# TECHNICAL MANUAL

PERKIN ELMER

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# OPERATOR SAFETY SUMMARY

All PHI Systems have been designed to assure operator safety. However, like all other sophisticated instruments, continual operator safety is dependent on the proper use of system components. Such use is described in the manuals supplied with each unit.

LISTED BELOW ARE WARNINGS APPLICABLE TO THIS EQUIPMENT. ALL PERSONNEL INVOLVED IN THE OPERATION AND MAINTENANCE OF THIS EQUIPMENT MUST FULLY UNDERSTAND THE WARNINGS AND THE PROCEDURES BY WHICH THE HAZARD IS TO BE REDUCED OR ELIMINATED.

**WARNING**

THE PRODUCT(S) COVERED IN THIS MANUAL HAS BEEN DESIGNED TO FUNCTION SAFELY WITH THE REQUIRED DEGREE OF PRECISION WHEN USED IN THE PRESCRIBED MANNER.

WE DO NOT RECOMMEND THAT THIS EQUIPMENT BE MODIFIED FOR ANY NON-STANDARD APPLICATION SINCE HAZARDOUS CONDITIONS MAY RESULT. DUE TO THE FACT THAT PHYSICAL ELECTRONICS DIVISION OF PERKIN-ELMER HAS NO CONTROL OVER CUSTOMER MODIFICATIONS TO PHI PRODUCTS SHIPPED, IT DISCLAIMS ANY RESPONSIBILITY FOR ANY MALFUNCTIONS OR ACCIDENTS THAT MAY RESULT!

**DANGER**
**ELECTRICAL SHOCK HAZARD**

HIGH VOLTAGES ARE PRESENT IN THE SYSTEM WHEN THE SYSTEM POWER INPUT LINES ARE CONNECTED. DISCONNECT INPUT POWER AT THE WALL BEFORE MAKING ANY ADJUSTMENTS. REFER SERVICING TO PERSONNEL WHO HAVE BEEN TRAINED AND HAVE WORKING EXPERIENCE WITH VOLTAGES IN EXCESS OF 50 VOLTS.

ALL ELECTRICAL CABLES ASSOCIATED WITH VARIOUS UNITS INCLUDED IN A SYSTEM ARE WELL SHIELDED. HOWEVER, CARE MUST BE TAKEN NEVER TO COME IN CONTACT WITH ANY ASSOCIATED TERMINALS WHEN THE POWER IS ON. SOME OF THESE LEADS CARRY POTENTIALLY LETHAL HIGH VOLTAGES. OTHER LEADS MAY CARRY SUFFICIENT RF POWER TO INFLICT SEVERE BURNS.

*RF INTERFERENCE*

*THIS EQUIPMENT GENERATES, USES, AND CAN RADIATE RADIO-FREQUENCY ENERGY, AND IF NOT INSTALLED AND USED IN ACCORDANCE WITH THE INSTRUCTION MANUAL, MAY CAUSE INTERFERENCE TO RADIO COMMUNICATIONS. OPERATION OF THIS EQUIPMENT IN A RESIDENTIAL AREA IS LIKELY TO CAUSE INTERFERENCE IN WHICH CASE THE USER AT HIS OWN EXPENSE WILL BE REQUIRED TO TAKE WHATEVER MEASURES MAY BE REQUIRED TO CORRECT THE INTERFERENCE.*

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# SECTION I
# INTRODUCTION

## 1.1 GENERAL INFORMATION

This manual is intended to assist users in the installation, operation, and maintenance of the PHI Model 10-410 Toroidal Monochromator. The manual is divided into five sections. Section I contains a brief description and specifications of the unit. Section II refers to checkout procedures and installation. Section III describes the operation of the toroidal monochromator while Section IV explains the theory of operation. Calibration and maintenance are discussed to Section V.

## 1.2 GENERAL DESCRIPTION

The PHI Model 10-410 Toroidal Monochromator is designed to be used on the PHI 5000 Series ESCA Systems and consists of a crystal assembly, a crystal manipulator and the monochromator housing. The monochromator housing contains two flanges for mounting onto the test chamber and the x-ray source. Mounting on test chambers of other systems can also be done, if they have a 6.95 inch port length and adequate clearance conditions.

The toroidal monochromator's crystal assembly and crystal manipulator are both mounted in the monochromator housing. The crystal assembly is composed of a set of precisely oriented quartz crystals. This set of crystals is aligned by the crystal manipulator, to produce a monochromated x-ray beam. The manipulator has three separate motions for crystal alignment: translation and two directions of tilt. A shutter is provided on the monochromator housing to shield the crystal assembly and monochromator during sputtering.

The monochromator provides the following advantages over the standard dual-anode x-ray source: improved energy resolution, lower spectral backgrounds, the absence of certain satellites and "ghost" peaks, and reduced sample damage.

## 1.3 SYSTEM CONFIGURATION

Figure 1-1 shows how the toroidal monochromator package interfaces with the analytical system. The package consists of the PHI Model 04-500 X-Ray Source, a linear motion device which connects the x-ray source to the monochromator, and associated cables and water lines. The toroidal monochromator bolts to, and is supported by, a six inch flange on the bell jar. If the system configuration includes a standard PHI Model 04-548 X-Ray Source, the PHI Model 22-040 High Voltage Supply is wired to both sources and switching between the two sources is controlled either manually or by the computer through the PHI Model 32-095 X-Ray Source Control. In order to use the alternate side of the dual anode source, the monochromator filament connector is moved to the dual anode source connector. In order to operate both sides of the standard source simultaneously, a standard filament cable is provided with the system. Anode cooling is provided by the standard PHI Model 16-020 Heat Exchanger with the two sources plumbed in series.

Because the x-ray source protrudes from the front of the instrument, a protective guard is provided to prevent damage to either the user or the instrument.

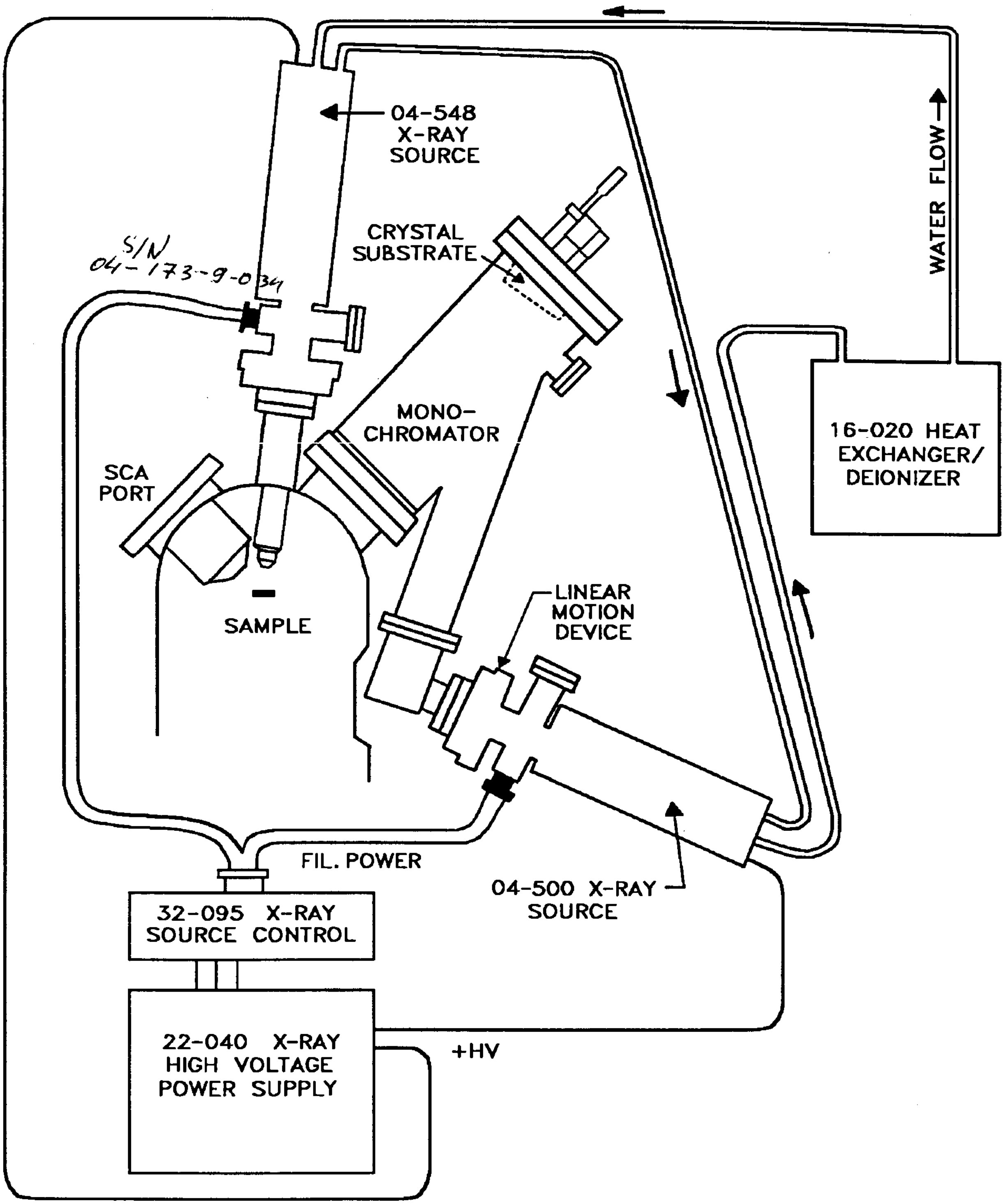


*Figure 1-1. PHI Model 10-410 Toroidal Monochromator Package/System Interface.*

## 1.4 SYSTEM SPECIFICATIONS

### 1.4.1 Count Rates and Resolution

The Al monochromator operating at 15 kV on a smooth, sputter-cleaned sample of silver (Ag $3d_{5/2}$ peak), will meet or exceed the curve defined by the following performance specifications for a 1.1 mm dia. aperture size:

**Omni-Focus Lens in high solid angle mode**

| Anode Power | Detector | Resolution FWHM (eV) | Sensitivity cps |
|---|---|---|---|
| 600 | PSD | ≤ 0.50 | 12,000 |
| 600 | PSD | ≤ 0.60 | 20,000 |
| 600 | SCD | ≤ 0.50 | 5,000 |
| 600 | SCD | ≤ 0.60 | 7,500 |

### 1.4.2 Optics Adjustments

The monochromator has the following adjustments for optical alignment:

| | |
|---|---|
| X-Ray Anode: | Linear Motion<br>Tilt. |
| Monochromator Crystal: | X- Tilt<br>Y- Tilt<br>Focus. |

### 1.4.3 Vacuum Performance

| | |
|---|---|
| Bakeout Temperature: | 150° C. |
| Base Pressure: | $< 5 \times 10^{-10}$ Torr. |

### 1.4.4 Other

| | |
|---|---|
| Maximum Anode Power: | 700 watts. |
| Room Temperature: | 20° C ± 5° C. |

# SECTION II
# INITIAL CHECKOUT AND INSTALLATION

## 2.1 GENERAL INFORMATION

The housing and crystals for the PHI Model 10-410 Toroidal Monochromator are shipped separately. Installation is required for the unit and must be performed by PHI service personnel.

Visual inspection of the system is required to insure that no damage occurred during shipping. If damage did occur, contact the freight company immediately.

# SECTION III
# OPERATION

## 3.1 GENERAL INFORMATION

This section contains information on using the toroidal monochromator. Various x-ray source preliminary operating procedures are required whenever a new x-ray source or filament has been installed, when the x-ray source has been exposed to air or when the x-ray source has not been operated for several months. Refer to Section III of the PHI Model 04-500/04-548 X-Ray Source manual for detailed procedures.

## 3.2 ALIGNMENT UNDER VACUUM

The toroidal monochromator has five adjustments. Three adjustments are located on the top of the monochromator housing: the x-tilt, y-tilt and focus. They are used to position the crystal assembly. Two adjustments, the linear motion and tilt, are used to position the x-ray source.

1. Perform the microscope calibration procedure before aligning the monochromator. (Refer to the calibration and maintenance section of the system manual for your instrument.)

2. Introduce a clean, flat silver specimen into the system.

3. Using the Al side of the dual anode x-ray source, scan the Ag $3d_{5/2}$ peak at 368 eV binding energy to determine the exact location of the peak maximum. Use a pass energy near 20 eV and the largest lens aperture. The peak should be slightly assymetric, with the maximum occurring just to the right of peak center. Note the binding energy of the peak.

4. Switch from the dual anode to the monochromatic x-ray source.

5. If the monochromator is far out of alignment, it is easiest to find the x-ray beam with a phosphor screen. In this case, replace the silver specimen with a phosphor screen (Part No. 618646) and perform steps 6 - 13. If some signal can be found with the monochromator x-ray source, skip steps 6 - 13 and proceed with step 14.

6. Adjust the x-ray source as follows. Center the tilt control by setting it to its mid-range position. Extend or retract the linear motion to give a distance of approximately 2.6 inches between the black plates (refer to Fig. 3-1).

7. Center the focus adjustment on the monochromator housing by setting it to its mid-range position (see Figs. 3-2 and 3-3).

8. Open the monochromator shutter by rotating the shutter feedthru fully counterclockwise (Fig. 3-2).

9. Turn on the monochromator x-ray source to 200 watts or more.

10. Adjust the x- and y-tilts on the monochromator housing (refer to Figs. 3-2 and 3-3) until a yellow-orange light appears on the phosphor screen. This light is from the reflection of the x-ray source filament off the crystal surface and appears elongated in the x-direction. Use the x-tilt to center the light on the phosphor.

11. Adjust the x-ray linear motion and the monochromator y-tilt until a blue light appears on the phosphor screen. The two adjustments must be made simultaneously to maintain the yellow-orange light on the phosphor. The blue light is produced by fluorescence from Al $K\alpha$ x-ray excitation. It will appear approximately 3 mm from the filament light, in the direction toward the monochromator.

12. Use the linear motion and y-tilt to center the blue light in the y-direction and to maximize its intensity. Use the x-tilt to center it in the x-direction.

13. Use the focus adjust and the y-tilt to obtain the sharpest and brightest image possible. Repeat step 12 to recenter the light, if necessary. Replace the phosphor screen with a clean, flat silver specimen.

14. Position the silver specimen at the focal point of the analyzer and sputter to remove carbon and oxygen contamination. Adjust the specimen tilt angle to 45° (horizontal).

15. Connect a picoammeter to measure target current.

16. Select the **[Setup Align]** software command to display the ESCA Alignment Menu and set up a narrow window acquisition range around the 368 eV

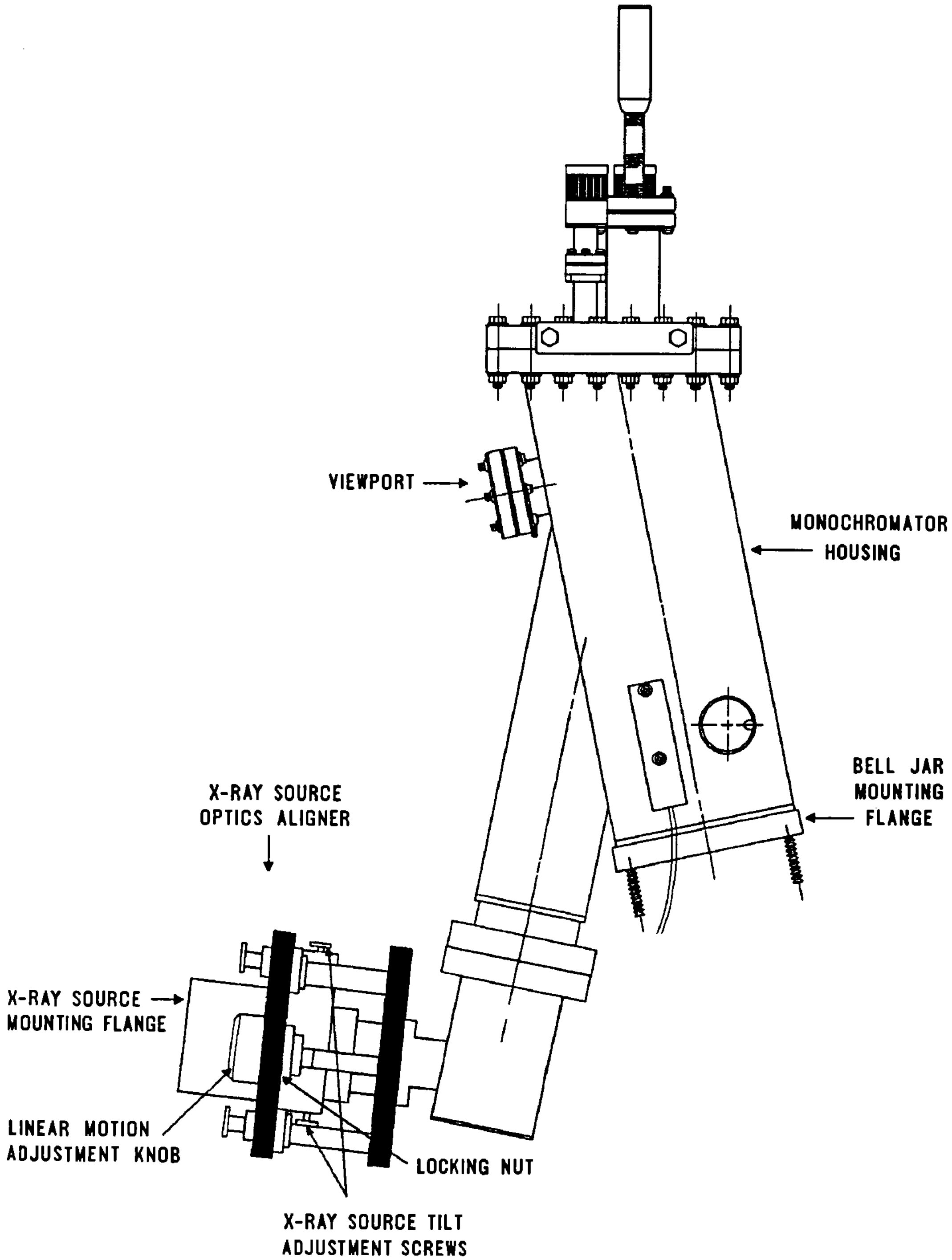


*Figure 3-1.* *Side View of the PHI Model 10-410 Toroidal Monochromator.*

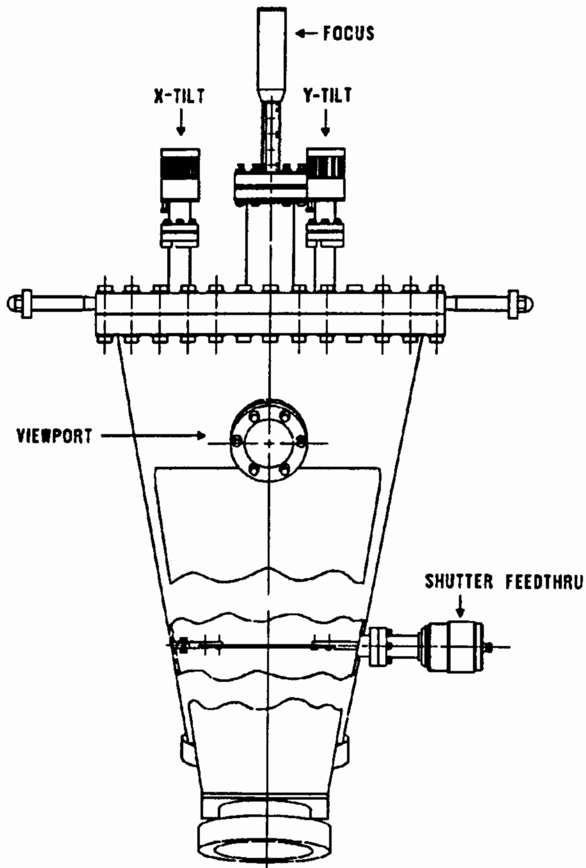


*Figure 3-2. Front View of the PHI Model 10-410 Toroidal Monochromator.*

line (Ag $3d_{5/2}$ peak). Using the same pass energy, work function and lens aperture as in step 3, perform the **[Refresh Acquire]** acquisition process.

17. Align the x-ray beam (i.e. move it in the y-direction) by adjusting the monochromator y-tilt and the x-ray linear motion to maximize the target current. The y-tilt and linear motion must be moved simultaneously for this adjustment. When the x-ray beam is properly aligned, the count rate and target current should be maximized and the peak energy should have the same value as in step 3.

18. Align the monochromator by adjusting the monochromator x-tilt to maximize the count rate. When the monochromator is properly aligned, the target current is typically 1-2 nA.

19. Acquire the Ag $3d_{5/2}$ spectrum and determine the peak's full width at half maximum (FWHM) and count rate.

20. Adjust the monochromator crystal by rotating the focus adjust knob one full revolution. Repeat steps 16 through 19.

21. Repeat step 20, making adjustments in both directions until the focus position giving the smallest FWHM is found.

22. Tilt the silver sample to 65-70°. Adjust the stage y-axis to maximize signal. The count rate should increase by approximately 50% at this tilt angle and resolution should improve.

23. When the monochromator has been aligned, lock all adjustments into position with the appropriate locking screws and nuts. Subsequent analyses require adjusting only sample position and tilt for maximum count rate.

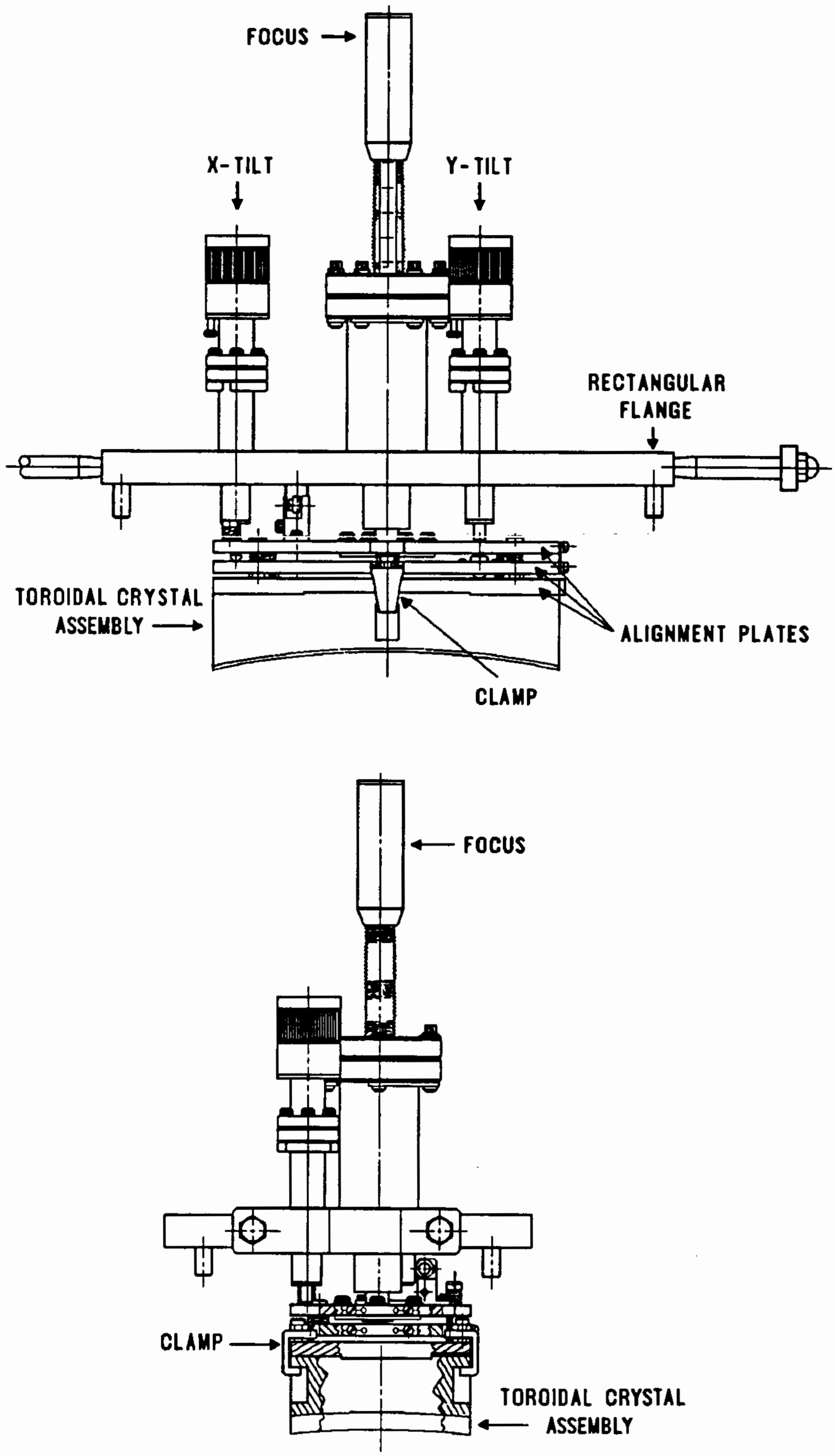


*Figure 3-3. Toroidal Crystal Assembly and Manipulator.*

## 3.3 SELECTION OF MONOCHROMATOR SOURCE

With the system configured in Figure 1-1, either the monochromated x-ray source or the standard x-ray source can be used in sample analysis. To use monochromated x-rays, first set the Model 32-095 X-ray Source Control to EXTERNAL. In the PHI software, enter the Hardware Configuration Menu and set the monochromated x-ray source parameter to YES. Next, enter the X-ray Menu and specify Al for the anode material and 600 watts for the anode power.

<u>*NOTE*</u>

*Only select the monochromated x-ray source parameter in the Hardware Configuration Menu for collecting data while using the monochromator. Choosing this parameter changes the sensitivity factors used in the quantification of data.*

## 3.4 RUNNING SPECIFICATIONS

1 When all adjustments have been optimized as described above, obtain a series of Ag $3d_{5/2}$ spectra over the range 370-366 eV at pass energies 4.45, 8.92, 17.90, 35.75, and 71.55 eV.

2. Draw baselines from the background at 370 to the background at 366 and determine the FWHMs.

3. Plot FWHM vs counts/second on semilog graph paper and draw a smooth curve through the points. The curve should meet or exceed the values shown in Table 3-1.

**Table 3-1. Toroidal Monochromator Specifications**

| Analysis Area | Resolution FWHM(eV) | Sensitivity SCD (cps) | Sensitivity PSD (cps) |
|---|---|---|---|
| 1.1 mm dia. | ≤ 0.50 | 5,000 | 12,000 |
| 1.1 mm dia. | ≤ 0.60 | 7,500 | 20,000 |
| 0.6 mm dia. | ≤ 0.50 | 2,500 | 7,500 |
| 0.6 mm dia. | ≤ 0.60 | 5,000 | 15,000 |
| 0.2 mm dia. | ≤ 0.50 | 250 | 750 |
| 0.2 mm dia. | ≤ 0.60 | 500 | 1,500 |

## 3.5 EXAMPLES OF OPERATION

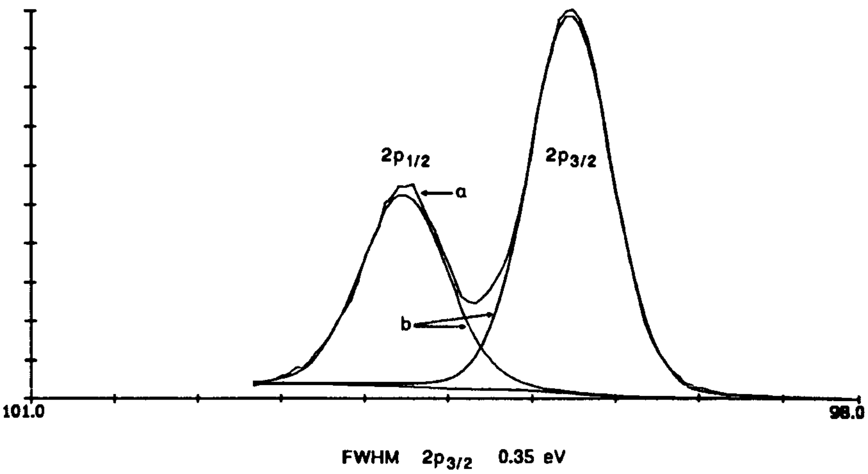


*Figure 3-4. Data acquired on a Si wafer with the monochromator operating at 600 W, using a 1.1 mm dia. aperature and PSD detector. This Si 2p spectrum shows several advantages of the monochromator. Line a shows the raw data. Without the monochromator, the Si 2p doublet would appear as a single peak. Line b shows the raw data curve fit by two peaks.*

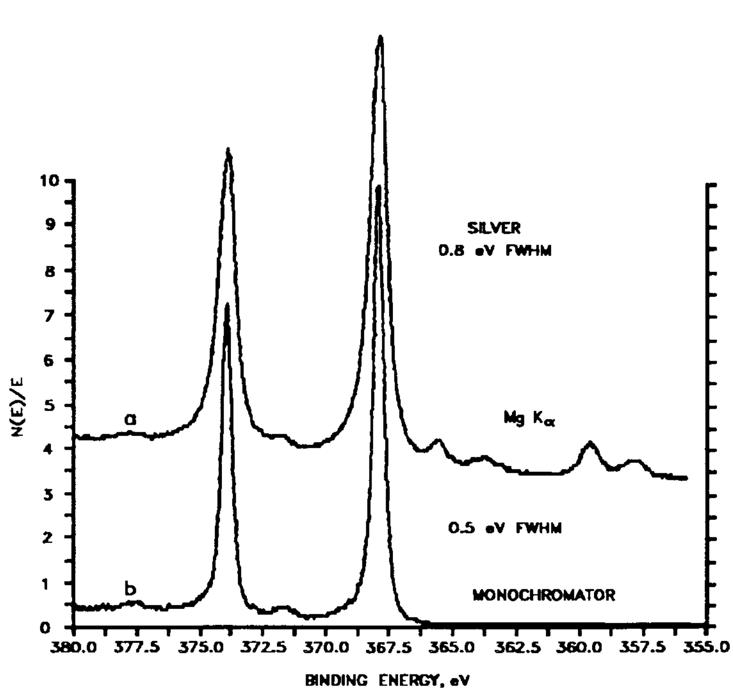


*Figure 3-5. Data acquired from a silver sample using both a standard x-ray source (a) and the monochromator (b). Use of the monochromator results in narrower peaks (0.50 eV line width), the disappearance of satellite peaks, and a decrease in background.*

# SECTION IV
# THEORY OF OPERATION

## 4.1 GENERAL INFORMATION

The toroidal monochromator operates according to Bragg's Law for x-ray diffraction. A single wavelength of $K\alpha$ x-radiation from a conventional aluminum anode is reflected from a (100) quartz crystal surface at a specific angle of reflection (Bragg's law). With the proper geometric configuration of the x-ray source, crystal substrate and analysis target, the reflected beam yields a highly focused, monochromatic source of x-rays.

## 4.2 X-RAY DIFFRACTION

Figure 4-1 illustrates the reflection of x-rays from the crystal and the necessary conditions for x-ray diffraction. In the specific case of Al $K\alpha$ radiation diffracted by a quartz (100) crystal,

$\lambda = 8.34$ Å,
$d = 4.255$ Å,
$\theta = 11.5°$.

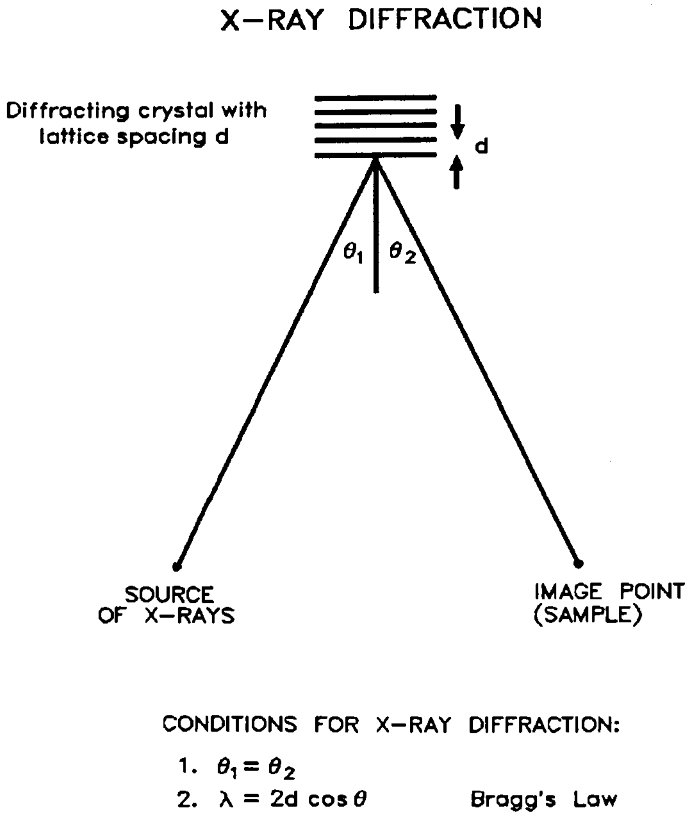


*Figure 4-1. Principles of x-ray diffraction.*

## 4.3 OPTICS CONFIGURATION

Figure 4-2 shows the optics configuration of the toroidal monochromator. The x-ray source, crystal substrate and analysis target are located in a specific geometry around a 500 mm diameter Rowland circle. The radiation is taken from the anode at a grazing angle so that it appears to form a line source. The quartz crystal is bent to a 500 mm radius with its center tangent to the Rowland circle. The reflected x-rays form an angle of 23° with the incident radiation. The input lens of the analyzer is at 90° with respect to the monochromatic x-radiation.

There are several adjustments provided for optical alignment. The linear motion device enables the user to position the anode on the Rowland circle. The crystal substrate assembly can be tilted in two directions and moved in the z (focus) direction with respect to the test chamber in order to place the sample on the Rowland circle.

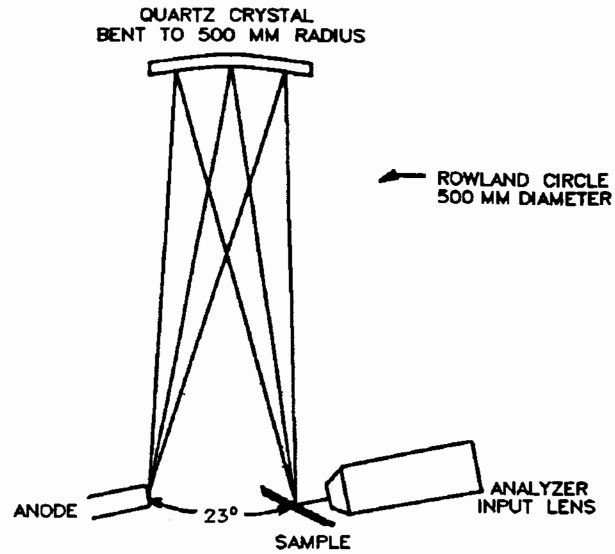


*Figure 4-2. Toroidal Monochromator Optics Configuration.*

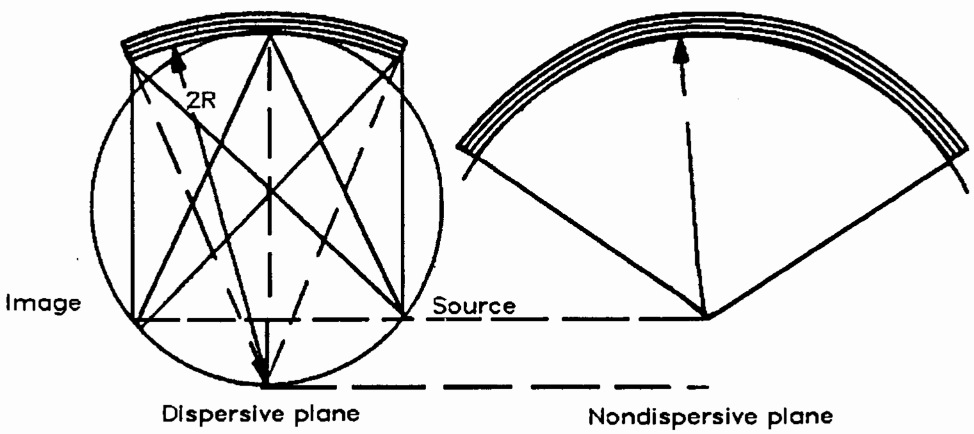


*Figure 4-3. Configuration of the toroidal monochromator, with three tilted Rowland circles. The crystals can be tilted in the plane of the Rowland circle for peak signal intensity.*

# SECTION V
# CALIBRATION AND MAINTENANCE

## 5.1 GENERAL INFORMATION

Refer to the system manual for complete system calibration. There are no calibration procedures other than the alignment described in Section III, and no maintenance procedures for the toroidal monochromator.

If the PHI Model 10-410 Toroidal Monochromator fails to perform specified functions, contact the PHI Customer Service Department.