the flow proportional counter if a crystal is used in the "normal" position, or only the flow proportional counter if a crystal is used in the "advanced" position (see Norelco instruction manual). Details of the detectors are given below in Section 3.9, and of the detector electronics in 3.10.

Signals from the flow proportional counter are transferred to the charge sensitive preamplifier 23 mounted above the specimen chamber and thence to the electronic counting circuits. The flow counter mounting arrangement was modified to insulate the flow counter from ground, thereby eliminating pickup of stray signals from multiple grounds. The preamplifier is also insulated from ground for the same reason. The gas feed lines to the flow counter were modified to make it possible to incorporate the gas density stabilizer 21, thereby making it possible to minimize changes in flow counter gain due to fluctuations in gas temperature and pressure (Spielberg, 1967b). The gas flow rate is monitored and controlled by the flow meter and needle valve 22.

3.5.5 Goniometer Modifications

Because the crystal chamber is turned 180° about its axis from its normal relationship to the goniometer, it was necessary to insert a drive link 11 between the goniometer and the detector arm assembly to avoid interference with the normal mechanical limits of the goniometer rotation. The stepper motor 12 and the shaft angle encoder 13 are located in the same place as in the diffractometer goniometer. Because the x-ray tube 14 is now positioned in front of the goniometer, it was necessary to extend the worm shaft to make the goniometer hand crank and angle dial 15 accessible.

3.5.6 Specimen Holder and Insertion

To permit full interchange of specimens between diffractometer and spectrograph, a special specimen carrier 18 was designed. The specimen 19 is placed on the carrier 18 which is placed, as shown in the figure, on the specimen insertion device 16. The insertion device 16 is used to insert the specimen carrier into the specimen chamber through the specimen insertion port in the bottom of the specimen chamber. The insertion is done with the aid of the mirror 17, which is placed so as to make the insertion port visible. The specimen carrier 18 has in its base two pin plungers, which serve to retain it in a specimen position in the carousel which is inside the specimen chamber 1. The specimen positions have been modified for this purpose by machining peripheral grooves in their walls to engage the pin plungers. The center portion of the carrier 18 is spring loaded so as to press the specimen holder 19 against a reference surface in the carousel. Some of the carriers 18 have been made

so that the center portion is capped with a "reradiator" of the same material as the x-ray tube target. Thus when the specimens are sufficiently transparent to x-rays, that portion of the white spectrum which would normally pass through the specimen and be lost will excite in the "reradiator" radiation characteristic of the tube target. This, in turn, irradiates the specimen from underneath, leading to enhanced specimen excitation.

Specimen rotation is provided by the gearmotor 20 with electrically actuated gear shift, giving specimen rotation speeds of 20 and 1 rpm. The cover 28 for the specimen insertion port has been modified by the installation of pin plungers to engage a groove machined in the walls of the port. To insure that the cover will seal against vacuum slide mounted pressure fingers are used to press the cover up against the O-ring in the port. The pressure fingers are withdrawn when it is desired to remove the cover.

3.5.7 Vacuum System

Analysis and detection of elements having atomic number less than about 20 is made possible or greatly enhanced by evacuating the specimen and crystal chambers. The vacuum exhaust port 24 on the crystal chamber is connected by means of a feed through in the floor of the glove box to a mechanical pump, as indicated in Figure 1. Vacuum within the specimen chamber and crystal chamber is monitored by means of the thermocouple gage 25, which is mounted at the vacuum exhaust port of the crystal chamber rather than at the vacuum pump (as is customarily done) in order to obtain a more realistic measure of the vacuum. The control

and meter for reading the gage (Hastings-Raydist Model VT-4) is also within the glove box. Vacuum obtained depends upon leak rates in the various chamber seals and the window of the flow proportional counter. When using a flow counter having a 1 micron polypropylene window the vacuum is better than 0.5 Torr, and with a 6 micron mylar window better than 0.2 Torr.

3.6 High Voltage and Other Feedthroughs and Cables

3.6.1 Diffractometer High Voltage Feedthrough

Some consideration was given to the possibility of making a hermetic seal directly between the floor of the glove boxes and the high voltage cables between the x-ray generators and the x-ray tubes. However, some tests revealed that the internal wiring of the high voltage cables was not hermetically imbedded in its insulation. It was therefore necessary to design and fabricate special high voltage feedthroughs for both the spectrograph and the diffractometer. These feedthroughs were to be capable of sustaining a voltage of 60 kV dc, and of carrying filament current for the x-ray tube (approximately 5 amps). A drawing of the high voltage feedthrough for the diffractometer (item 6 of Figure 6) is shown in Figure 12. The external shell of the feedthrough is a long stainless steel cylinder 6 with a flange welded to its upper end and a side arm welded near its lower end. An internal threaded cap 21 is welded inside the upper part of the long cylinder. This cap, and an inserted bakelite socket 19, which is referred to as a Federal connector*, hermetically separates the feedthrough into two sections. Another

---

* Federal Standard No. 72 - "Shockproof Cable Terminal and Receptacle for X-ray Equipment", General Service Administration. See also ASA Spec. C86.1-1958.

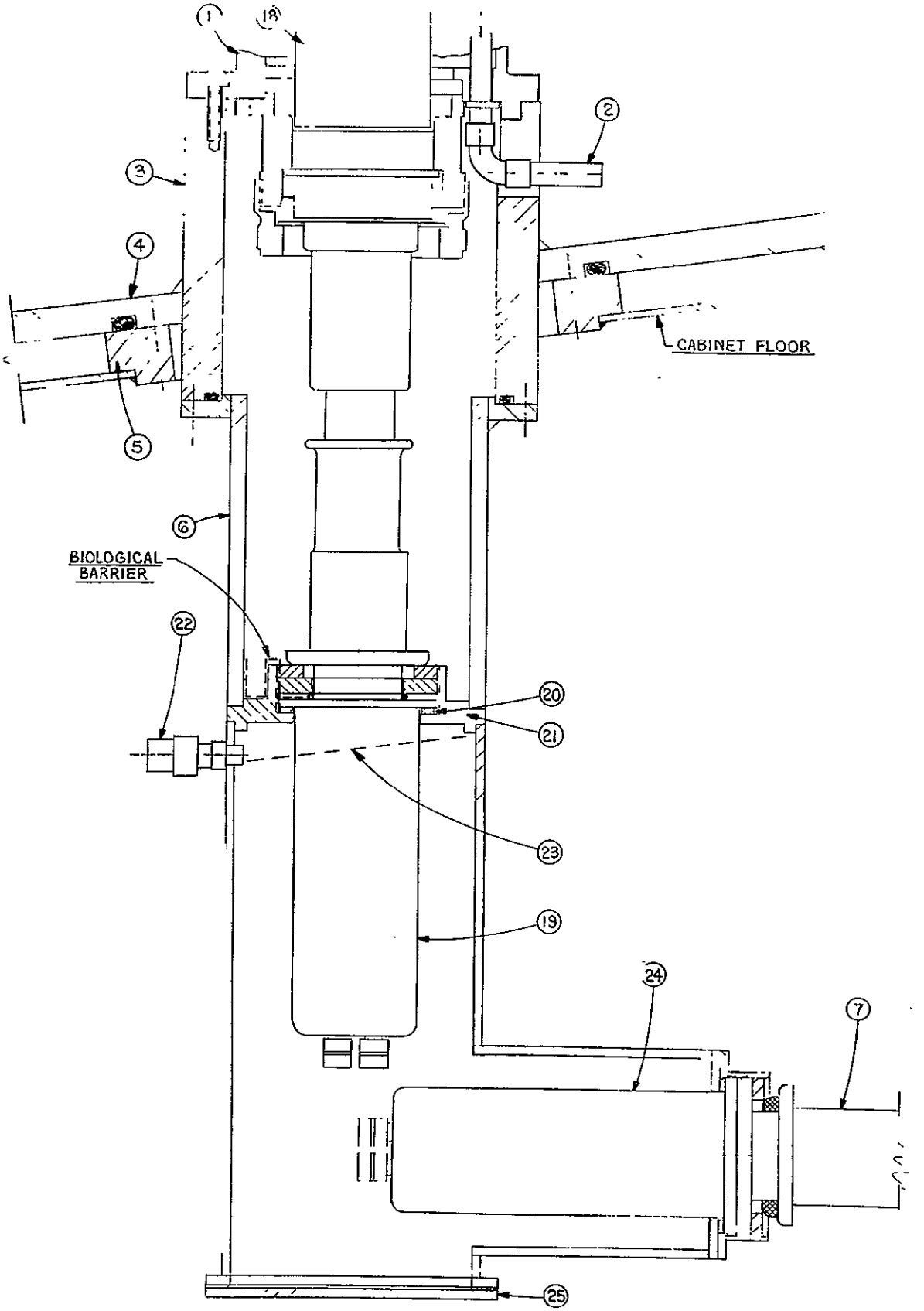


Figure 12

High voltage feedthrough for diffractometer

Federal connector 24 is inserted into the side arm. Hermetic sealing of the connectors is obtained from the gasket 20. An externally threaded nut, not designated in the figure, is screwed into the cap 21 to press the flange of the Federal connector 19 against the gasket 20. Similar arrangements are carried out for the connector 24. The feedthrough is filled with special high voltage insulating oil (Shell Diala) to the level 23 (note that the feedthrough is tilted by 7.5° from the vertical). The oil is vented to room air through the breather valve 22. Access to the interior of the feedthrough for the purpose of making electrical connections between the sockets 19 and 24, and for filling with oil is obtained by removing the gasketed cover plate 25. The upper part of the feedthrough assembly (above the cap 21 and Federal connector 19) is inside the biological barrier, while the lower part is outside the barrier, as indicated in Figure 12. The lower part of the feedthrough assembly, and its entire exterior, is outside the biological barrier. A specially fabricated short high voltage cable 18 is inserted into the tube tower 1 (only the lower portion of the tower is shown in Figure 12) and plugs into the Federal connector 19. A special stiffening clamp (not shown in the figure) was placed around the middle of the ultrashort cable to facilitate assembly. The tube tower support 3, to which the tube tower is bolted from above and the high voltage feedthrough from below, is welded to the base plate 4, as discussed in Section 3.3 above. The base plate 4 was made in two parts, to facilitate fabrication. The inner part, shown in Figure 12, was made small enough that the welded assembly of items 3 and 4 of Figure 12 could be more easily handled with normal machine shop equipment for various

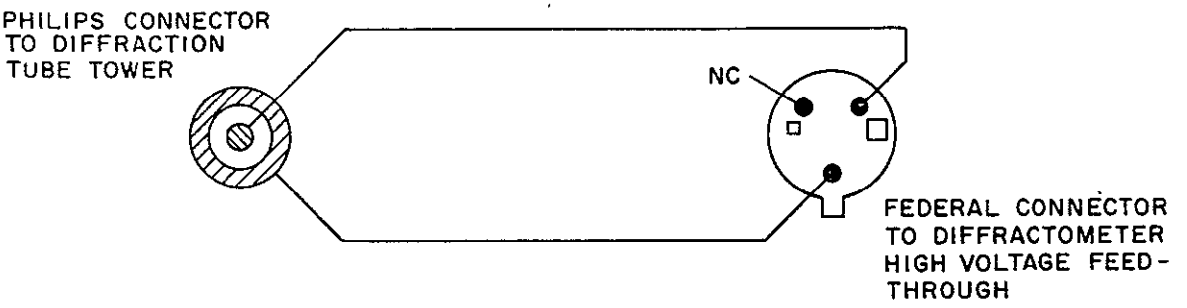
finish machining procedures. It was then bolted to the outer part of the base plate. The outer part of the base plate was provided with leveling screws to facilitate assembly and test of the diffractometer system before shipment to LRL. As installed in the glove box, the diffractometer base plate 4 is bolted to and supported by the flange 5 welded in the glove box floor, and hence the base plate leveling screws are backed out and should not make contact with the glove box floor. Cooling water for the x-ray tube tower comes through the copper tubing 2.

3.6.2 Spectrograph High Voltage Feedthrough

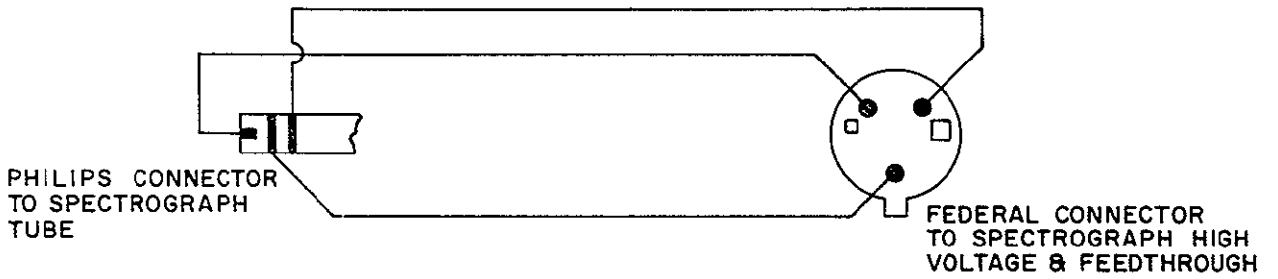
The high voltage feedthrough for the spectrograph is of much simpler design than the feedthrough for the diffractometer. Its construction details are given essentially by the lower part of Figure 12 from item 21 on down. Item 21 is in this case altered to provide a flange for bolting to the floor of the glove box.

3.6.3 High Voltage Cables

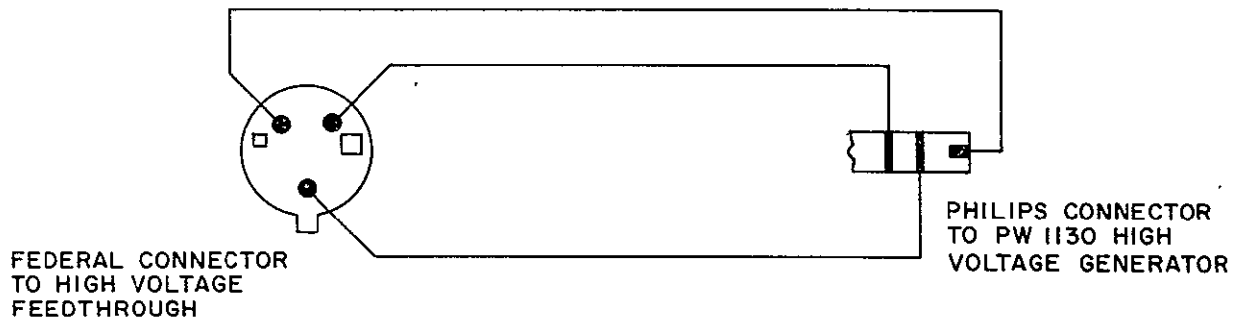
The two high voltage cables (7 in Figures 6 and 12) connecting from the high voltage generators to the high voltage feedthrough assemblies are approximately 14 feet long, and are interchangeable between the two instruments. One end of each cable is terminated with a Federal connector plug to match the connector in the high voltage feedthrough, and the other end is terminated with a plug to mate the high voltage socket in the high voltage generator. The wiring diagrams of the cables, which do not follow the usual wiring convention for medical x-ray cables, are shown in Figure 13. Replacement cables, if ever required,



(a) DIFFRACTOMETER HIGH VOLTAGE CABLE (1 REQ'D)



(b) SPECTROGRAPH HIGH VOLTAGE CABLE (1 REQ'D)



(c) GENERATOR HIGH VOLTAGE CABLE (2 REQ'D)

NOTE : CONNECTOR CONTACTS VIEWED END ON
FROM SOCKET OR SIDEWAYS.

Figure 13

Wiring connections for x-ray high voltage cables

should be made up according to the diagram of Figure 13 to avoid serious damage to the high voltage generators.

3.6.4 Other Feedthroughs and Cables

Other feedthroughs for the floor of the glove boxes are commercially available feedthroughs and are specified on drawing No. C179. These include high density multi-pin connectors for power and logic cables, shielded connectors for detector high voltage and signals, and water, gas and vacuum fittings. Signal, logic, power, and high voltage cables are separated from each other to minimize "cross-talk". These should be routed along the walls of the laboratory in appropriate trays as indicated in Figure 2.

3.7 Panel Board and Control Circuits

3.7.1 Panel Board Layout

Each panel board fulfills two functions: (1) control of instrument parameters when in the manual mode of operation; and (2) display of various instrument parameters whether in manual or computer mode of operation. The layout of the panel board for the diffractometer is shown in Figure 14. The layout of the panel board for the spectrograph is the same, except for the omission of the shutter and φ marker controls, which are not required.

There are six major divisions or groupings of controls and displays. At the top of the panel board are the "Control Transfer" group, consisting of a rocker switch and two indicator lights, and the"Emergency Off" group, consisting of a single non-illuminated red push button. When this button is depressed, both the x-ray high voltage generator and all the electronic controls

Figure 14
Panel Board

and displays are shut off. The detector electronics, however, are not affected by this push button. Below these two groups are the "Recorder" group, consisting of illuminated push buttons for raising and lowering the recorder pen and for starting and stopping the recorder chart paper drive, and the "Shutter" group, consisting of two illuminated push buttons for opening and closing the shutter in the tube tower and an indicator light which is illuminated when the x-ray high voltage is on. Below these two groups is the "Ø" or Specimen Rotation Group, which has illuminated push buttons for turning the specimen revolution marker on or off, for setting the speed of the specimen rotation (fast or slow), and for starting or stopping the specimen rotation. Below this group is the "TWO THETA" group. This consists of a numerical readout of the goniometer 2θ position (supplied by the shaft angle encoder); a series of illuminated push buttons for selecting the frequency of stepping pulses for the goniometer drive corresponding to scanning speeds of 50, 5, 2, 1, 0.5, 0.2 and 0.1 degrees 2θ per minute; illuminated push buttons for selecting forward or reverse directions of the goniometer (increasing or decreasing 2θ) and for starting or stopping the goniometer; and an indication light which is illuminated when the goniometer has been driven to an upper or lower mechanical limit.

The following color code is used for the illuminated push button and indicator lights: Amber for x-rays on and for x-ray shutter open; blue for stop buttons, x-ray shutter closed, and mechanical limit indicator; green for start buttons; and white for all others. When operating in the computer control mode, all illum-

inated push buttons lose their control function but are still used as indicators when the corresponding function is activated by the computer control, thus permitting the panel board to serve as a monitor or visual readout of the operating conditions of the system.

3.7.2 Control and Information Flow

Figure 15 shows schematically the lines of flow for the control and information functions. All such functions, except the signal from the x-ray detectors, are channelled through the control circuits which are housed within the cabinets atop the x-ray high voltage generator. Control commands are delivered from either the panel board or the computer (via the computer interface circuits, which will also be housed within the cabinets atop the generators), depending upon the status of the control gate. Information to be displayed is directly routed from the control circuits to the panel board, and information for the computer is directly routed to the computer interface circuits. The control circuits drive the stepper motor, and activate the x-ray tube tower shutter, specimen rotation device, strip chart recorder and pen lift. They also accomplish readout of the shaft angle encoder, and sense the status of the mechanical limit switches, specimen rotation mark, and x-ray shutter.

3.7.3 Technical Description

Detailed technical descriptions of the control circuits and panel boards are given in the Appendix.

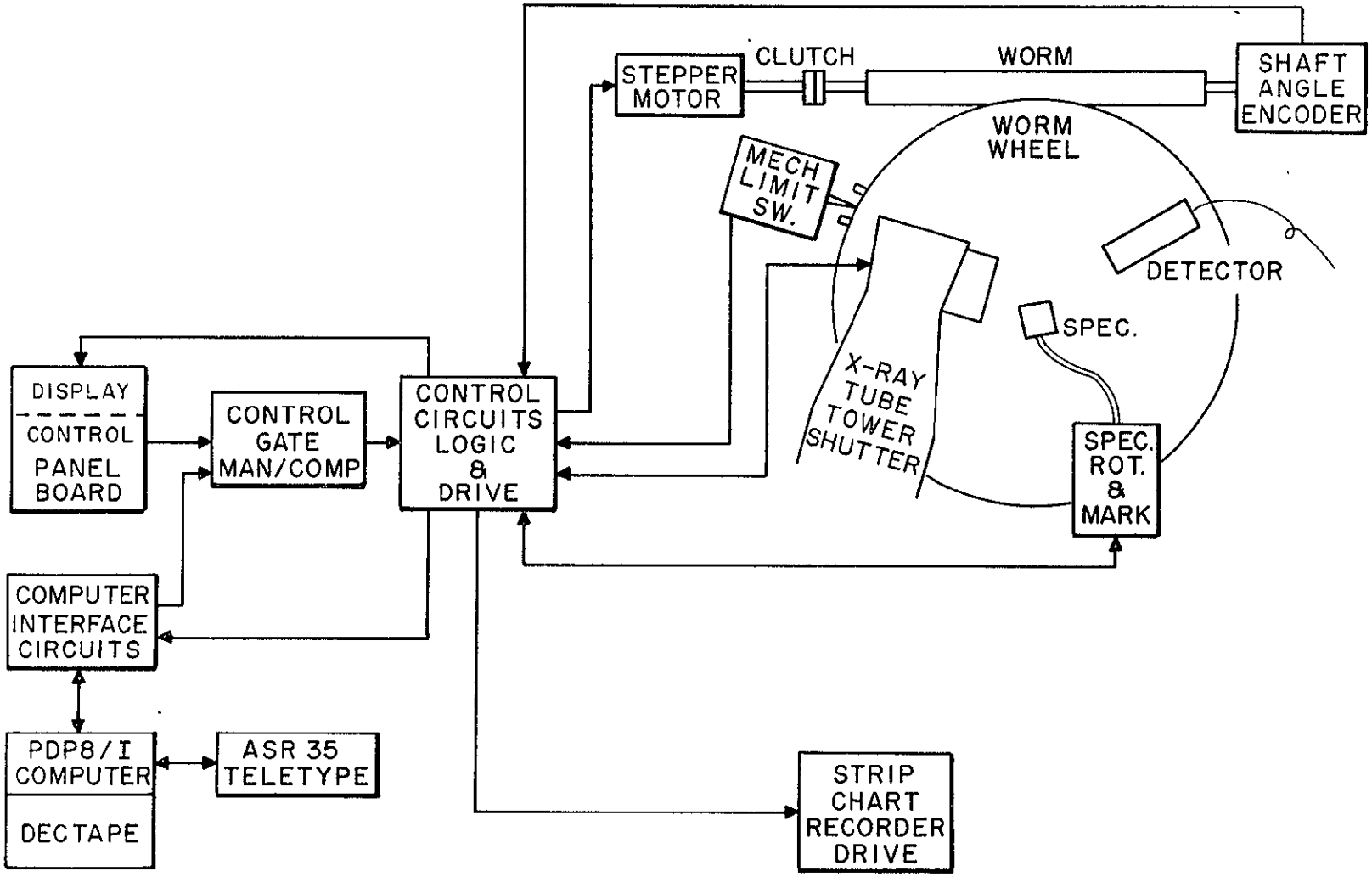


Figure 15

Diffractometer control and information flow chart

3.8 X-Ray High Voltage Generators

Two standard commercial x-ray high voltage generators, Philips Type PW 1130, are included in the system, one each for the diffractometer and the spectrograph. These generators supply full-wave bridge rectified and filtered high voltage in the range from 20 kV to 60 kV, up to a maximum current of 80 mA, subject to a maximum total power of 3 kW. High voltage setting and stabilization is accomplished by monitoring the high voltage to drive a combination motor-driven variable transformer and saturable reactor in series with the primary winding of the high voltage transformer. Current setting and stabilization is similarly accomplished by monitoring the x-ray tube current to drive a saturable reactor in the primary winding of the x-ray tube filament transformer. The stability and ripple specifications are 25 volts peak-to-peak per mA and 0.1% for both kV and mA respectively. Solid state devices, including the high voltage rectifiers, are used throughout the generator. Front panel controls on the generator include tube tower shutters timers and start buttons for the diffraction high voltage, on-off buttons, a rotary switch for setting the high voltage in 5 kV steps, a rotary switch for setting the x-ray tube current in 5 mA steps, and an auxiliary control for interpolating between mA steps. There is also a push button for activating an optional high voltage and water switch. This device is used when two x-ray tubes are to be operated alternately from this generator.

The generator interlock circuits have been modified so that the generator cannot be turned on unless the control circuits are activated. Details are given in the Appendix. Both generators

have been mounted on welded steel dollies, so as to make them readily moveable to permit servicing. A full description of the generators is given in the appropriate instruction manual.

3.9 X-Ray Detectors

3.9.1 Scintillation Counters

The scintillation counters were constructed according to designs and techniques which have been used in our Laboratories for a number of years. The scintillation crystals are thallium activated NaI, cleaved and mounted by Harshaw Chemical Co. in holders designed and fabricated by us. Parameters of the cleaved and mounted scintillation crystals are given in Table III. The photomultiplier tubes were selected for best resolution from a group of Amperex XP1110 tubes. Magnetic shielding is provided by wrapping the tubes with Conetic foil. For CuKα radiation, these scintillation counters give a pulse height distribution having a full width at half-maximum intensity of 45-48%. The preamplifier circuit is mounted at the base of the photomultiplier socket, and is described in Section 3.10 below. When mounted the complete scintillation counter assembly is insulated from ground to reduce noise pick-up.

3.9.2 Flow Proportional Counter

The flow proportional counter has been modified from that normally used with the Norelco spectrograph, by the use of a larger diameter center anode wire (125 microns) of tungsten, in order to reduce the likelihood of counting rate dependent shifts of the pulse amplitude distribution (Spielberg, 1966, 1967a, 1967b). With this modification the operating voltage necessary to detect

Table III

SCINTILLATION COUNTER PARAMETERS

	Diffractometer	Spectrograph
Beryllium window thickness	125 microns	125 microns
Scintillation crystal:		
NaI(Tl)		
Thickness	0.5 mm	1 mm
Dimensions	22 x 5 mm	22 mm diam

Aℓ K radiation (8.32 A) using P-10 gas at 1.1 atmosphere pressure is 2400 volts when using the charge sensitive preamplifier (3.4×10^{12} volts/coulomb), and a voltage gain of about 150 on the main amplifier. The normal 6μ mylar window was replaced with a 1 micron polypropylene window, using the standard Norelco thin window kit. The flow counter mounting arrangement and gas flow system has already been described above. The high voltage and signal cable from the flow counter is brought out through the gas flow system to a standard commercial preamplifier.

3.10 Detector Electronics

The detector electronic circuits follow usual practice and are shown in block diagram in Figure 16. The input selection circuit, in addition to facilitating selection of either the scintillation or flow proportional counter, also supplies power for the scintillation counter preamplifier. The schematic diagram of the input selector is shown in Figure 17. The circuit also provides a driver stage for driving low impedance inputs. The commercial components selected conform to the NIM system (AEC TID 20893 revised). Detailed descriptions are found in the manufacturer's instruction manuals.

The scintillation counter preamplifier was designed for compactness and light weight and requires only two cable connections, one for the photomultiplier high voltage and one cable for both preamplifier power and preamplifier signal. A schematic of this preamplifier is shown in Figure 18.

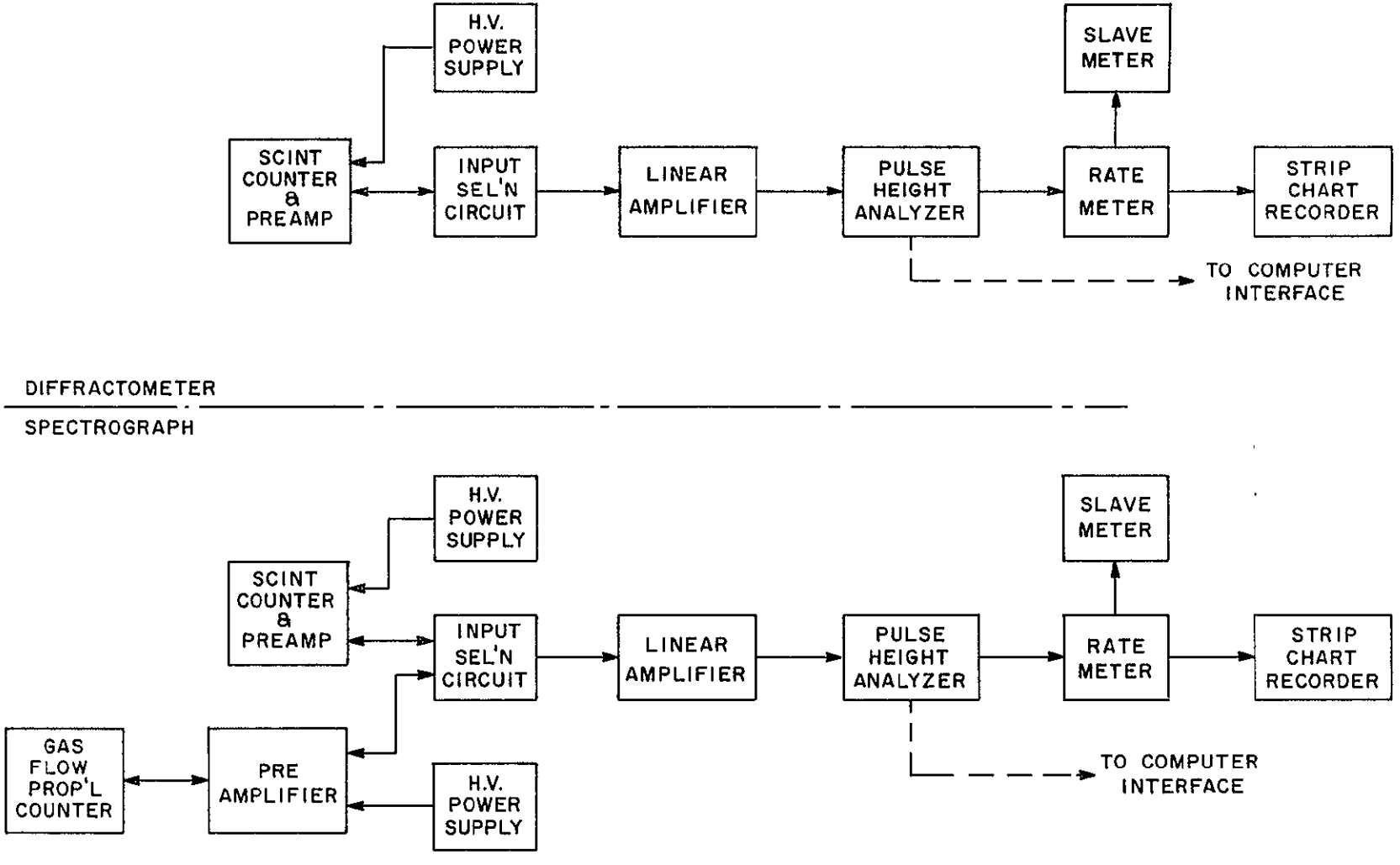


Figure 16

Detector circuits block diagram

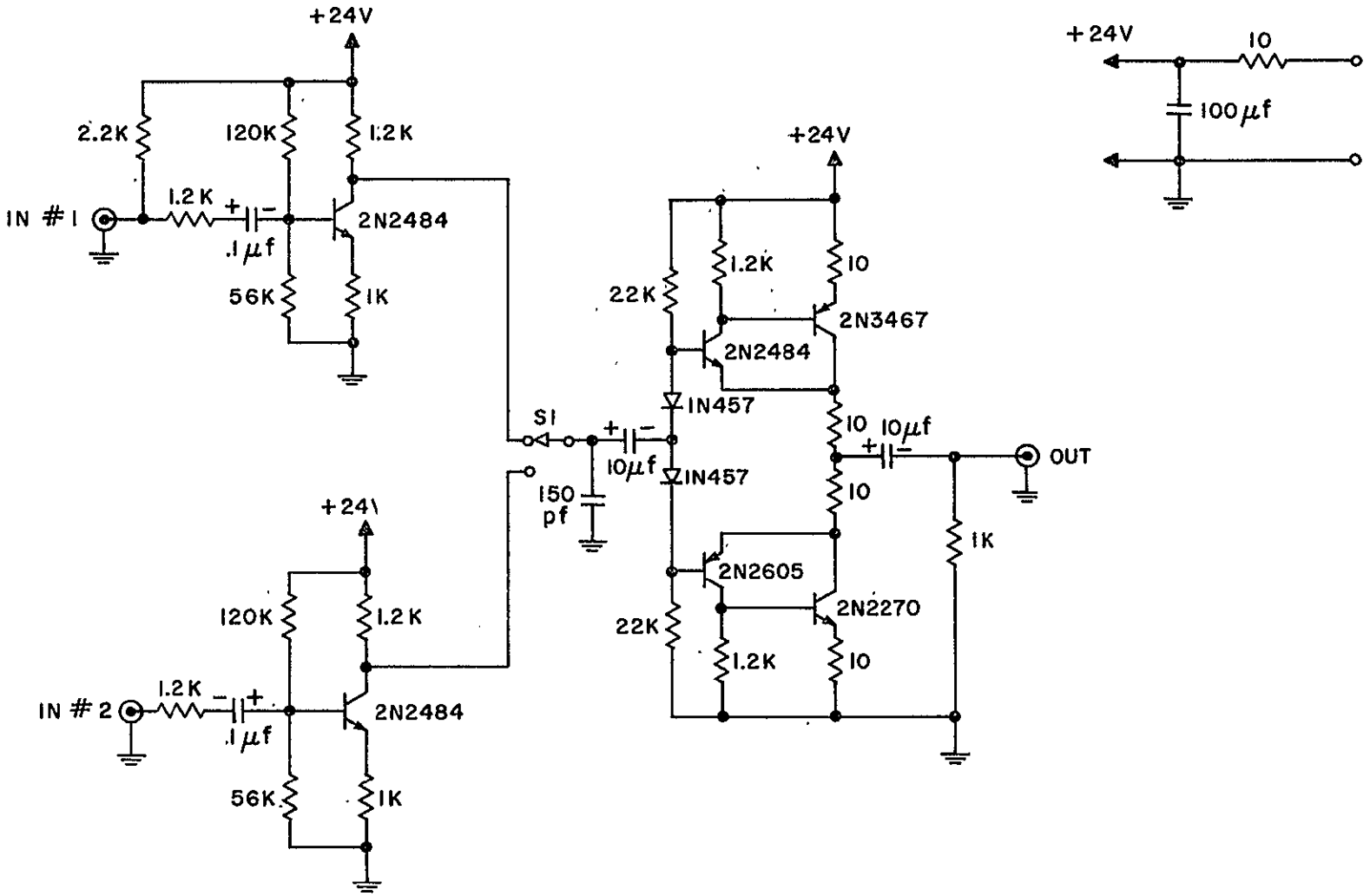


Figure 17

Schematic of input selector and impedance translator

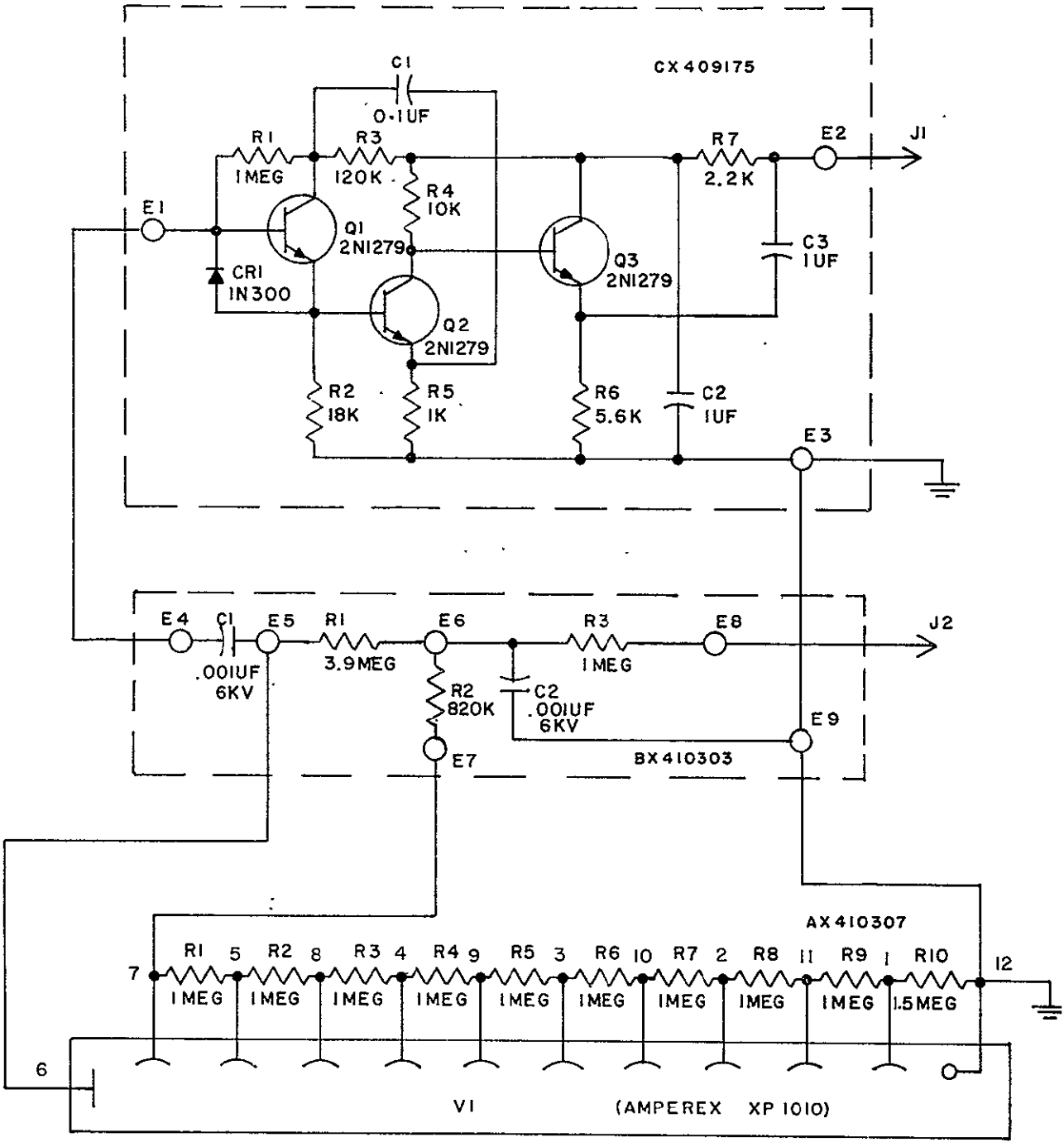


Figure 18

Circuit schematic for scintillation counter

The preamplifier for the flow proportional counter is Ortec Model 109 PC. The output from the input selection circuit is amplified by an Ortec Model 410 linear amplifier, whose output pulses are routed to an Ortec Model 406A single channel analyzer. The analyzer was modified by the manufacturer to provide a window width of up to 10 volts, rather than the 1 volt window normally supplied. The output from the single channel analyzer is then routed to a Hamner Model NR-10 ratemeter having full scale ranges from 10 to 10^5 counts per second and a zero suppression feature. The Hamner ratemeter was modified to drive a "slave" meter readout placed on a moveable stand inside the glove box. The slave meter is of considerable convenience when the instrument operator is standing at the glove box. The output of the ratemeter is used to drive a Moseley 17504A ten-inch strip chart recorder. No scaling circuits are provided since it was planned that scaling would be done by the computer. Thus the output of the single channel analyzer may be connected to the computer interface circuits. High voltage power for the scintillation counter is provided by a Fluke Model 4126 supply, and for the flow proportional counter by a Keithley Model 246 supply.

4. INSTALLATION AND ALIGNMENT

Both instruments were carefully aligned and tested before shipment. The simplest installation procedure would have been to remove the glove boxes from their normal positions at LRL and insert the instruments from the ends of the glove boxes. This was not permitted. Therefore the glass windows were removed from the glove boxes and the partially disassembled instruments were

passed through the window openings, reassembled and realigned within the glove boxes. The installation procedure is described below in some detail to facilitate reinstallation should it ever prove necessary to remove the instruments.

4.1 Diffractometer

4.1.1 Assemblies

The diffractometer was dismantled to the following assemblies for installation purposes:

A. The base plate and tube tower assembly, consisting of items 1, 2, 3, 4, 8, 16 and 17 of Figure 6 (without the x-ray tube), were passed through the glove box window, placed on the flange 5 and bolted down. The installation of this assembly required that one person be inside the glove box to support the apparatus as it was passed through the window. As indicated in Section 3.6.1 above, it was necessary to withdraw the leveling screws in the corners of the base plate.

B. The high voltage feedthrough assembly (Figure 6, item 6) was installed by first attaching the short voltage cable (item 18 of Figure 12) to the bottom of the tube tower. To assure proper alignment of the Federal connector of this cable with the mating receptacle in the high voltage feedthrough assembly, the cable nut was screwed on loosely at first (using the tool fabricated and supplied for this

purpose), the high voltage feedthrough assembly was pushed up onto the cable engaging the Federal connectors and then twisted to align with the bolt holes in the base of the tube tower support 3. The feedthrough assembly was then withdrawn carefully so as not to twist or rotate the short high voltage cable, and then the cable nut was screwed down tightly. The high voltage feedthrough assembly was then pushed up onto the cable once again and bolted to the tube tower support. The breather valve (22, Figure 12), which had been closed for shipment, was then opened. Water connections between the tube tower support and the water feedthroughs in the floor of the glove box were made using copper tubing and Swagelok fittings.

C. The goniometer assembly, consisting of the goniometer 10 and items 9, 12, 13, 14 of Figure 6 was passed through the glove box window and placed in its proper position on the baseplate 4.

D. The scintillation counter 11, x-ray tube, slave meter, and electrical cables were then installed. Before installation of the high voltage cable 7, continuity of the electrical connections between the high voltage feedthrough assembly and the x-ray tube were checked with an ohmeter. Similarly, after installation of the high voltage cable 7, continuity was checked again before inserting the

the other end of the cable into the high voltage generator. It was also necessary to check that the jumper link in the rear of the x-ray high voltage generator was placed in the proper position for the filament voltage of the diffraction x-ray tube, as specified in the instruction manual for the x-ray high voltage generator.

4.1.2 Alignment

Alignment was carried out using the devices described in 3.4.7 above, and the procedure described in detail by Parrish and Lowitzsch (1959), modified for this instrument as follows. The goniometer radius from the axis of rotation to the receiving slit has already been established in the construction of the instrument. The radius from the axis of rotation to the focal spot is set by the insertion of a gage block between the housing (16, Figure 6) of the divergence Soller slit assembly and the pin-hole assembly appropriately mounted on the goniometer axis. Positioning of the median lines of the various slits is accomplished by checking the alignment of scribe marks on the coverplates and housings of the respective assemblies with respect to the matched edges of the goniometer height fixture and the machined triangle for the Ψ angle. A spirit level is not used in the alignment procedure because the reference plane of the base plate is constrained to the mounting flange welded into the floor of the glove box. This flange is not level. A rough leveling procedure, however, is possible through the use of a protractor head from a combination square. Positioning of the divergence, anti-scatter,

and receiving slits is done during the x-ray alignment procedure, rather than during the mechanical alignment. Because the alignment procedure had been carefully carried out before shipment to LRL, only minor adjustments were required during the installation.

The zero-angle calibration and 2:1 settings are carried out using the narrowest receiving slit; the other slits are subsequently adjusted with x-rays using the recessed adjusting screws on the handwheel (Figure 6, item 12) to center them about the narrowest slit. The 2:1 setting is carried out using a double slit device having the overall shape of a specimen holder and fitting on the specimen platform (Figure 8, item 7). The top of the double slit device is covered with a scribed fluorescent screen which permits orienting of the double slit in the beam. This fluorescent screen also permits observation of the irradiated specimen areas at varous angles, thus making possible the alignment of the divergence slits. The anti-scatter slits are each individually adjusted with the diffractometer set to detect a strong diffraction line, according to the criteria given by Parrish and Lowitzsch (1959).

The alignment procedures may be carried out at higher detected intensities than those suggested by Parrish and Lowitzsch because of the modern electronic circuits now used; nevertheless the use of attenuating filters is still required.

4.1.3 Detector and Electronics Settings

It is usually desirable to set the controls for the detector high voltage power supply, signal amplifier, etc., before the

x-ray alignment is very far advanced. The settings should be made in accordance with the general principles discussed in Sections 5.5.2 and 5.5.3 below.

4.1.4 Shaft Angle Encoder Setting

The least count of the shaft angle encoder corresponds to 0.01° 2θ, which is the smallest scale division of the goniometer 2θ dial, and is also the size of a single step resulting from an advance of the stepper motor. To avoid ambiguity in the 2θ readout and control, particularly because of the slight momentary overshoot of the stepper motor, it is necessary that the indexing of the shaft angle encoder, goniometer 2θ dial, and stepper motor be synchronized to within a small fraction of a step (about 0.002° 2θ).

Synchronization of the goniometer 2θ dial and the shaft angle encoder is done by setting the dial precisely on an index mark, engaging the goniometer clutch, loosening a clamp on the encoder shaft, and rotating the shaft until its readout on the panel board corresponds to the value of 2θ given by the goniometer dial. The encoder shaft rotation should be set so that the brush giving the least count is precisely in the middle of a step. This is done by loosening the clamps on the encoder housing and rotating the housing to the correct position as judged by observing the shape of the output pulse from that brush with an oscilloscope. Synchronization with the stepper motor is accomplished by turning the power on to insure that the stepper motor is electrically clamped, disengaging the goniometer clutch, setting the goniometer manually by the hand crank precisely on an index mark, and then reengaging the clutch.

4.2 Spectrograph

4.2.1 Assemblies

The spectrograph was dismantled to the following assemblies for installation purposes:

A. The base plate and goniometer assembly, consisting of the base plate and items 4, 5 and 26 of Figures 10 and 11, was passed through the glove box window and set on the floor of the glove box (again with one person inside the glove box to support the apparatus). The goniometer 4 had been strapped to the base plate for shipping. This strap was removed after the assembly was placed in the glove box.

B. The specimen chamber-crystal chamber assembly, consisting of items 1, 2, 6, 20, 21, 22, 23, 24 and 25, was then passed through the window, set in place on locating pins in the base plate and bolted down. The link 11 between goniometer and specimen chamber was installed.

C. The high voltage feedthrough assembly was bolted to the appropriate flange in the floor of the glove box, and its vent valve opened. The W target x-ray tube was installed on the stand 26, aligned with the scribe mark in the specimen chamber, and clamped. The short high voltage cable was connected between the x-ray tube and the high voltage feedthrough, and the water

connections made between the x-ray tube and the floor of the glove box.

D. Various small parts such as the scintillation counter, slave meter, vacuum gage meter, etc., were installed, and appropriate electrical, gas and vacuum connections were made.

4.2.2 Alignment

The alignment procedure used is based on that described in the Norelco instruction manual, catalog no. 015 011 00, for the Universal Vacuum X-ray Spectrograph, but modified as appropriate to this instrument. Because alignment had been done prior to shipping, only minor adjustments were necessary at LRL. Collinearity of the goniometer and crystal chamber axes of rotation was tested by passing an aligning rod through the bearing surfaces, the direction of insertion being from the crystal chamber to the goniometer. Required adjustments were made by means of the goniometer leveling screws and displacement of the goniometer in its track. With the detector arm at the approximate 0° 2θ position, as determined by visual inspection, the scintillation detector and its associated collimator are visually adjusted to have their median lines collinear with the median line of the lower section of the dual collimator assembly. (The flow proportional counter is placed in the "advanced" position to keep it out of the x-ray beam.) The actual 0° 2θ position is determined with x-rays excited in a specimen (e.g., Cu), with the dual collimator rotated so that the high resolution section is in position, and with adjustments in the angular orientation of the detector (or exit) collimator for maximum intensity.

The first crystal sled, having one fixed crystal holder and one adjustable crystal holder, is installed in the crystal chamber. The fixed holder is the innermost holder when the sled is in place. The two crystal holders are positioned so that the crystal surfaces will be parallel to each other and oriented for measurements at small values of 2θ. The LiF 200 crystal is mounted in the fixed holder and the KAP 001 in the adjustable holder. To adjust the LiF 200 crystal, it is placed in the x-ray beam (with the flow proportional counter still in the "advanced" position), the goniometer is set to the appropriate 2θ angle for the x-ray wavelength used (e.g., CuKα) and the 2:1 adjusting device (Figure 11, item 27) employed for fine adjustment. The flow proportional counter is then placed in the "normal" position in the x-ray beam and adjusted, following the procedure given in the Norelco Universal X-ray Spectrograph manual. Adjustment of the KAP 001 crystal is preferably done with long wavelength x-rays (Aℓ or SiKα), and this is readily done with the special tools provided for this purpose. With the flow proportional counter in the normal position, a specimen providing long wavelength x-rays in place, the KAP 001 crystal is placed in the x-ray beam. The crystal chamber cover 7 is removed and the special lucite alignment cover and Allen wrench placed over the crystal chamber making sure that proper O-ring seals are in position. The goniometer is set to the appropriate 2θ angle, the alignment cover is rotated to such a position that the Allen wrench can engage the adjusting screw in the adjustable crystal holder, and the spectrograph pumped down to operating vacuum. The alignment is then made using the Allen wrench for the adjustment. The 2:1 adjusting device and the flow proportional counter

adjusting screws are not changed because they have been fixed in the earlier steps. For scanning through the peak, the Allen wrench is retracted from the crystal holder and sled so as to avoid interference with the free motion of the crystal. Bleeding of the spectrograph to normal pressure after the vacuum pump is shut off is accomplished by withdrawing the Allen wrench from the lucite alignment cover, adequate care being taken to insure that the atmosphere is let in very slowly to avoid rupturing the fragile window of the flow proportional counter.

The second crystal sled is provided with two adjustable holders, the innermost of which is positioned to be operable with the scintillation counter at low values of 2θ (flow proportional counter advanced out of the x-ray beam), using a Quartz 203 crystal. The outer holder is positioned to be operable with the flow proportional counter in the "advanced" position at high values of 2θ, using a PET 002 crystal. Adjustment of the Quartz 203 crystal is made only with the adjusting screw on the crystal holder. Adjustment of the PET 002 crystal is done by combined adjustment of its adjusting screw and the adjusting screw for the "advanced" position of the flow proportional counter, as described in the Norelco manual. The adjustment of the PET 002 crystal is preferably done with long wavelength radiation as was done in the case of the KAP 001 crystal.

4.2.3 Detector and Electronics Settings

Because of the variety of x-ray energies employed in the various parts of the spectrograph alignment, these settings are changed in the alignment procedure as appropriate. In general, for

alignment purposes, because the analyzing crystals serve as monochromators, it is usually not necessary to set the window of the pulse height analyzer. It is desirable, however, to use the attenuator on the main amplifier, as necessary, to avoid saturating the later stages of the amplifier and the pulse height analyzer. Detailed discussion is given in Section 5.5.2 and 5.5.3 below

4.2.4 Shaft Angle Encoder Setting

The same procedures described for the diffractometry are employ

4.2.5 Diaphragming and Orientation of X-Ray Tube

The two x-ray tubes supplied have been diaphragmed and scribed so that proper orientation is achieved when they are inserted into the specimen chamber. Should it be necessary to install a new x-ray tube, the following procedure is recommended. A 6.3 mm diam Pb diaphragm is placed over the window of the x-ray tube as shown in Figure 19. The mounting holes in the lead are large enough that the position of the diaphragm underneath the clamping screws can be altered. The size and position of the irradiated area is checked by irradiating a specimen of NaCℓ, which develops a brown stain under the action of x-rays. (The stain can be bleached by irradiating the NaCℓ with an infrared heat lamp.) The specimen should be prepared with a collodion solution as a binder and baked so that the salt will be thoroughly bound to the specimen holder and not spill out onto the spectrograph parts. The diaphragmed x-ray tube is installed in approximately the correct position. The specimen should be mounted on the specimen carrier 18 (Figures 10 and 11) in a

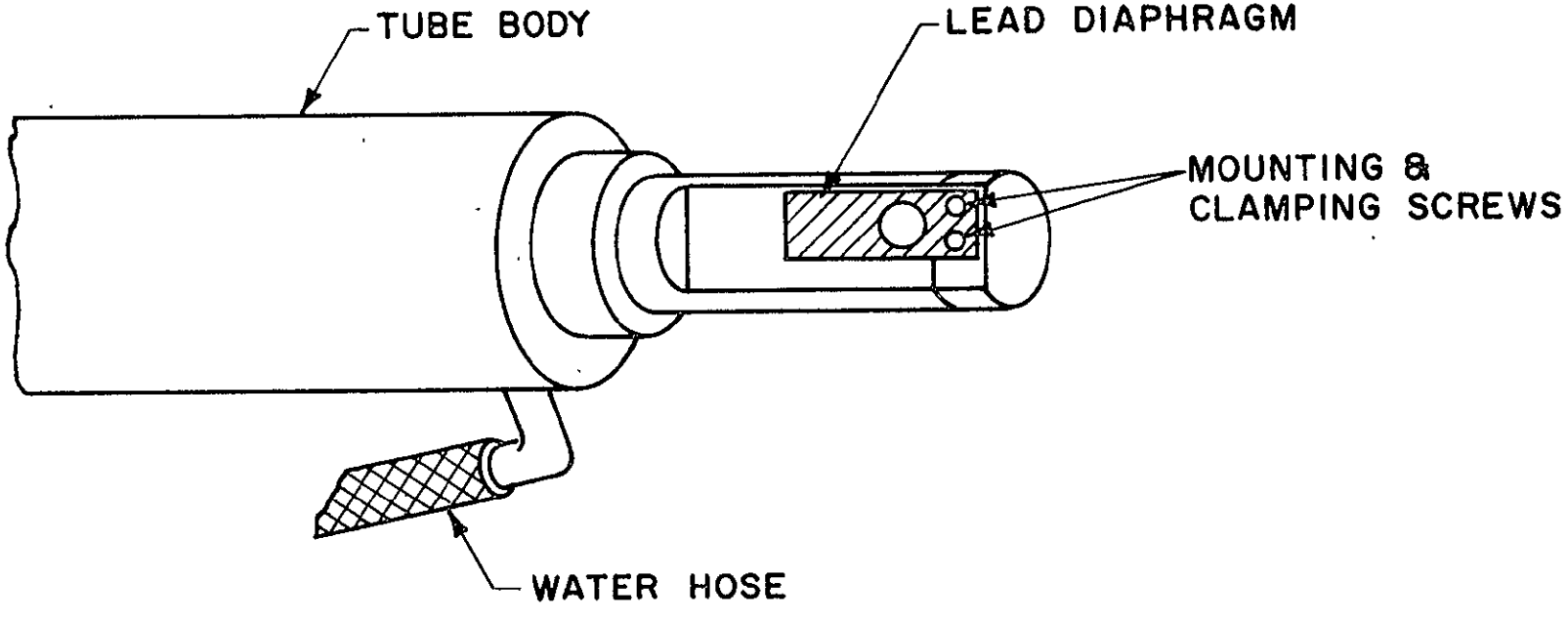


Figure 19

Diaphragmed x-ray spectrographic tube

defined orientation and this in turn in a defined orientation on the specimen insertion tool (Figures 10 and 11, item 16). The specimen insertion tool is also held in a defined orientation when inserting the specimen into one of the non-rotating specimen positions in the specimen carousel. The specimen is then turned into position beneath the x-ray tube, which is energized at 20 kV, 15 mA for about ten minutes. The x-ray tube is then shut off and the specimen removed for immediate examination of the stained area. From the known orientation of the specimen and holder when inserted, one determines in which direction to shift the diaphragm and/or alter the orientation of the x-ray tube by slight rotation about its axis. The results of the adjustment are noted with a fresh NaCℓ specimen and further adjustments are made if required. An acceptable irradiation pattern is shown in Figure 20. When an acceptable pattern is obtained, the x-ray tube should be scribed to correspond to the marks on the brass collar on the specimen chamber.

5. OPERATING INSTRUCTIONS

5.1 Control Circuits

The control circuits for each instrument are turned on from the electronics cabinet located atop the x-ray high voltage generator (Figure 2). This is done by depressing the green push button located directly above the panel containing the detector electronics modules. This activates the various control circuits, supplies power for the motors on the goniometer, illuminates the panel board (located atop the glove box) and activates interlocks on the high voltage generator. The control circuits are

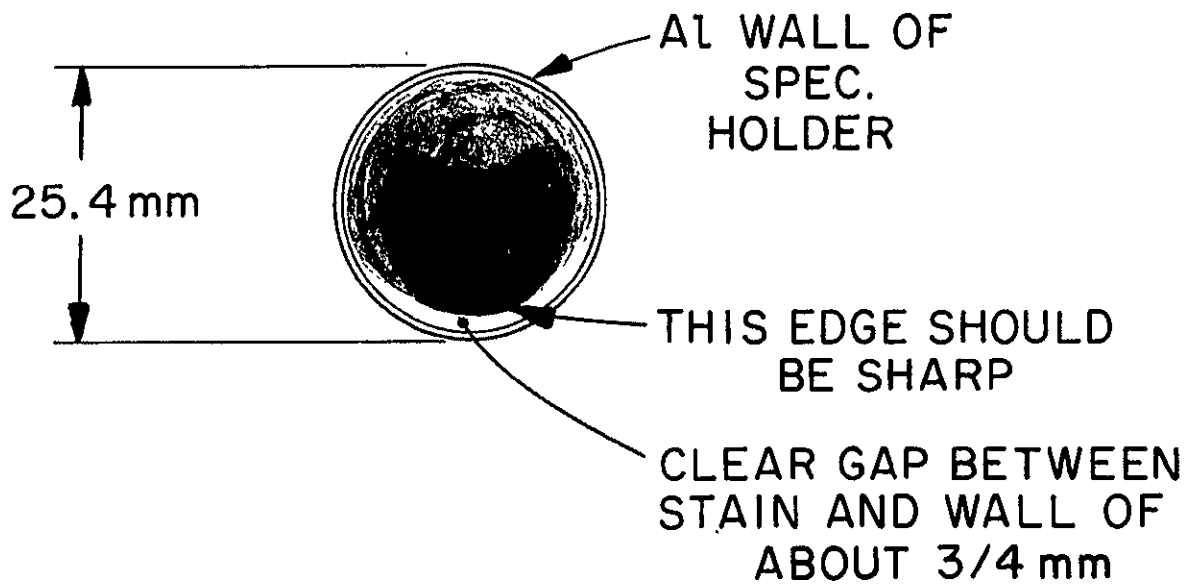


Figure 20

Irradiation Pattern of Specimen

turned off by either depressing the adjacent red push button on the same panel or by depressing the red "emergency off" button on the appropriate panel board. When the control circuits are turned off all motion of the goniometer is stopped. The high voltage is shut down.

The various detector electronics circuits including the strip chart recorder are turned on and off by their individual switches.

The instruments are placed under panel board control by depressing the "manual" side of the control transfer switch in the upper left hand corner of the panel board (Figure 14). Regardless of whether the instruments are under manual or computer control, the panel board indicates the status of the various parameters displayed. When the control circuits are first turned on, the panel board will have only white and blue indicator lights illuminated, indicating that all motors are stopped, tube tower shutter is closed, and x-ray tubes are not energized. The goniometer stepper motor (TWO THETA) and the specimen rotation motor (Ø) are started by depressing the appropriate green ("start") push button, and the tube tower shutter (diffractometer only) is opened by depressing the appropriate amber ("open") button. These functions are deactivated by depressing the blue buttons ("stop" or "closed"). The stepper motor will also stop whenever the 2θ readout coincides with one of the thumbwheel settings at the bottom of the panel board, or if the goniometer has been driven against a mechanical limit. In the latter case the blue mechanical limit indicator will be lit. The stepper motor can then be started only by reversing its direction. When

stopped by thumbwheel setting, the stepper motor may be started again by pushing the "start" button. The speeds of both motors (stepper and specimen rotation) and the direction of the stepper motor may be selected, by depressing appropriate push buttons, while the motors are either running or stopped. It is recommended that the stepper motor be stopped before reversing its direction at 50°/min. The strip chart recorder pen lift and chart drive mechanisms may also be operated from the panel board. If the Ø marker "on" button (diffractometer only) is depressed, the recorder event marker pen will be actuated after every 360° rotation of the specimen holder. (Normally the Ø marker would only be used with slow specimen rotation speed.)

5.2 X-Ray High Voltage Generators

The x-ray high voltage generators are turned on by depressing the "on" button on their control panels, provided the following pre-conditions are satisfied: the system control circuits (see above) are activated, sufficient cooling water flow and pressure are maintained, the various generator cabinet interlock switches are actuated, and the generator kV and mA selector switches are set to their minimum values. The high voltage generators are normally shut off by depressing the "off" button on the generator control panels, or shutting down the system control circuits. Detailed directions for operating the generators are given in the manufacturer's instruction manual. The diffractometer tube tower shutter is operated from the panel board and control circuits only. The other shutters on the tube tower are operated from the high voltage generator in accordance with the instructions in the manufacturer's manual.

The x-ray tube used for the diffractometer is rated at 1 kw target dissipation. Target voltage should not exceed 50 kV. The x-ray tube used for the spectrograph is rated at 2.7 kw target dissipation. The generator delivers a maximum current of 80 mA, but the target current should be limited to 70 mA at 30 kV and 40 mA at 20 kV. Target voltage may be set at any value in the 20 to 60 kV range.

5.3 Diffractometer

5.3.1 Specimen Preparation, Insertion and Removal

Normal powder diffractometer specimen preparation techniques are applicable. Although the instrument has been designed to minimize the amount of specimen preparation required to obtain a pattern, accurate analysis of the pattern is always enhanced by careful specimen preparation (Parrish, 1960). Because of the smaller radius of the diffractometer, more concern should be given to specimen surface displacement (Parrish and Lowitzsch, 1959) and related aberrations.

Insertion and removal of a specimen must be done rather carefully because of the compact design of the diffractometer. The x-ray tube tower shutter must be closed by depressing the appropriate button on the panel board. The goniometer is then turned (either by operating the stepper motor at slewing speed or by disengaging the clutch and using the hand crank) to a low value of 2θ to obtain sufficient clearance to remove the radiation shield (Figure 6, item 15). Removal and insertion of the specimen at this position is rather difficult, particularly when operating with gloves, and it is necessary to turn the goniometer back to a high enough value of 2θ so that the lower

portion of the specimen holder may be grasped with the left hand and disengaged (or engaged) from the rotatable platform (Figure 6, item 7). Care must be taken to avoid touching or disturbing the specimen surface itself. The operation is greatly assisted by making use of one of the mirrors furnished to view the specimen holder and rotatable platform. The goniometer must be returned to a low value of 2θ to replace the radiation shield. If the clutch is disengaged, care must be taken to re-engage it with the goniometer set precisely on a dial index mark, as indicated (see Section 5.3.3 below)

5.3.2 Selection of Parameters

The following optical parameters specified in Table I are subject to selection by the operator: receiving Soller slits, divergence slit, anti-scatter slit, and receiving slit. These are located within the housing 13 and on the indexed handwheels 17, 14, and 12 (Figure 6) respectively. Normally the 2 mm foil spacing Soller slits are used. Increased resolution is obtainable with the 1 mm spacing Soller slits at the price of decreased intensity. The appropriate β-filter (V for Cr target x-ray tube) should be inserted in the slot provided at the front of the Soller slit assembly. The choice of divergence slit determines the azimuthal aperture and hence has a major effect on intensity obtainable with the instrument, but there is a minimum 2θ angle for the use of a given aperture divergence slit. Table IV lists the divergence slits and the minimum scan angle and corresponding maximum value of d-spacing for which they should be used. Also listed in the table are the appropriate possible choices for the anti-scatter and receiving slits. In general, the higher

Table IV

SLIT COMBINATIONS FOR DIFFRACTOMETER

Slit No.	Divergence Slit Minimum 2θ	Divergence Slit Maximum d CrKα	Divergence Slit d CuKα	Anti-Scatter Slit No.	Receiving Slit No.
1	50°	2.71Å	1.82Å	1	1,2,3
2	30°	4.43Å	2.98Å	2	1,2,3,4
3	20°	6.60Å	4.44Å	3	2,3,4
4	10°	13.14Å	8.84Å	4	2,3,4

the number assigned to designate a given receiving slit, the higher the resolution, but the less the intensity achievable with the slit. Selection of no. 3 receiving slit, no. 3 divergence slit, and no. 3 anti-scatter slit is a generally applicable choice. The effects of the various slits on instrumental performance are discussed in the literature (Parrish and Lowitzsch 1959; Parrish and Wilson, 1959; Wilson, 1953).

Selection of scanning speed, dictated by the particular measurements required, may be made from a useful range of 0.1 to 5° 2θ per minute. The 50° per minute speed would normally be used only for slewing from one angle to another. To obtain a strip chart with 2θ increasing to the right, the scanning should be carried out in the "rev" (decreasing 2θ) direction. The ratemeter time constant should be selected to be consistent with the scanning speed so as to avoid distortion of line profiles. Limits of a scanning range should be set on the thumbwheel selector switches on the panel board. In normal work, the fast specimen rotation should be used; however, if the crystallite distribution as determined by different reflections is to be studied, then the slow specimen rotation may be used with the instrument set on a diffraction peak. For such studies the ø marker may be actuated from the panel board.

5.3.3 Clutch Engagement

As already noted, if the goniometer clutch is disengaged, then the goniometer dial should be carefully set on an indexed scale division before reengaging the clutch. This will assure unambiguous readout and synchronization between the shaft angle encoder and the stepper motor.

5.3.4 Limits of Scanning Range

Because of the short radius of the diffractometer, the goniometer should be operated at values of 2θ less than about 90° to avoid mechanically jamming the instrument. Similarly there is a lower limit of about -5 to -10°. The mechanical limit switches have been set accordingly. All significant diffraction lines for identification purposes appear within this permissible range.

5.4 Spectrograph

5.4.1 Specimen Insertion and Removal

The spectrograph has been designed to use the same specimens used in the diffractometer. As shown in Figures 10 and 11, the specimen holder 19, which is the same holder used for the diffractometer, is placed on a specimen carrier 18. This in turn is placed on the specimen insertion device 16. As shown in the figures 10 and 11 the insertion device is in its "unlocked" state, which means that the small end of the locking slide has been pressed against the center shaft of the device. Assuming that the bleeder valve in the cover 7 of the crystal chamber has been opened and that the spectrometer has been bled to ambient pressure, the cover 28 for the specimen insertion port on the bottom of the specimen chamber 1 is removed by first retracting the slide mounted pressure fingers, grasping the cover 28 with the hand and pulling it down. This is done with the aid of the mirror 17 which has been placed beneath the specimen chamber to permit viewing of the specimen insertion port. The specimen insertion device 16 with specimen carrier 18 and specimen holder 19 mounted in place, is grasped in the hand, with

the base of the device resting in the palm and the tips of the fingers around the periphery of the upper part of the device, and (using the mirror 17) is guided into place so that the upper part fits squarely into the specimen insertion port. The fingers are released while the base of the device is pushed upward, thereby seating the specimen carrier into the carousel. The carrier is seated when the pin plungers in the base of the carrier engage the peripheral groove machined in the wall of the receiving hole in the carousel. The pressure against the base of the specimen insertion device is relaxed, and the device is then withdrawn from the specimen insertion port. Care must be taken that the specimen insertion device remains in the unlocked state. When the specimen insertion device is withdrawn, inspection of the image in the mirror 17 of the base of the specimen carrier must verify that the specimen carrier has been properly seated. The cover 28 for the specimen insertion port should be replaced, pushing up firmly to make sure that its pin plungers are engaged in their retaining groove, and the slide mounted pressure fingers are pushed forward underneath the surface of the cover, thereby pressing the cover upward against a gasket. By means of the large knob underneath the specimen chamber, the specimen carousel is turned to bring the specimen into position beneath the x-ray tube. (The Norelco instruction manual may be consulted also, but it should be kept in mind that this instrument configuration is inverted from that used in the normal Norelco instrument.)

To remove a specimen from the instrument, the carousel is turned to bring the specimen into position at the specimen insertion port, the vacuum is released, and the port cover 28 removed.

The specimen insertion device, still in the "unlocked" stage, is inserted into the port, the base of the device is pushed upward with the palm of the hand so that the internal fingers are inserted into and expanded against the recess on the bottom side of the specimen carrier, and the device is then locked by using the thumb to remove the large end of the locking slide against the center shaft of the device. This ensures that the specimen insertion device firmly grips the base of the specimen carrier, so that it may then be withdrawn from the carousel by pulling the specimen insertion device downward. After the specimen carrier is withdrawn, it may be released from the specimen insertion device by pushing the small end of the locking slide against the center shaft. Occasionally this will not suffice to release the specimen carrier, particularly if the insertion device was canted when the specimen carrier was gripped for withdrawal. In such a case, a slight rotation of the knurled ring at the base of the specimen insertion device will serve to withdraw the internal fingers. It will then be necessary to return the knurled ring to its original position for handling other carriers. It is usually best to remove the diffractometer specimen holder 19 itself from the specimen carrier before releasing the locking slide in order to avoid jarring the specimen unduly when the internal fingers spring back into the unlocked state.

5.4.2 Evacuation of the Spectrograph

In pumping out the spectrograph, and also in bringing it back to ambient pressure, care must be taken to avoid rapid changes in pressure which may rupture the mylar window on the crystal chamber or the even more delicate one-micron polypropylene window on the flow proportional counter. Appropriate valving

should be provided in the line leading to the vacuum pump to ensure that initial evacuation is not done too rapidly. Similarly, when the instrument is bled to ambient pressure, the bleeder valve in the crystal chamber cover should not be opened too rapidly. The vacuum is monitored by the Hastings vacuum gage placed within the glove box. It should be noted that the thermocouple sensor for this vacuum gage is located very close to the crystal chamber and thus gives an accurate indication of the vacuum within the instrument. As long as the instrument is kept fairly "clean", little difficulty should be encountered in obtaining vacuum gage readings less than 0.5 mm Hg.

5.4.3 Selection of Parameters

The choice of collimator, crystal, and detector is made by means of the appropriate knobs and handles, as indicated in the Norelco instruction manual. For the widest range of applications, the most useful crystals of those furnished are the LiF 200 and the KAP 001. These crystals have been mounted on their crystal slide in such a way that either the scintillation counter or the flow proportional counter may be used with them. If it is desired to obtain higher resolution than is possible with these crystals with the collimator selector knob in its "fine" position, then the slide carrying the Quartz 203 and PET 002 crystals should be installed. This is done by removing the cover (Figures 10 and 11) of the crystal chamber (being careful not to disturb the position of the collimator selector knob), positioning the goniometer so that the crystal slide 10 can be withdrawn without interference from the flow counter, pushing up on the positioning arm in the assembly 9 by means of the

lever provided, and withdrawing the crystal slide. The slide carrying the Quartz 203 and PET 002 crystals is inserted on the bars of the crystal holder support, and the positioning arm is pulled down again. Since the crystal holders were aligned during the initial installation, it should not be necessary to realign the instrument on changing crystal slides, if the change is carried out carefully. The cover 7 of the specimen chamber should then be replaced.

Similar considerations to those applicable to the diffractometer may be used to guide the speed and direction of scan, and in the choice of specimen rotation speed. The slow speed may be used to orient a specimen in its own plane for possible enhancement of intensity from a particular constituent of the specimen. The measurements would then be carried out with the specimen not rotating further. The Ø marker is not provided with the spectrograph. Also there is no shutter control for the spectrograph, there being no shutter on the instrument. The same care in engaging and disengaging the goniometer clutch should be used with the spectrograph as with the diffractometer.

5.4.4 Limits of Scanning Range

The goniometer should not be driven to values of 2θ much above 90° in order to prevent the scintillation counter from striking the base plate. The mechanical limit switches have been set to prevent this eventuality. With the crystals furnished with the instrument, this restriction is of no consequence (see Table II and Section 3.5.2). When the PET 002 crystal is used, the flow proportional counter is advanced 30° from the goniometer reading,

and its maximum 2θ is therefore 120°. Since the lightest element measurable with the PET 002 crystal is Si, for which the K_α line appears at 2θ = 109°, the scanning range restriction causes no problem.

5.5 Detectors and Associated Electronics

5.5.1 Diffractometer Scintillation Counter

The diffractometer should be set at the peak of a strong powder line or, alternatively, a radioactive source (Fe^{55}, emitting Mn K radiation) may be used for preliminary settings when the high voltage generator is off. The source should be placed over the window of the scintillation counter. The switch on the input selection circuit should be set at position 1. Detailed instructions on the various other components are found in their manufacturers' manuals. See Figure 16. The settings of the high voltage power supply, amplifier controls, pulse height analyzer, etc., are not particularly critical, although it is preferable that the pulses being selected by the pulse height analyzer have their average size in the lower part of the analyzer range to minimize overloading and base line shift effects. Thus an average pulse height input to the analyzer for pulses due to CrKα radiation of the order of 2 or 3 volts is quite satisfactory. The lower level and window discriminators should be set to pass 97-99+% of the pulse distribution from Cr K radiation. Similarly, operation of the scintillation counter at a power supply voltage in the range from 850 to 1000 volts gives satisfactory performance with the photomultipliers and preamplifiers used. (Note positive polarity is used.) The Ortec Model 410 main amplifier should be operated in the double delay line mode, with

integration time constant "out", negative input polarity, and appropriate gain and attenuation settings. Typical values are: coarse grain 1, fine grain 3.0, input attenuator 2; but these are not critical. The unipolar 93 ohm output is used to supply input signals to the pulse height analyzer. The ratemeter and strip chart recorder are used as required. The ratemeter signal selector switch should be set for positive pulse input.

5.5.2 Spectrograph Scintillation Counter

The spectrograph should be set at the peak of a strong spectral line from a major constituent of a specimen, with the input selection circuit set at position 1. If it is desired to set the pulse height analyzer to pass primarily one particular energy radiation, then the same procedure should be followed as for the diffractometer scintillation counter. As often used however, particularly when scanning for a range of elements, the discriminators should be set for much wider limits, or it may even be desirable to operate the pulse height analyzer in the integral mode. Because of the wide range of energies of the spectra used in x-ray spectrochemical analysis, and in order to minimize the number of settings required, it is preferable to set the controls for the scintillation counter detector circuits first for the lowest energy to be detected, and to use the input attenuator switch on the Ortec Model 410 main amplifier to reduce the size of the pulses obtained from the detection of higher energy radiations. Thus, if Ti Kα radiation (4.5 keV) is being detected with the input attenuator set at 5, giving an average output pulse height of 2 volts, then for Sn Kα (25 keV) radiation, the attenuator should be set at 20, giving an average output pulse

height of 2.8 volts. (An attenuator setting at 10, while giving an average output pulse height of 5.6 volts, well within the range of the amplifier, would mean that even higher energy radiation, due to scatter and higher order reflections, if incident on the detector in sufficient intensity, would overload the circuits.) The settings given here are consistent with the settings given below for the flow counter, and thus minimize the changes required when switching from one counter to the other.

5.5.3 Spectrograph Flow Proportional Counter

There are some conflicting requirements which should be considered in selecting the operating parameters for the flow counter. It is desired on the one hand that the gas multiplication be kept at a minimum to reduce the likelihood of counting rate dependent shifts in the pulse height distribution (Spielberg, 1967a); on the other hand it must be high enough that the resulting pulses are roughly an order of magnitude larger than the pulses induced by various switching and noise transients. The operating voltage should not exceed the limits imposed by the insulation and surface leakage characteristics of the various components. The settings given below represent a satisfactory compromise among these requirements.

The delivery pressure from the P-10 gas supply source should be set at about 2 to 4 psig. The needle valve on the gas flow meter (Figures 10 and 11, item 22) is then opened sufficiently so that the gas flow is greater than the full-scale reading of the flow meter (2.0 cfh). After a few minutes of flushing at this rate, the needle valve is adjusted so that the indicated flow rate is

about 0.1 or 0.2 cfh. The gain of the Ortec 109PC amplifier (Figures 10 and 11, item 23) is set at X10, and the input selection circuit is set at position 2. The main amplifier gain controls and the high voltage supply (Keithley Model 246) are set for the lower energies to be detected, making their average pulse height fall in the lower part of the range of the pulse height analyzer. The attenuator control is then used to reduce the pulse heights from higher energy radiations to values compatible with the 0 to 10 volt input pulse range of the pulse height analyzer. Some typical choices of parameters for AℓKα radiation (1.5 keV) are as follows: Keithley high voltage supply - 2400 volts (positive polarity); Ortec linear amplifier - negative input polarity, double delay line mode, integration time constant "out", coarse gain 1, fine gain 3.0, input attenuator 1. These settings gave an average pulse height at the pulse height analyzer of about 2.2 volts. Using the same settings, but with the amplifier input attenuator set at 10, the average pulse height for CuKα radiation (8 keV) was about 1.8 or 1.9 volts.

The same considerations as discussed for the scintillation counter apply to the settings of the pulse height analyzer when using the flow proportional counter.

6. PERFORMANCE AND RESULTS

Only a limited amount of time was available for testing the diffractometer and spectrograph, both at Philips Laboratories and at LRL.

6.1 Diffractometer

6.1.1 Quartz Specimens

A number of charts were obtained for specimens consisting of pure SiO_2 powder and these show very clearly the high intensities and resolution obtainable from well crystallized specimens. Figure 21 is such a chart, obtained using $CrK\alpha$ radiation with a V filter at 40 kV, 20 mA, and divergence, antiscatter and receiving slits #3 (see Tables I and IV). Full scale deflection represents a counting rate of 10,000 counts per second. It is apparent from the chart that net peak intensity for the 100 reflection is about 9300 cps. The net peak intensity for the 111 reflection is in excess of 60,000 cps. Table V gives a comparison of intensities obtained from the 111 reflection from specimens of SiO_2 powder. It should be noted that with a specimen containing about 1 milligram of sample a counting rate of 13,600 counts per second was obtained under high resolution conditions.

6.1.2 Apollo XI Samples

At the time of installation some lunar samples from the Apollo XI mission were made available. A specimen was prepared and a diffractometer chart obtained. This chart featured good diffraction intensities and line shapes, indicating well crystallized material. Due to limited time and reference data available at the installation site, an analysis of the diffraction pattern was not attempted. This chart has not been released to us and is therefore not included in this report.

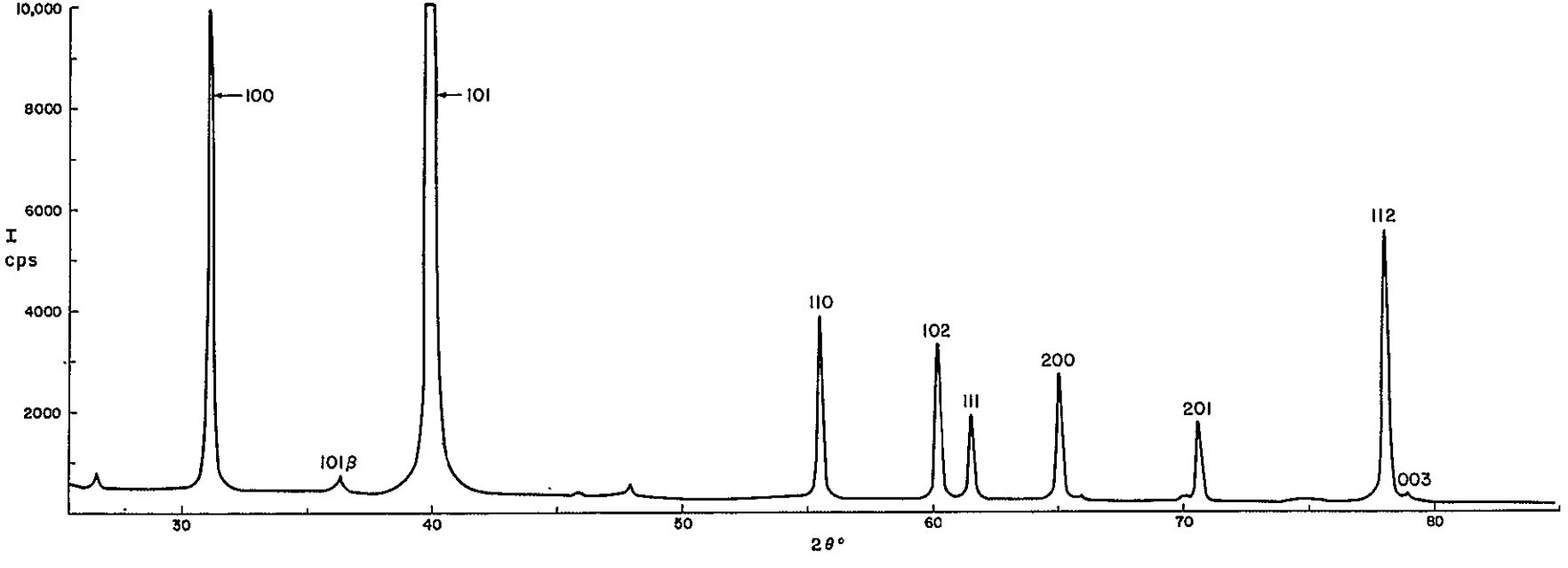


Figure 21

Diffractometer chart SiO_2 CrKα radiation

Table V

DIFFRACTED INTENSITIES FOR 111 REFLECTION FROM SiO_2 SPECIMENS

Experimental conditions: $CrK\alpha$ radiation, V filter, 40 kV, 25 mA, receiving Soller slits $\delta = 4.8°$

Slit Selections (See Table I) Diverg.	Anti-Scatter	Rec'g	Width of line (at 1/2 peak intensity) (deg. 2θ)	Net Peak Intensity (Counts/Sec)
Thick specimen:				
2	2	2	.32	76,000
3	3	3	.24	51,000
4	4	4	.23	16,000
Thin specimen ~ 1 mg:				
4	4	4	.19	13,600

6.2 Spectrograph

6.2.1 Pure Thick Specimens

With a "thick" specimen of Cu powder, using the W target x-ray tube at 20 kV, 5 mA, fine collimator and the LiF 200 crystal, the peak intensity for the CuKα line was 14,500 cps. The line width at half maximum intensity was 0.42° 2θ (peak position 45° 2θ). With a "thick" SiO_2 specimen, W target x-ray tube at 40 kV, 20 mA, coarse collimator, KAP 001 crystal, the net intensity at the peak of SiKα line (2θ = 31°) was 1750 cps. The line width at half maximum intensity was 0.7° 2θ.

6.2.2 Pure Thin Specimens

With a "thin" diffractometer specimen, estimated to contain about one milligram of SiO_2 powder, W target x-ray tube at 40 kV, 60 mA, specimen carrier having W "reradiator" (see Section 3.5.6) the net peak intensity for the SiKα line was 3250 cps over a background of 250 cps. Assuming that a net peak intensity of 10% over the background is detectible, this result implies sensitivity to 10 micrograms of SiO_2. Tests of the effectiveness of the "reradiator" indicated that it yielded at best a 10% increase in net peak intensity, but at the price of 20-40% increased background.

6.2.3 Apollo XI Samples

Using the same specimen prepared for the diffractometer (see Section 6.1.2), all the major constituents (within the range of the instrument) reported from optical spectrographic measurements were observed, in particular the titanium and iron constituents. No quantitative estimates were possible because of the lack of calibration standards.

7. REFERENCES

1. Henke, Burton L., (1963) in Advance in X-ray Analysis, Vol. 6, William M. Mueller and Marie Fay, eds., Plenum Press, New York, pp. 361-376.

2. Parrish, W. (1959) in Seventh Natl. Conf. on Clays and Clay Minerals, Pergamon Press, New York, pp. 230-259.

3. Parrish, William (1960) in Proc. Fifth International Instruments and Measurements Conference, Stockholm, pp. 346-359.

4. Parrish, William (1968). X-Ray Analysis of Lunar Materials, Philips Labs Technical Report No. 227, NASA Contract NASW-1477, September 1968.

5. Parrish, W. and K. Lowitzsch (1959). American Mineralogist 44, 765-787.

6. Parrish, W. and A. J. C. Wilson (1959). International Tables for X-Ray Crystallography, Vol. 2, Kynoch Press, Birmingham, England, pp. 216-234.

7. Spielberg, N. (1959) Philips Research Reports 14, 215-236.

8. Spielberg, N. (1962) Jour. Appl. Phys. 33, 2033-2035.

9. Spielberg, N. (1966) Rev. Sci. Instr. 37, 1268-1269.

10. Spielberg, N. (1967a) Rev. Sci. Instr. 38, 291.

11. Spielberg, N. (1967b) in Advances in X-ray Analysis, Vol. 10, John B. Newkirk and Gavin R. Mallett, eds., Plenum Press, New York, pp. 534-545.

12. Spielberg, N. and M. Mack (1958) Norelco Reporter 5, 109.

13. Spielberg, N., W. Parrish and K. Lowitzsch (1959) Spectrochim Acta 13, 564-583.

14. Wilson, A. J. C. (1953) Mathematical Theory of X-Ray Powder Diffractometry, Centrex Publishing Co., Eindhoven, Netherlands.