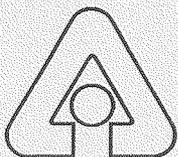
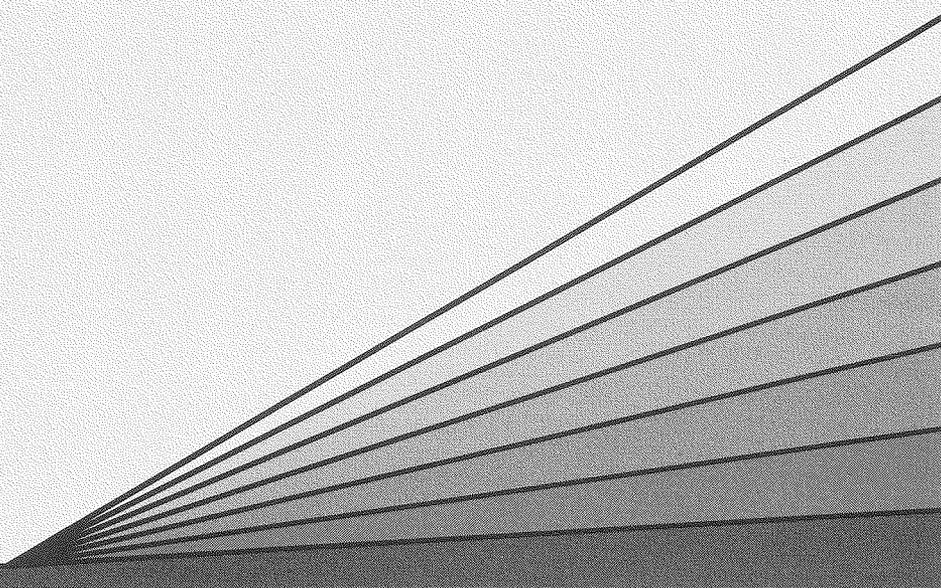


High Heat Load Monochromator Specifications

by Wah Keat Lee and Dennis Mills

February 1993

Advanced Photon Source



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Experimental Facilities Division
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APS High Heat Load Monochromator

Wah Keat Lee and Dennis Mills
Advanced Photon Source
February 1993

Introduction

This document contains the design specifications of the APS high heat load (HHL) monochromator and associated accessories as of February 1993. It should be noted that work is continuing on many parts of the monochromator including the mechanical design, crystal cooling designs, etc. Where appropriate, we have tried to add supporting documentation, references to published papers, and calculations from which we based our decisions.

The underlying philosophy behind performance specifications of this monochromator was to fabricate a device that would be useful to as many APS users as possible, that is, the design should be as generic as possible. In other words, we believe that this design will be capable of operating on both bending magnet and ID beamlines (with the appropriate changes to the cooling and crystals) with both flat and inclined crystal geometries and with a variety of coolants. It was strongly felt that this monochromator should have good energy scanning capabilities over the classical energy range of about 4 to 20 keV with Si (111) crystals. For this reason, a design incorporating one rotation stage to drive both the first and second crystals was considered most promising [1,2]. Separate rotary stages for the first and second crystals can sometimes provide more flexibility in their capacities to carry heavy loads (for heavily cooled first crystals or sagittal benders of second crystals), but their tuning capabilities were considered inferior to the single axis approach.

Much thought was also given to the vacuum requirements of this monochromator. Because this double crystal monochromator (DCM) is designed to operate in the 4 - 20 keV range, the final decision was to choose a high vacuum device (10^{-7} to 10^{-8} torr) but not to require a true UHV device. The impetus for this choice was to facilitate crystal changes, reduce the effort and time for design, and to keep the overall cost of the system at a reasonable level. It was realized that some groups may be interested in a true UHV-compatible monochromator design, and hence APS is currently investigating possible design approaches for a UHV device.

A is shown. Figure 2 shows the P_t and P_p plotted as a function of energy of the first (from 4.2 to 12.6 keV) and third (from 12.6 to 37.8 keV) harmonic energies. This plot reminds one that the highest powers and power densities occur only over selected energy ranges.

Table I - Undulator A Physical Parameters
(at closed gap of 11.5 mm)

Parameters	Value
Period Length [cm]	3.3
Device Length [m]	2.40
Number of periods	72
Max. Magnetic field B_0 [T]	0.72
Critical energy E_c [keV]	23.6
Max. deflection parameter	2.23
Total power [kW]	3.8
Peak power [kW/mrad ²]	134

High Heat Load Monochromator Design

Introduction and Background Information

The purpose of the DCM is to select from the incident polychromatic x-ray beam a desired x-ray wavelength (i.e., to monochromatize the beam). The DCM is typically the first optical component on which the x-ray beam impinges. As such, the DCM will experience extremely high (x-ray) radiation levels, and materials that degrade under radiation exposure must be avoided. To eliminate ozone production (from the interaction of the radiation with the oxygen in the air) and for maintaining general cleanliness of all optical components, the monochromator must be operated in a vacuum at a pressure of 10^{-7} to 10^{-8} Torr. Hence, all components used in the design of the DCM must be compatible with these vacuum levels. When the APS is fully operational, the DCM will be expected to function 24 hrs/day for long periods of time (several months). The overall lifetime of the DCM should be in excess of 5 years.

A schematic of a DCM is shown in Figure 3. The first crystal of the DCM actually provides the monochromatizing action, while the second crystal simply redirects the monochromatized beam parallel to the incoming beam. Typical x-

ray energies of interest are in the 4 -20 keV range corresponding to Bragg angles from 5 to 30 degrees for a Si (111) crystal ($d=3.135\text{\AA}$).

Crystal Geometries

We are currently planning to use “flat” crystal (crystal surface normal in the scattering plane) for wiggler radiation and “inclined” crystals (crystal surface not cut parallel to the atomic planes with the surface normal perpendicular to the scattering plane) for the higher power density undulator radiation. The DCM must therefore be compatible with both these two crystal geometries. Schematics of the flat crystal geometry are shown in Figures 4a and 4b. In the present design, the second crystal will be long enough that a translation will not be required) Schematics of the inclined crystal geometry are shown in Figures 5a and 5b. There are several differences between the “flat” and the “inclined” geometries. In the flat geometry, the Bragg planes are parallel to the surface, whereas they are not in the inclined geometry. At $\theta = 0$ in the flat geometry, the crystal surface is in the horizontal plane, but in the inclined geometry, it is at an angle to the horizontal plane. For the APS DCM, this inclination angle will be between 70 and 85 degrees. In general, the size of the inclined crystal will be considerably longer and narrower than that of the flat crystal, with the long dimension in the direction of propagation of the x-ray beam. In addition, an inclined crystal DCM requires several more degrees of freedom than the flat crystal DCM. These differences will be described in detail in later sections.

Technical Requirements

A summary of critical performance specifications for the DCM are listed below:

- fixed exit operation (<50 micron vertical beam motion across scanning range),
- 35 mm offset between incoming and outgoing beams,
- tunable over the classical energy range (4-20keV) with Si (111) crystals,
- angular resolution of the first crystal: 5 microrads or better,
- high vacuum (HV) compatible (10^{-7} to 10^{-8} Torr range),
- design independent of details of the first crystal geometry and cooling scheme

Typical Mode of Operation

In the standard mode of operation for this DCM, data are collected when the DCM is motionless. That is, data are not taken "on the fly" (while the monochromator is scanning energy, although this may be entirely possible over small energy ranges). However, we wish to be able to scan the DCM over its entire energy range without losing the diffracted exit beam. We plan to drive Y_1 synchronously with the drive for θ , thus obtaining a fixed output beam offset while scanning energy (θ). (see Figure 8). As mentioned previously, the angular width of the crystal's transmission function is only several arc seconds, and this is the degree of change that can be tolerated in the relative angle between the first and second crystals as the unit is scanned in θ (and hence Y_1 is moved). It is a requirement that the exit beam that sets the tolerance on the yaw of Y_1 is not lost. Because data collection periods can extend over several days or weeks, mechanical stability is critical. The relative angle between the two crystals should therefore be able to be maintained to well within one tenth the adjustment range of the fine θ_{adj} over this period of time. The stability required by the user of the DCM is considerably more stringent (arc second stability over the measuring period). A feedback system, to be supplied by the APS, may be necessary to maintain the higher degree of stability feeding back to a piezoelectric transducer (PZT) on the fine θ_{adj} to attain the required stability.

Overall Physical Dimensions

The DCM will reside in the first optics enclosure (FOE) of the synchrotron beamline. The overall layout of the FOE is shown in Figure 6, and the approximate location of the DCM is marked. One of the more critical dimensions is the distance from the beam centerline to the shielding wall. We have only 0.5 meter clearance in this direction and would like to have a clearance of at least 0.2 meter from the wall to any part of the DCM. The incoming beam height is 1.400 meters, relative to the floor of the experiment hall, and with the 35 mm monochromatized beam offset (from the incident white beam), the outgoing beam height will be 1.435 meters. The flange-to-flange length (along the beam direction) of the monochromator vacuum tank should be kept to 1.5 meters or less.

Vacuum System

The vacuum system consists of all vacuum components (chamber, flanges, feedthroughs, rotary seals, etc.) associated with the monochromator, including

ion pumps, power supplies, and controllers but excluding the roughing system. The vacuum system will be designed to support a UHV environment (i.e., internal/full penetration welds should be used and outgassing material avoided), although the vacuum for the monochromator is targeted for 10^{-7} to 10^{-8} Torr during operation. The assembled apparatus shall be leak-checked to 10^{-9} Torr-l/sec or better and maintain a vacuum of better than 1×10^{-8} torr under conditions of no beam.

The vacuum chamber must be fabricated of stainless steel to facilitate UHV compatibility and also to aid in the containment of scattered radiation that can interact with air to produce ozone from the first optical component. The chamber should have a wall thickness of at least 0.25 inch. All flanges less than 13.25 inches should be Conflats of standard sizes. For ease of access, the two side panels of the chamber should be removable, possibly two large 27.125-inch flanges. Those flanges in excess of 13.25 inches (diameter) are to be of a proven wire seal design with readily available preformed gaskets. For ease of access to the tank, we would consider the use of quick release flange couplings. The input flange should be a 6-inch Conflat, while the output flange should be an 8-inch Conflat for connection to the rest of the beamline. The output flange must be configured such that, if the first crystal were to be removed, the white beam would pass through unimpeded. Entrance and exit beam sizes are shown in Figure 7. Infrared (IR) transmitting windows must be appropriately placed on the chamber so that the temperature on the surface of the first crystal in either the flat or inclined geometry can be monitored by an IR camera placed outside the chamber. The IR windows should transmit in the $2 \mu\text{m}$ to $11 \mu\text{m}$ wavelength range. Near the exit port, a feedthrough (linear or rotary, depending on final design) should be available for moving a fluorescent screen in and out of the diffracted beam. A quartz window must be available for viewing the fluorescent screen during diagnostic tests. Two sets of coolant feedthroughs should be available, one for use with water, liquid gallium (at temperatures of about $50\text{-}80^\circ\text{C}$), or liquid nitrogen for cooling the first crystal and the other set for water alone, which may be required to cool the second crystal. These feedthroughs should be located near the axis of rotation so that torque on the crystals from the coolant tubes is minimized as the monochromator changes energy (i.e., θ changes). The chamber must have all the necessary electrical feedthroughs used to control or monitor the in-vacuum devices such as PZTs, motors, etc., provided by the vendor. In addition, flanges for feedthroughs for ten thermocouples and actuator (PZTs, motors, etc.) on the crystal mounts should be provided by the vendor. These feedthroughs can be shared in a common flange with feedthroughs required by vendor-supplied actuators or can be mounted in separate flanges. Strain relief shall be provided for connections to all electrical feedthroughs. If separate flanges are used, they should be blanked

off for testing and delivery. Figure 8 shows the schematic of the UHV chamber. The mechanical, rotary, coolant, and electrical feedthroughs are not shown.

The vacuum pumping system should maintain a high vacuum (10^{-7} - 10^{-8} Torr) environment inside the vacuum chamber with beam present. The APS beamline vacuum systems will use Perkin Elmer ion pumps. For compatibility, the same pumps should be used for the monochromator vacuum chamber. The pump should be sized to maintain the desired vacuum of 1×10^{-8} Torr or better with beam present. If the ion/roughing pump flanges are located at or near the bottom of the vacuum chamber, a baffling system should be included in the design to prevent coolant from entering the pumps in case of an in-vacuum coolant leak. (This might be a lip around the flanges to prevent pools of coolant from running into the pump and a cover so that coolant cannot drop directly into the pumps from above.) There should be a leak valve on the chamber for venting purposes. A gate valve between the chamber and the pumps is to be included to allow the pumps to remain operating while the vacuum chamber is vented to atmosphere. A grounded screen shall be provided between the ion pump and the gate valve. Gate valves are to have metal-sealed bonnets with Viton O-ring sealed gates. The valve should be mounted so that the Viton faces away from the chamber (for protection of the Viton seal from radiation). Gates are to maintain a vacuum of 1×10^{-9} Torr against atmosphere applied in either direction. The vendor shall supply the APS with the complete vacuum pumping system, including the pumps, power supplies, gauges, and controllers (Granville Phillips preferred). All pump controllers and gauge readouts should have computer interface (IEEE 488) capability for remote monitoring purposes.

All materials used, including the translation/rotation stages and mounts described in the Mechanical Design section, that reside in the vacuum chamber must be properly cleaned and must be UHV compatible. Electrochemical polishing of all in-vacuum surfaces is recommended. The vacuum chamber and any mechanical parts inside the vacuum chamber must be bakeable to 100°C . All materials used inside the vacuum chamber must have vapor pressures of less than 10^{-8} Torr at 100°C . Residual gas spectra of the assembled monochromator at room temperature and at 100°C shall be provided by the vendor. The parameters used in acquiring the spectra shall also be stated by the vendor.

Mechanical Design:

Figures 9 and 10 show schematics of the monochromator motions. The monochromator must be able to adapt to both the flat and inclined crystal cases and be flexible enough to take minor changes in the cooled crystal manifold. For ease of visualization, the flat crystal case (i.e., the case where the crystal

surface is cut parallel to the diffracting planes) is considered first. The crystals are positioned such that the surface normal (and hence normal to the atomic planes) of the first crystal is perpendicular to and passes through the θ -rotation axis, while the surface of the second crystal (which is parallel to the first crystal) lies in a plane that contains the θ -rotation axis.

Because the range of the θ rotation is quite large, care must be taken so that the coolant lines flex properly without undue strain during rotation, which could misalign one crystal relative to the other. If the θ -rotation stage is attached to the flange nearest the shielding wall, it would allow the easiest and most convenient access to the crystals via the outer flange (further from the wall). Because, since the distance from the beam centerline to the wall in that direction is only 0.5 meter, this may not be possible. One alternative is to attach the rotation stage to the outer flange and have the flange sit on a rail system. Then, for access, the whole flange (rotation stages, crystals and all) would translate away from the wall (the UHV chamber would stay put).

To compensate for any angular changes that occur between the first and second crystals (from thermal/mechanical instabilities, for example) rotations χ_2 and θ_{adj} are needed. Of these two rotations, much better control and sensitivity are needed for θ_{adj} . Two ranges of adjustment are required for the θ_{adj} , a coarse one for gross alignment of the first and second crystals and a fine one for maintaining parallelism of the atomic planes to an arc second or so. Previous experience has shown that PZT devices work very well in this application for the fine θ_{adj} because they are easy to incorporate into a feedback loop designed to keep the crystals parallel. (The feedback electronics are not to be supplied by the vendor.) Because of the limited range of PZTs (typically, 10 to 50 microns), the coarse adjustment has traditionally been made via a mechanical adjustment or an independent rotational stage onto which the fine adjustment is mounted. If the coarse adjustment is made by mechanical means, this adjustment can be made by assembling a screwdriver-like device on a linear rotary feedthrough to provide *in situ* changes with the monochromator at some particular rotation angle θ . However, recent advances in PZT technology now permit much longer linear extensions to be made and perhaps the fine and coarse adjustments can be incorporated into one. We will entertain either type of arrangement in the proposed design.

Except for the main θ -rotation axis, all other rotation axes should pass through the center of the front face of the crystal that is being rotated. For this reason, the rotation stages should ride on the translation stages instead of the reverse.

The vendor shall supply all the necessary rotation or translation devices including encoders (where appropriate) and stepper motors. In addition, the vendor shall supply all the necessary mounting hardware. If the motion devices (for example, stepper motors) require cooling, the vendor shall supply all the necessary water-cooled mounts, tubes, and the appropriate vacuum feedthroughs. The cooling vacuum feedthrough for the first crystal assembly must be independent of all the other coolant feedthroughs. The APS shall provide the vendor with details of the crystal assembly for mounting purposes.

Although not shown explicitly in any of the drawings, the second crystal may need to be cooled in some fashion.

The vendor shall supply a kinematic mounting plate to accept the first and second crystal mounting plates. The positions of the mounting plates relative to the beam centers are shown in Figure 14. The mounting plate should permit repositioning of the crystals and their mount to within twice the stated accuracy of the motions on which they are connected.

Precision machining is expected on all mechanical components, and the use of shims to achieve fine alignment shall be avoided. All internal mechanical assemblies (except the θ rotation stage and shaft) shall be constructed so that disassembly and reassembly can be easily done through the use of locating pins and or machined shoulders.

Listed below are the specifications for required in-vacuum motions. The roll, pitch, and yaw motions of the translations stages are defined as follows: yaw is a rotation about the x-axis, pitch is a rotation about the y-axis, and tilt or roll is a rotation about the z-axis, where the xyz axes are defined in Figures 9 and 10. It is the tolerance on the yaw that is more critical because alterations in yaw change the Bragg angle.

In-Vacuum Motion Range and Accuracy Specifications:

Motion:	θ rotation (remotely controllable)
Function:	Changes the angle between the incoming beam and the atomic planes for both crystals
Range:	90° (360° preferred)
Resolution:	1 arc second
Load Capacity:	Weight of all attached stages and maximum of 15 kg of crystals and stainless steel

manifold (first and second crystals and mounts)

Motion: θ_{adj} rotation coarse (manually controllable, remote operation optional) and fine (remotely controllable)

Function: Fine tunes θ of the first crystal to compensate for any mechanical/thermal instabilities. (Aligns the Bragg planes to the correct angle) Should have a coarse and a fine adjustment.

Fine Range: 2 arc min

Fine Resolution: 0.1 arc second

Load Capacity: Weight of all attached stages and maximum of 10 kg of crystal and mounts

Coarse Range: $\pm 1.0^\circ$

Coarse Resol.: 1 arc minute

Load Capacity: Weight of all attached stages and maximum of 10 kg of crystal and mounting

Motion: Y_1 translation (remotely controllable)

Function: Moves first crystal perpendicular to the Bragg planes so that the incoming beam hits the center of the crystal

Range: 10 mm

Resolution: 25 microns or better

Load Capacity: Weight of all attached stages and maximum of 10 kg of crystal and stainless steel manifold

Yaw: 1 arc second over range of travel

Roll and pitch: 1 arc minute over range of travel

Motion: X_2 translation (remotely controllable)

Function: Moves the second crystal horizontally in and out of the x-ray beam. (for alignment purposes in the inclined geometry)

Range: 25 mm

Resolution: 0.1 mm

Load Capacity: Weight of all attached stages and maximum of 5 kg of crystal/mount

Motion:	χ_2 rotation (remotely controllable)
Function:	Adjusts the tilt of the second crystal to match that of the first
Range:	$\pm 5^\circ$
Resolution:	1.0 arc minute
Load Capacity:	All attached stages and 3 kG of crystal and mount

Support Stand Motion Range and Accuracy Specifications:

Motion:	X_1 translation
Function:	Moves the first crystal horizontally in and out of the x-ray beam (for alignment purposes in the inclined geometry)
Range:	20 mm
Resolution:	0.1 mm

The above specifications are required for first crystal alignment. In addition, for general alignment of the entire chamber, the support stand must have Y-translation capability (same specifications as X_1) and roll, pitch, and yaw capabilities.

In addition to these motions, some of the required motions will be incorporated into the crystal mounts. To clarify, the following motions will be supplied by us:

Crystal Mount Motion Range and Accuracy Specifications:

Motion:	ρ_1 rotation (manually adjustable)
Function:	Rotates the first crystal about the reciprocal (used in the inclined geometry only)
Range:	$\pm 5^\circ$
Resolution:	0.05°
Load Capacity:	maximum of 10 kg of crystal and stainless steel manifold

Motion:	ρ_2 rotation (remotely controllable)
Function:	Rotates the second crystal about the

	reciprocal lattice vector (Used in inclined geometry only)
Range:	$\pm 5^\circ$
Resolution:	0.05°
Load Capacity:	maximum of 5 kg of crystal and mount

(The X_1 , X_2 , ρ_1 , and ρ_2 degrees of freedom are needed only in the inclined geometry.)

Crystal Assembly Size and Mass

The crystal assembly comprises the crystal itself, coolant manifold, and the input/output cooling tube connections. The crystal assembly will be fully developed by the APS. It is described in this document for the purpose of defining its size and mass. In the flat geometry case, the first crystal will be approximately 100 mm by 100 mm by 25 mm thick, while the second crystal will probably be about 250 mm by 100 mm by 10 mm thick. In the inclined geometry case, the first crystal will be approximately 250 mm by 75 mm by 25 mm thick, while the second crystal will probably be about 250 mm by 75 mm by 10 mm. The actual cooling scheme and manifold are still under research and development. For the inclined geometry case, the angle of inclination may vary between 70 and 85 degrees. In both the crystal geometries, the total weight of the first crystal assembly including the manifold, coolant, and mounting plates should be less than 10 kg, while the weight of the second crystal assembly including the manifold, coolant, and mounting plates will be less than 5 kg. The design of the monochromator must be independent of the details of the first crystal cooling manifold.

Controls/Interfacing/Cabling

The vendor shall supply the APS with all the necessary devices (motors, PZTs, PZT controllers, encoders, microsteppers, power supplies, cables, etc.) for the monochromator motions. Because the DCM will be computer controlled, all devices and drives should be computer compatible. Cables should be long enough to permit control of the monochromator at a remote location 20 m from the monochromator assembly. The current plan at the APS is to use a UNIX-based workstation to communicate with the DCM motion drivers via a VME crate. Cables inside the vacuum chamber must be appropriately shielded to withstand the radiation (e.g., Teflon insulation is unacceptable). The vendor shall not supply the APS with the stepping motor controllers, computer I/O boards, and equipment that are computer/operating system specific.

Diagnostics

Diagnostics for the monochromator are straight forward and will be composed of a remotely controllable fluorescent screen in the vacuum chamber that can be positioned in the path of the diffracted beam (avoiding the direct beam). The screen will be viewed through a quartz viewing port in the vacuum chamber by a television camera and monitor system. (Quartz is chosen because it does not darken under the influence of x-ray radiation as rapidly as plate glass.) Therefore, the requirement is that the vacuum chamber has a remotely controllable linear or rotary feedthrough near the exit port for moving the fluorescent screen in and out of the beam.

Hardware Deliverables

The contractor shall deliver to the APS the complete vacuum/mechanical system of the DCM. The following is a list of the hardware deliverables.

1. A vacuum chamber with (a) all necessary IR windows, (b) one quartz window, (c) first crystal coolant input/output feedthroughs (compatible with water, liquid gallium, or liquid nitrogen), (d) second crystal water input and output feedthroughs, (e) blank-off flanges for any ports or feedthroughs not utilized, (f) a linear or rotary feedthrough for the fluorescent screen, (g) all necessary electrical feedthroughs, (h) all coolant transport tubes inside the vacuum chamber, and (i) any rail/translation system that is necessary for removing the flanges during crystal changes.
2. A vacuum pumping system including (a) ion pump (b) all pumping-system-related power supplies, (c) all pumping system controllers, (d) all vacuum gauges and monitoring devices, and (e) all connections to the vacuum chamber, including adapter flanges (if necessary), and all cables.
3. All the mechanical motion devices described in the Mechanical Description section above including (a) all translation stages, (b) all rotation stages, (c) all PZT or inchworm devices, (d) all gear reducers, (e) all encoders, (f) all the necessary motors, (g) all the power supplies needed, (h) all the adapter and/or mounting pieces to achieve the required motions, and (i) all cables inside and outside of the vacuum chamber necessary to power the devices.
4. The vendor shall not supply any computer-or operating-system-specific piece of equipment, for example, stepper motor

controllers, computer I/O boards, etc., the silicon crystals, or the heat removal apparatus. The vendor shall also not supply the vacuum roughing system or the television camera and monitor system.

Liquid Gallium Pump

Liquid Gallium Cooling of X-ray Optics

The increased cooling capability of liquid gallium over water (for a given flow rate) has been well known for some time [4-6]. The pertinent physical properties of water and liquid gallium are given below in Table III.

Table III
Physical Properties of Water (27° C) and Liquid Gallium (30°C)

<u>Property</u>	<u>Gallium</u>	<u>Water</u>
Density (gm/cm ³)	6.09	0.998
Melting point (°C)	29.8	0.0
Boiling point (°C)	2403	100
Thermal conduc. (W/cm-°C)	0.28	0.00613
Specific heat (J/g-°C)	0.373	4.179
Vapor pressure (Torr)	10 ⁻¹⁰	31.8

Other advantages of liquid gallium, in addition to its superior thermal properties, are its extremely low vapor pressure, high boiling temperature, high surface tension, and high thermal and electrical conductivity as compared to water. Our experience has been that seals that leak slightly when pressurized with water to not leak when pressurized with gallium. We attribute this to gallium's high surface tension. The low vapor pressure allows one to have small leaks in the vacuum system without deterioration of the chamber pressure. A high boiling temperature and good thermal conductivity mean that, should flow be accidentally be reduced in the crystal, some heat will still be conducted and local boiling of the coolant will not occur (which could result in a critical heat flux and burn out situation). And finally, the electrical conductivity means that the gallium can be pumped electromechanically, that is the pump need not have any moving parts [7], and hence vibrations from the pump itself should be virtually eliminated. Flow-induced vibrations arising from sharp turns in tubing and manifold, convolutions in bellows, or abrupt changes in the diameter of coolant carriers can be a serious problem. In collaboration with vibration

experts from the Materials and Components Technology (MCT) Division at ANL, we are currently investigating approaches that will minimize this phenomena. Gallium is a rather inert material and elaborate safety precautions are not necessary. (Safety precautions for gallium can be found in Appendix A of this document.) Liquid gallium does react adversely with some materials, in particular aluminum. Fortunately, we have found that there seem to be no deleterious effects on silicon, stainless steel, and Teflon tubing from exposure to liquid gallium at modest temperatures ($< 50^{\circ}\text{C}$).

The specifications for the commercially procured liquid gallium pump are given below.

Material Specifications

- The permanent magnets will be constructed of neodymium-iron-boron magnetic material. Specifications are listed in Appendix B.
- The magnetic return path will be made of high grade magnet iron. Specifications are listed in Appendix C.
- All parts of the system that come in contact with gallium will be made of nonmagnetic 304 stainless steel or 321 hydraulic stainless steel tubing. The only exception that is allowed is the use of Teflon as part of a flow gauge or as part of a visual level indicator. The flow gauge may have to be made of stainless steel depending on its location. All nonstainless steel parts must be approved by ANL before construction.
- Aluminum will be avoided in the construction of the system for all parts associated with the pump.

Performance Specifications

- The pump must achieve a volume flow rate of 4 gallons per minute (gpm) while operating with a head pressure of 75 pounds per square inch (psi).
- The temperature of the gallium delivered by the system should be automatically controllable between the temperatures of 40° and 70°C and regulated to $\pm 3^{\circ}\text{C}$ with any steady state heat load from 0 to 5 kwatts. The recovery time from large transients (power on/off) should be no more than 5 minutes.
- The system should be able to withstand internal pressures of 200 psi and be able to be operated under vacuum (< 1 torr).

- The gallium-to-water heat exchanger should be able to remove 5 kW of power (with a 10 KW option) with water cooling of <5 gpm and 80 psi.
- The DC current supply should supply 0 - 3000 A at 0 - 3 volts and should have an AC ripple on the output of less than 0.5 percent. The power supply leads must be at least 10 feet in length.
- Local readout for (1) pressure and temperature sensors (2) controls for adjusting the temperature of the gallium and (3) controls for adjusting the flow of the gallium.
- The remote status panel (19" rack mountable) should be operable at a distance of 50 feet from the pump. It should contain (1) readouts for the pressure and temperature sensors, (2) controls for adjusting the temperature of the gallium, and (3) controls for adjusting the flow rate of the gallium. All readouts should be via either RS-232 or GPIB. Arrangement of panel display of parameters will be reviewed before construction.

Power specifications

- 120 / 220 VAC

General features

- All connections should be easy to assemble and not produce torque on tubing and/or crystal. Recommend using type Cajon/Swagelok, VCR, Face Seal Fitting with stainless steel insert connectors. ANL permission needed for the use of other connectors.
- Gallium level indicator on the pump.
- Spill containment pan under pump.
- Heaters on pump to keep gallium from freezing during non-operational periods.
- Bypass valves to allow pump to run while crystals are being exchanged.
- Power supply (current and voltage) and gallium pump (flow, pressure, level, temperature) should be alarmed/interlocked so that an out-of-range value for these levels can be used to shut down the beam.

A schematic drawing of the DC current liquid gallium pump is shown in Figure 12. The APS prototype pump performance (flow rate and pressure as a function of current) is shown in Figures 13 and 14.

High Heat Load Crystals

Inclined Crystal Approach

The solution for dealing with the high heat load problem for the APS Undulator A will include the use of inclined crystals. Briefly, the inclined crystal geometry spreads the beam footprint on the surface of the crystal, while maintaining a symmetric reflection, that is; the asymmetry parameter, $b = k_{in}.n/k_{out}.n = -1$. A conventional flat symmetric crystal spreads the beam by a factor of $1/\sin\theta$, while the inclined crystal spreads the beam by a factor of $1/\cos\beta\sin\theta$, where θ is the Bragg angle and β is the inclination angle. Details of the inclined crystal geometry can be found in references [8-13]. The small angular divergence of Undulator A together with its high power density makes the inclined crystal an ideal candidate for dealing with heat load from this ID. Due to the sizes of the crystals involved, slits will be used to only allow the central cone of the undulator radiation through to the monochromator. Appendix D provides further information about the inclined crystal geometry and some useful relationships for determining beam sizes and shapes.

Modeling and Experimental Results

The APS has completed several experimental and computational studies on the performance of the inclined crystal geometry. Independent simulations using the 8x8 matrix approach and 4th order dispersion surface calculations have shown that the inclined crystal diffraction properties are essentially like those of flat symmetric crystals. Figure 15 compares the reflection curves of an 85° inclined crystal with a flat crystal at 5 keV and 13.84 keV. The minor differences will be mentioned later.

Finite element analysis of the APS Undulator A thermal loading on back-cooled inclined silicon monochromators have also been performed. Table IV summarizes some of the important results. At closed gap and 4.2 keV (worst case power loading), with an inclination angle of 85°, the thermally induced slope error is only 56 microradians. Even better performance is expected with the actual crystals because of more

Case	Slit	β	θ	Harmonic (keV)	Length (cm)	Width (cm)	Thickness (cm)	Coolant	$(\Delta T)_{max}$ $^{\circ}C$	$(U_{y0})_{max}$ (μm)	$(slope)_{max}$ (μr)
IC-062	no	85°	8.99°	E3=12.6 (K=2.2)	95	15	1.0	Ga	39	5.5	26
IC-063	no	85°	8.99°	E3=12.6 (K=2.2)	95	15	0.5	Ga	33	5.5	24
IC-064	yes	80°	8.99°	E3=12.6 (K=2.2)	15	3	1.0	Ga	58	1.9	68
IC-065	yes	80°	5.7°	E3=19.9 (K=1.5)	23	3	1.0	Ga	23	1.9	40
IC-066	yes	85°	13.96°	E1=8.2 (K=1.2)	18	5	1.0	Ga	21	3.5	15
IC-067	yes	85°	27.96°	E1=4.2 (K=2.2)	9	5	1.0	Ga	92	0.6	56
IC-068	yes	85°	27.96°	E1=4.2 (K=2.2)	29	15	1.0	Ga	70	1.0	50
IC-069	no	85°	27.96°	E1=4.2 (K=2.2)	29	15	1.0	Ga	142	3.2	120

Table IV: Summary of FEA results. Explanation of data: A "yes" in the slit column is for cases where a 3.6mm x 1.8mm slit is used to only let the central cone of the undulator through. β is the inclination angle. Length and width are the size of the crystal used. For the case with no slits, the crystal length and width was selected so that the entire undulator beam was intercepted. Thickness is the thickness of the crystal used. ΔT is the maximum surface temperature of the crystal. U_{y0} is the displacement on the surface of the crystal due to thermal expansion. The main quantity of interest is the maximum slope error on the surface of the crystal, the last column.

efficient heat exchange and a smaller distance between the diffraction surface and the coolant.

Experimental studies have confirmed that inclined crystals do behave essentially like flat crystals. In addition, inclined crystals with liquid gallium cooling have successfully handled the heat loads of the ANL/CHESS undulator (379 W total power, 48 W/mm² peak power density, reference 2) and the X-25 focused wiggler at NSLS [38 W total power, 118 W/mm² peak power density,(3)].

Crystal Design

From the simulations and experimental results, we have ascertained that the maximum surface power density that our current cooling schemes (i.e., slotted crystals with liquid gallium coolant at 1 - 2 gpm) can handle without substantial thermal distortion on the crystal is about 4 or 5 W/mm². (although we hope to improve on this with pin post type of heat exchanger and improved bonding techniques with slotted crystals, which will permit greater flow rates for the liquid gallium see the Fabrication section.) Our design of inclined crystals for use with the APS Undulator A is therefore aimed at achieving surface power densities of less than 5 W/mm². Due to the lengths of the crystals involved, only the central cone of the undulator radiation will be accepted. Slits 3.6 mm wide and 1.8 mm high will be used upstream of the DCM. At 30m from the source, this is the approximate size of the central radiation cone up to its 5 σ value. In addition, for ease of alignment, the inclination angle should be kept as small as possible. Also, we would like to have one crystal that is capable of handling the heat load from 8 - 20 keV. With these thoughts in mind, our plan is to use two sets of crystals to cover the 4 - 20 keV range for the Undulator A radiation. Either the silicon (111) or silicon (220) reflections can be used. Table V shows some of the parameters of the crystal for the case of Si(111), which we plan to use. A set of 78° inclined crystals will cover the energy range of 8 - 20 keV, while a set of 85° inclined crystals will cover the 4 - 9 keV range. Figures 16a and 16b show schematically the crystal and the beam footprints of a silicon (111) crystal, with 78° inclination, that will operate in the 8 - 20 keV range. The size of the first crystal will be about 8 inches long by 2 inches wide, while the second crystal will be about 10 inches long by 2 inches wide. The additional length is required on the second crystal for the motion of the beam along the crystal during an energy scan. The crystals have unusual shapes in order to avoid blocking the incoming or outgoing beams.

Footprint dimensions of inclined geometry
w = 3.600000mm, h = 1.800000mm, Cross to 3rd harmonic at 13.000000 keV
beta = 78.000000, Si(111) reflection, beamoffset = 35.000000mm

energy	theta	diagonal	width	powerdensity	beamtrans
3.000000	41.234436	28.039058	1.822414	25.947597	26.549684
4.000000	29.627304	37.640923	1.830137	16.000107	35.399579
5.000000	23.296222	47.177384	1.833745	10.810045	44.249473
6.000000	19.242725	56.689655	1.835714	7.713812	53.099368
7.000000	16.408827	66.189979	1.836905	5.694563	61.949262
8.000000	14.310588	75.683439	1.837678	4.288185	70.799157
9.000000	12.692227	85.172558	1.838210	3.254133	79.649052
10.000000	11.404976	94.658747	1.838590	2.453818	88.498946
11.000000	10.356123	104.342858	1.838871	1.798137	97.348841
12.000000	9.484768	113.625439	1.839085	1.221148	106.198736
13.000000	8.749200	123.106859	1.839252	4.643352	115.048630
14.000000	8.119873	132.587378	1.839384	4.076152	123.898525
15.000000	7.575256	142.067182	1.839491	3.603348	132.748420
16.000000	7.099282	151.546409	1.839578	3.204413	141.598314
17.000000	6.679714	161.025164	1.839651	2.864190	150.448209
18.000000	6.307068	170.503527	1.839712	2.571271	159.298103
19.000000	5.973875	179.991561	1.839763	2.316919	168.147998
20.000000	5.674175	189.459317	1.839807	2.094343	176.997893

Footprint dimensions of inclined geometry
w = 3.600000mm, h = 1.800000mm, Cross to 3rd harmonic at 13.000000 keV
beta = 85.000000, Si(111) reflection, beamoffset = 35.000000mm

energy	theta	diagonal	width	powerdensity	beamtrans
3.000000	41.234436	64.606066	1.803879	10.877128	26.549684
4.000000	29.627304	86.494674	1.805188	6.707180	35.399579
5.000000	23.296222	108.300025	1.805795	4.531527	44.249473
6.000000	19.242726	130.072673	1.806125	3.233599	53.099368
7.000000	16.408827	151.828610	1.806324	2.387138	61.949262
8.000000	14.310588	173.574736	1.806453	1.797590	70.799157
9.000000	12.692227	195.314572	1.806542	1.364120	79.649052
10.000000	11.404976	217.050117	1.806605	1.028631	88.498946
11.000000	10.356123	238.782598	1.806652	0.753772	97.348841
12.000000	9.484768	260.512812	1.806688	0.511900	106.198736
13.000000	8.749200	282.241300	1.806716	1.946474	115.048630
14.000000	8.119873	303.968441	1.806738	1.708706	123.898525
15.000000	7.575256	325.694512	1.806756	1.510509	132.748420
16.000000	7.099282	347.419718	1.806770	1.343277	141.598314
17.000000	6.679714	369.144214	1.806782	1.200657	150.448209
18.000000	6.307068	390.868121	1.806792	1.077866	159.298103
19.000000	5.973875	412.591532	1.806801	0.971243	168.147998
20.000000	5.674175	434.314524	1.806808	0.877940	176.997893

Table V: Some parameters for a set of Si(111) crystals. Beta is the inclination angle. The beams have been slitted down to 3.6mm x 1.8mm at 30m from the undulator A source to allow only the central cone of the radiation through. Theta is the Bragg angle. Diagonal and width refers to the size of the beam footprint on the surface of the crystal. Power density is the power density on the surface of the crystal. Beamtrans is the distance between the centers of the footprints for different energies for the second crystal. For example, for the case of the 78 degree inclined crystal, the distance on the surface of the second crystal between the 8 keV and 20 keV footprints is about 177 - 71 = 106 mm. This is seen in Figure 18b.

Fabrication

The current plan at the APS is to have the high heat load inclined crystals fabricated by Rockwell Power Systems incorporating their pin post type of heat exchanger [14]. The whole crystal assembly will include the pin post heat exchangers, coolant distribution manifold, and the inlet-outlet manifold. (see Fig.17). Both the silicon/silicon and the silicon/metal bonding will be done with frit glass. RPS has substantial experience in silicon bonding and has provided John Arthur of SSRL [14] with a similar device, although in that device, O-ring seals were used. Due to our concern about radiation damage, our plan is not to have any O-rings in the crystal assembly. Instead, the silicon will be bonded directly to the metal manifolds. Due to the compatibility of the thermal expansion properties, the current plan by RPS is to use a Fe-Ni alloy instead of stainless steel for the metal part of the assembly. In-house tests have shown that liquid gallium does not appear to attack the alloy. Details of the actual metal manifold and mounting scheme are still under investigation. For compatibility with our liquid gallium pump, the crystal should withstand 100 psi pressure and have about a 20 psi pressure drop at a 5 gpm flow rate.

Some Subtle Aspects of the Inclined Crystal Geometry

Although the diffraction properties of the inclined crystal geometry are essentially the same as those of the flat crystal geometry, there are indeed some minor differences. They are the following:

- (1) Sensitivity to rotations about the reciprocal lattice vector H . Figure 18 shows the dependence of the asymmetry parameter b , on ρ , the rotation about H . Thus, it may be useful to incorporate that degree of freedom into the monochromator or the crystal mount.
- (2) A slight change in beam shape through a double crystal monochromator. The amount of change in the beam shape depends on the location of the crystal relative to the source and the beam divergence. For the APS Undulator A, with the monochromator at 30 m from the source, the change in shape is on the order of 0.1 mm.
- (3) $\theta_{in} \neq \theta_{out}$. There are very slight differences between θ_{in} and θ_{out} of an inclined reflection. Figure 19 shows the difference in the case of an 85° inclined crystal at 13.84 keV.

- (4) The diffracted beam from one reflection no longer lies in the plane spanned by H and the incoming beam. Figure 20 shows the amount of out-of-plane angular difference in the case of an 85° inclined crystal at 13.84 keV. The result is that a doubly diffracted beam from a double crystal monochromator will be translated horizontally by a small amount. For the APS Undulator A beam, with the monochromator at 30 m from the source and a 35 mm vertical beam offset, the amount of horizontal displacement is about 5 microns.
- (5) For an 85° inclined DCM on the APS undulator A beam diffracting in the vertical plane, the beam from the monochromator will be displaced vertically by about 1 mm. This displacement, for practical purposes, is dependant only on the inclination angle. The motion of the beam for an 85° inclined DCM during an 8 - 20 keV energy scan moves less than a micron. In addition, there is a very small horizontal displacement on the order of 0.1 microns.

Recently, Robert Blasdell of the APS has incorporated the inclined crystal geometry into the ray-tracing program SHADOW. We shall be pursuing the sensitivity of the inclined crystal geometry to many of the above mentioned parameters (ρ , mismatched inclination angles between the first and second crystals, relative misalignments between the first and second crystals, etc.) over the next several months.

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Fig. 1: Energy of the first harmonics of APS Undulator A as a function of the magnetic gap with the Storage Ring operating at 7 GeV.

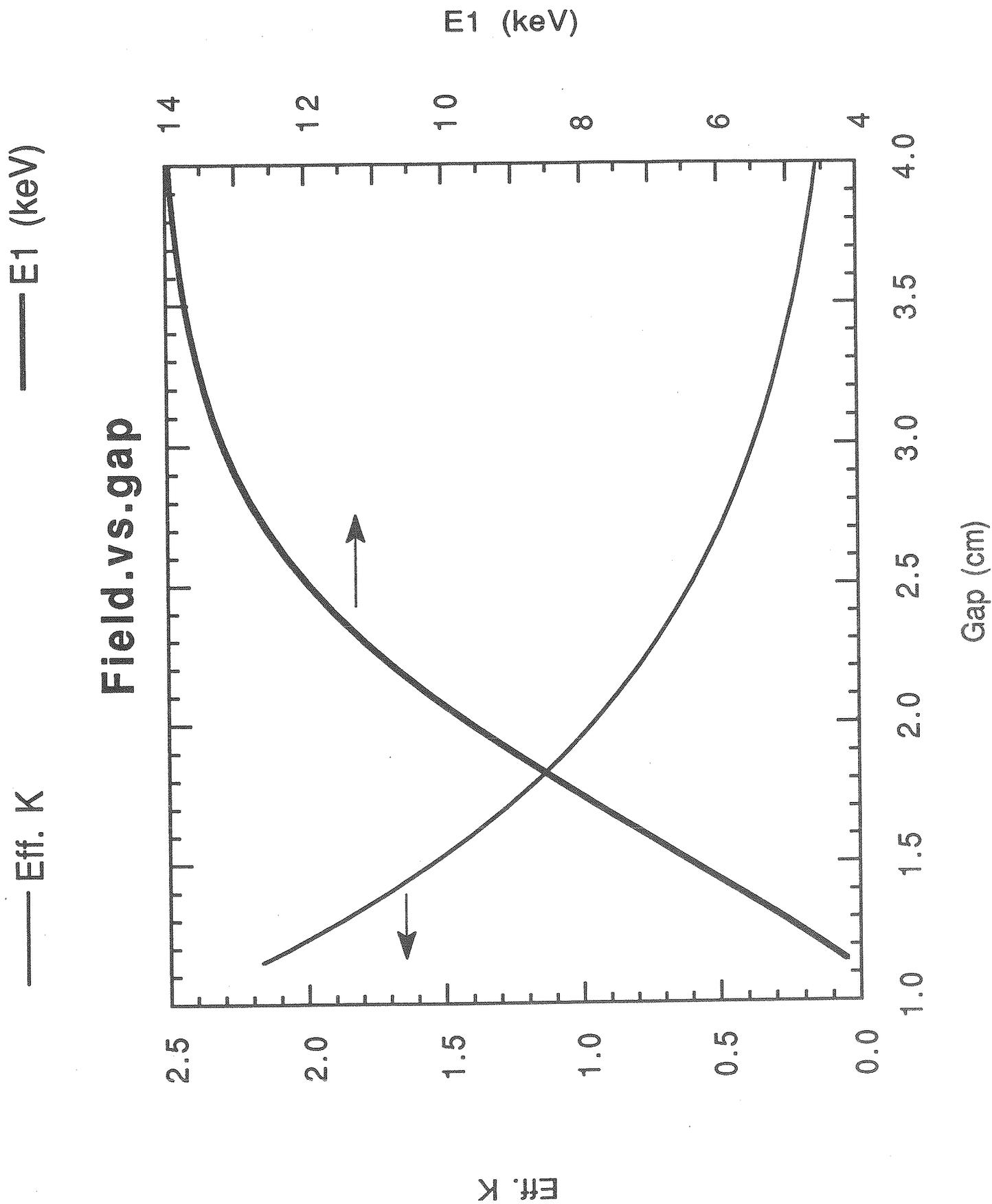
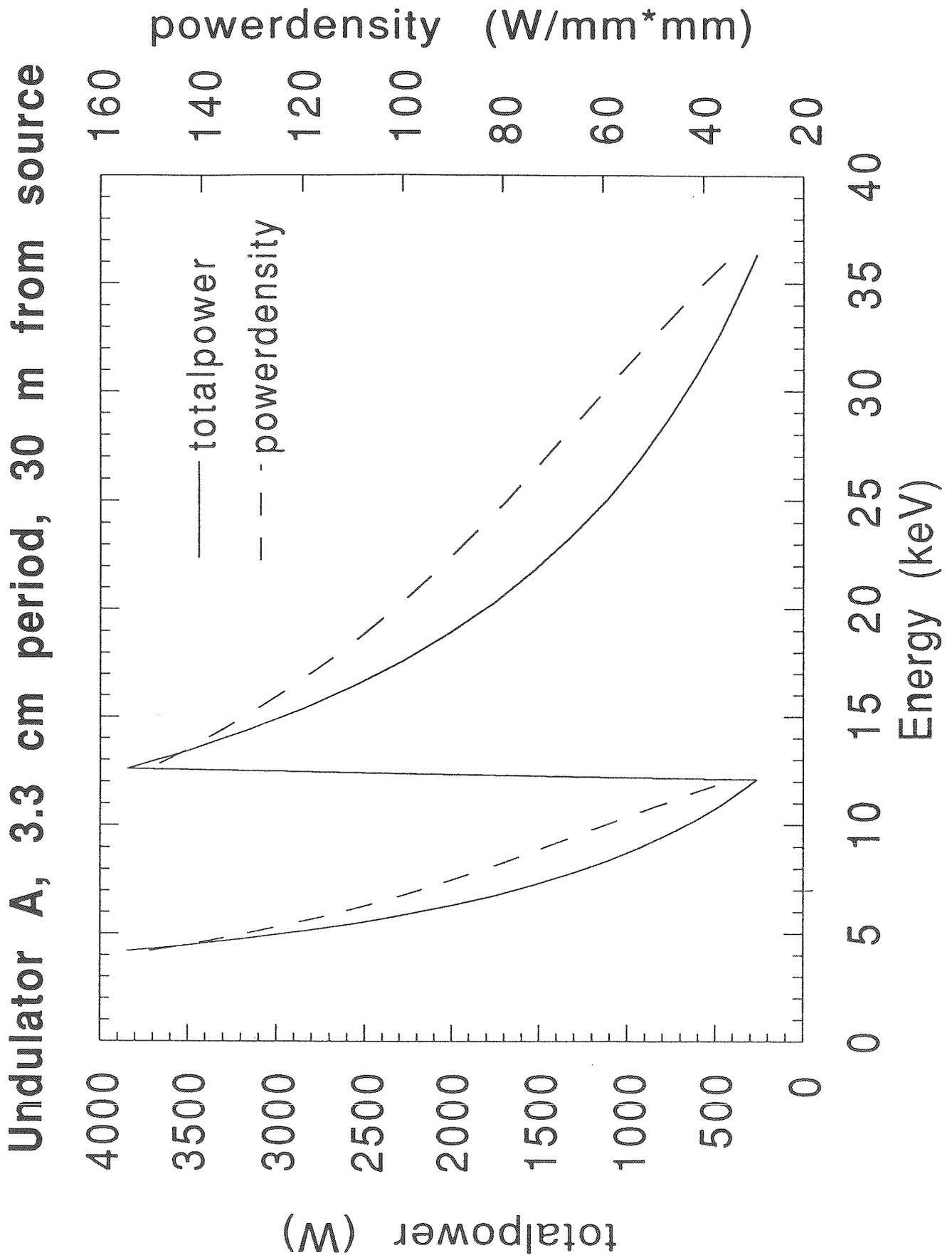


Fig. 2: Total power and peak power density (30 meters from the source) as a function of the first and third harmonic energy of APS Undulator A. Calculations made for 100 ma and 7 GeV operation.



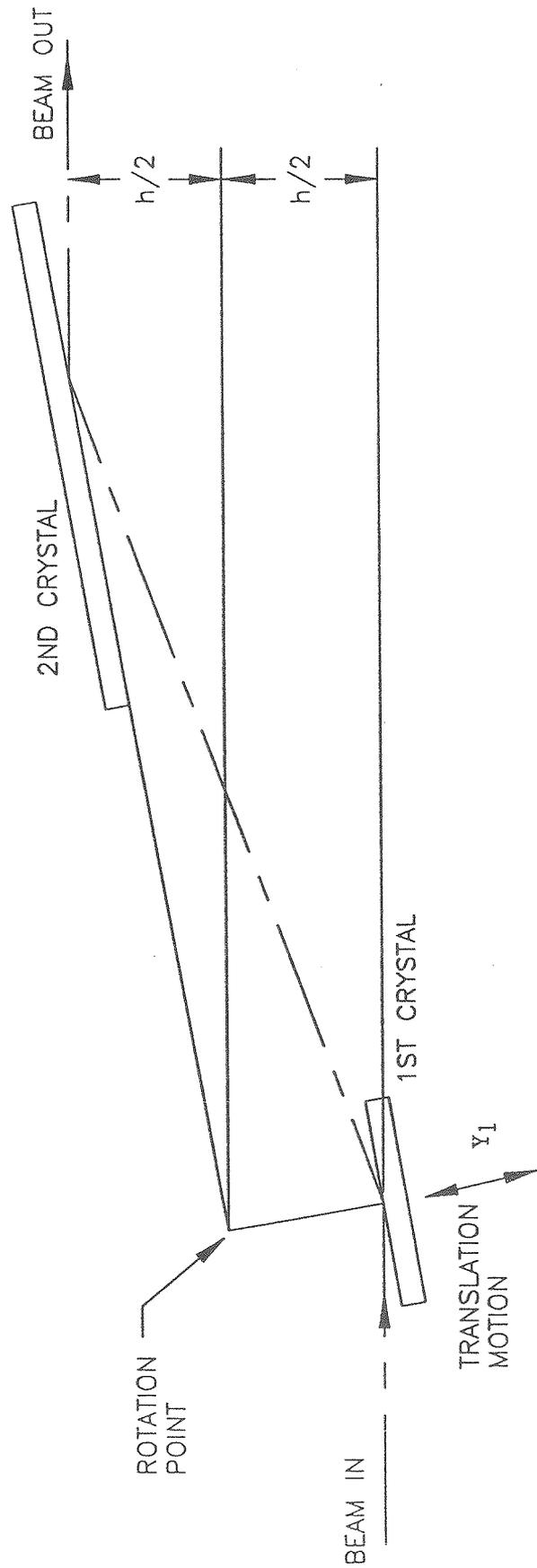


Fig. 3: Schematic of double crystal monochromator.

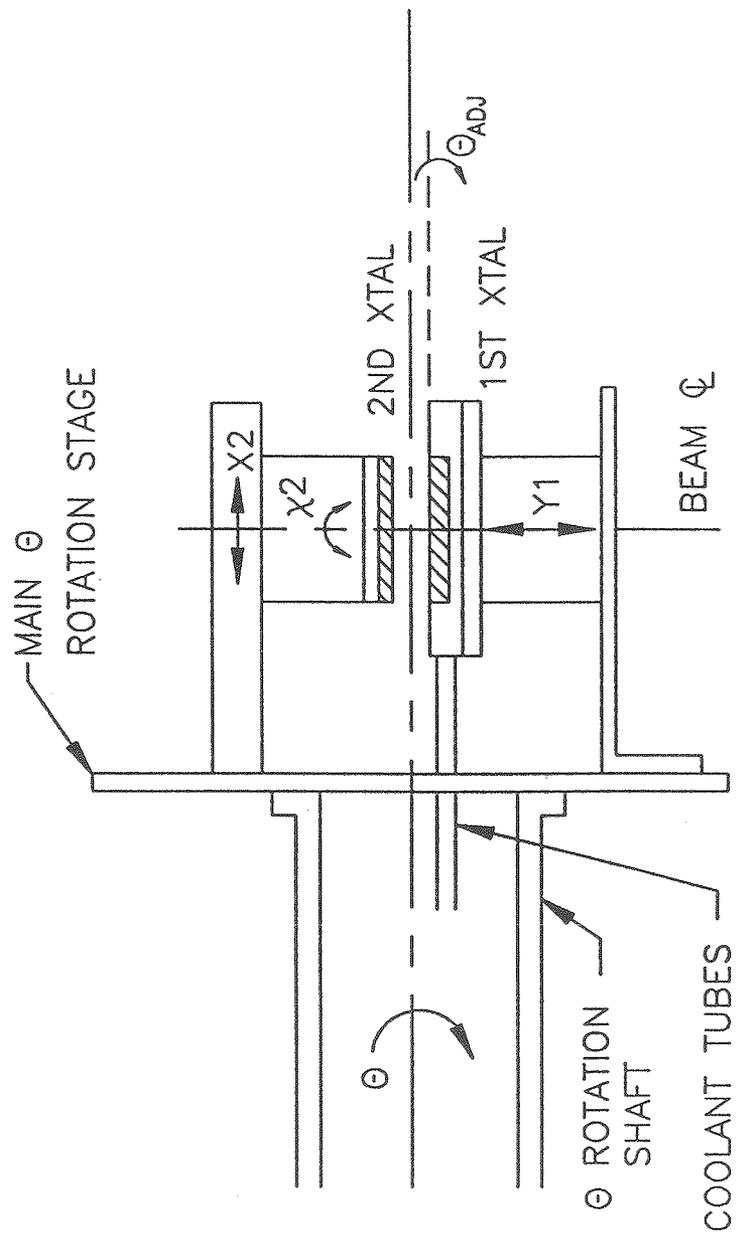


Fig. 4(a): Schematic of flat crystal geometry, upstream view.

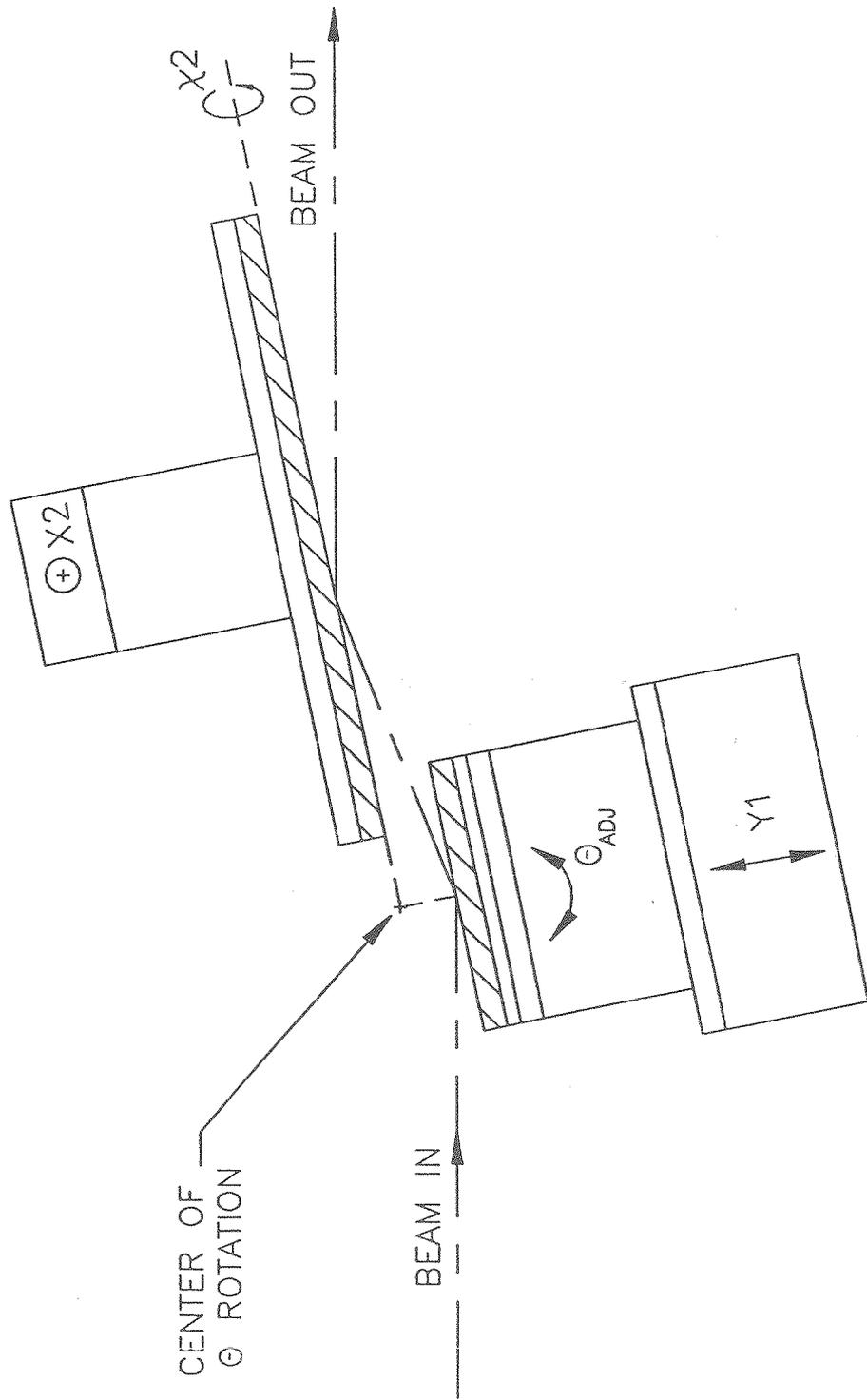


Fig. 4(b): Schematic of flat crystal geometry, side view.

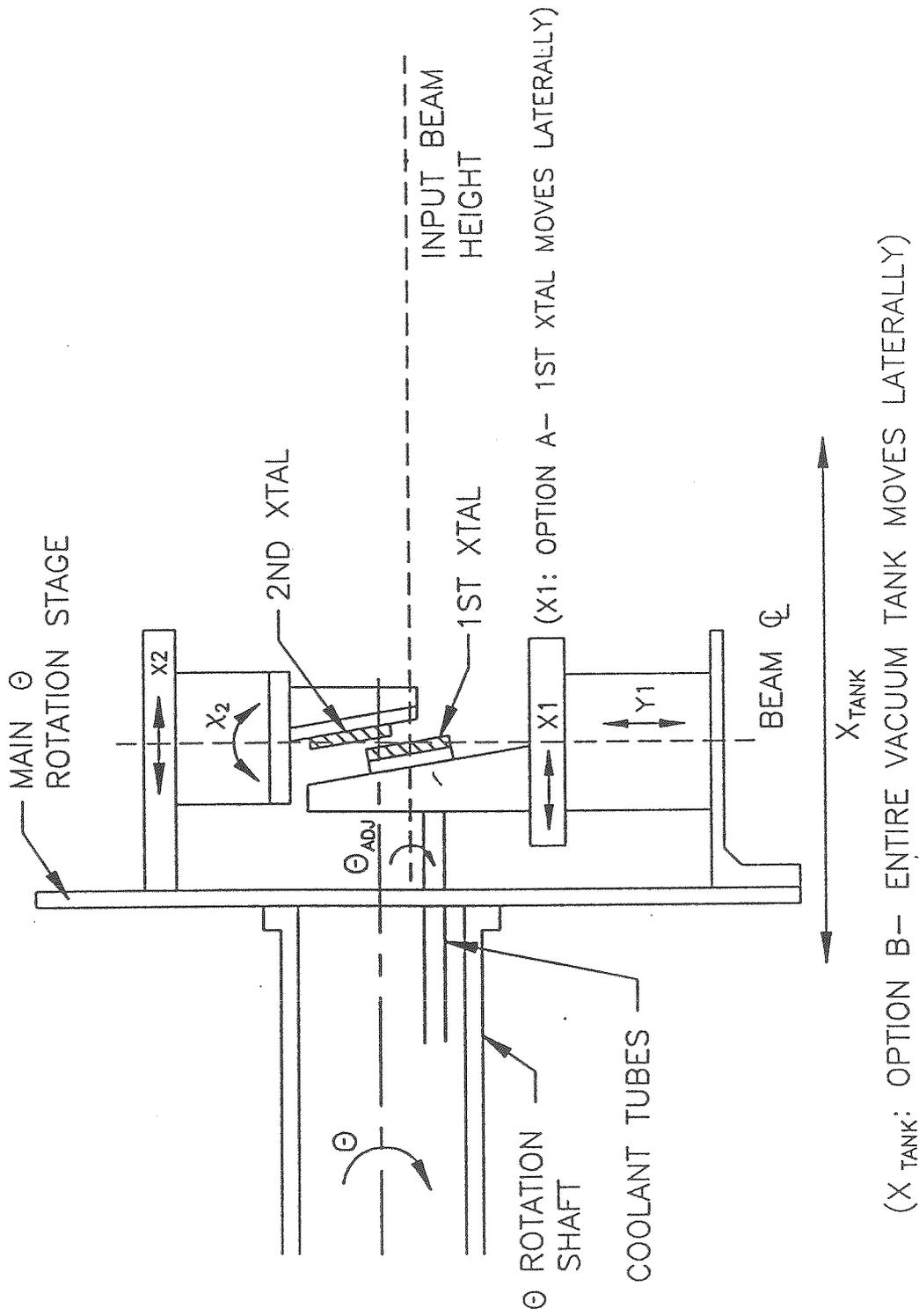
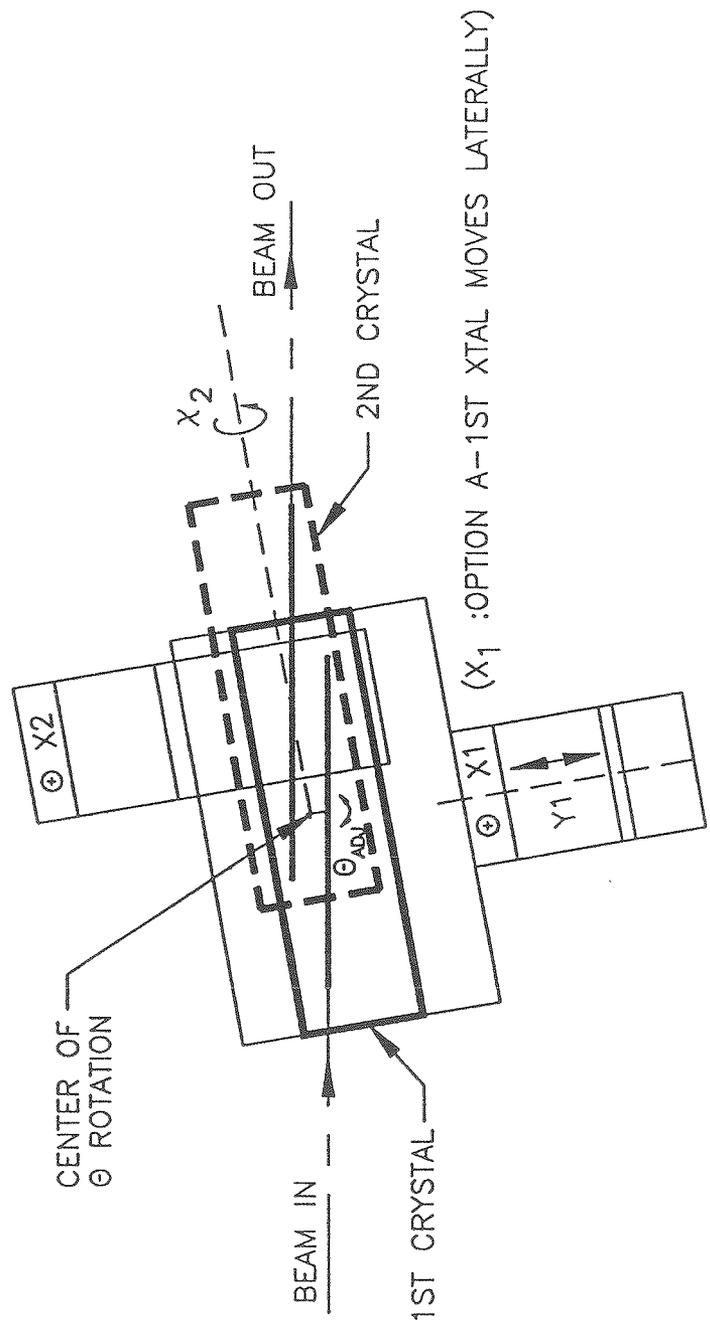


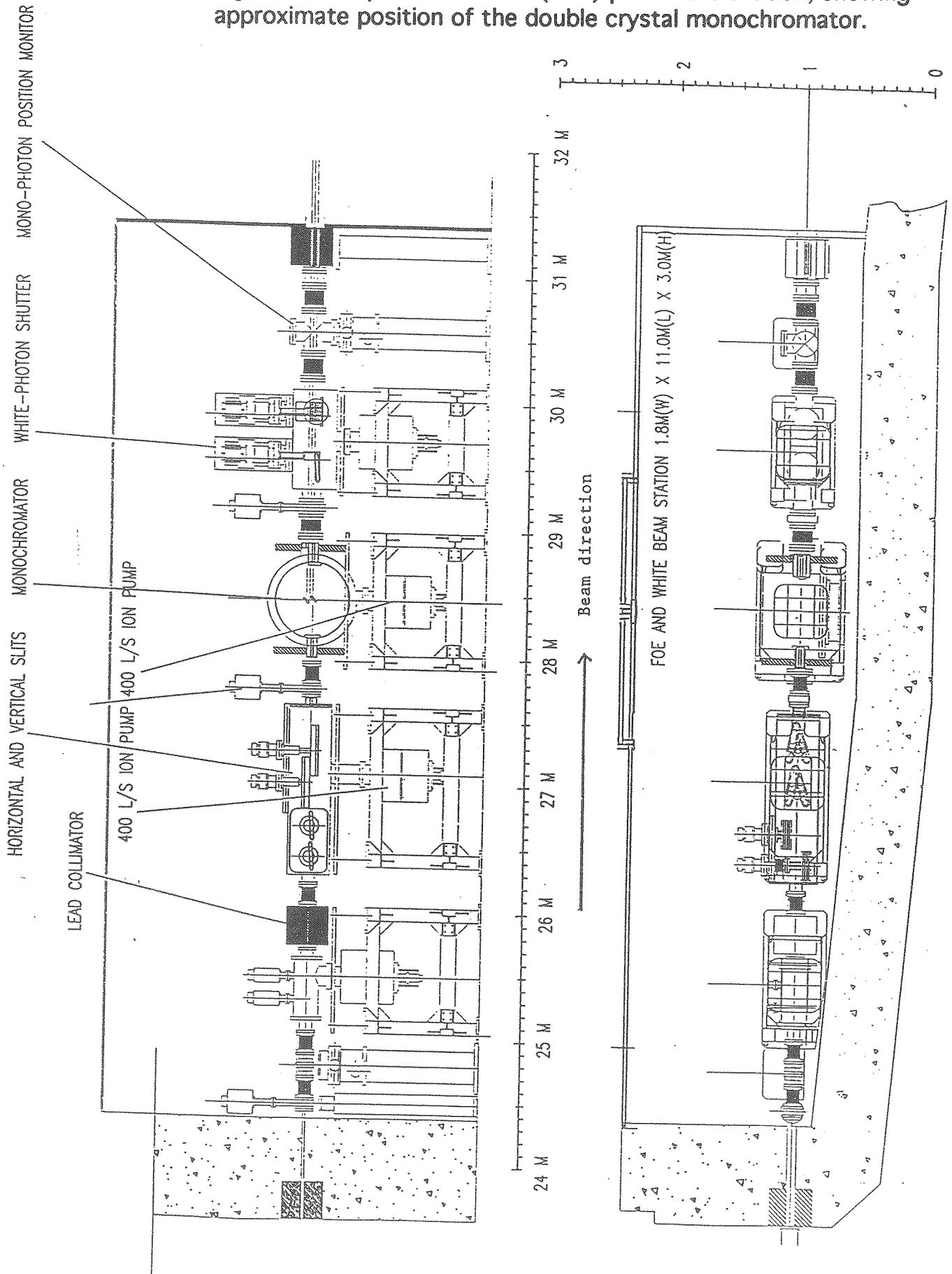
Fig. 5(a): Schematic of inclined crystal geometry, upstream view.

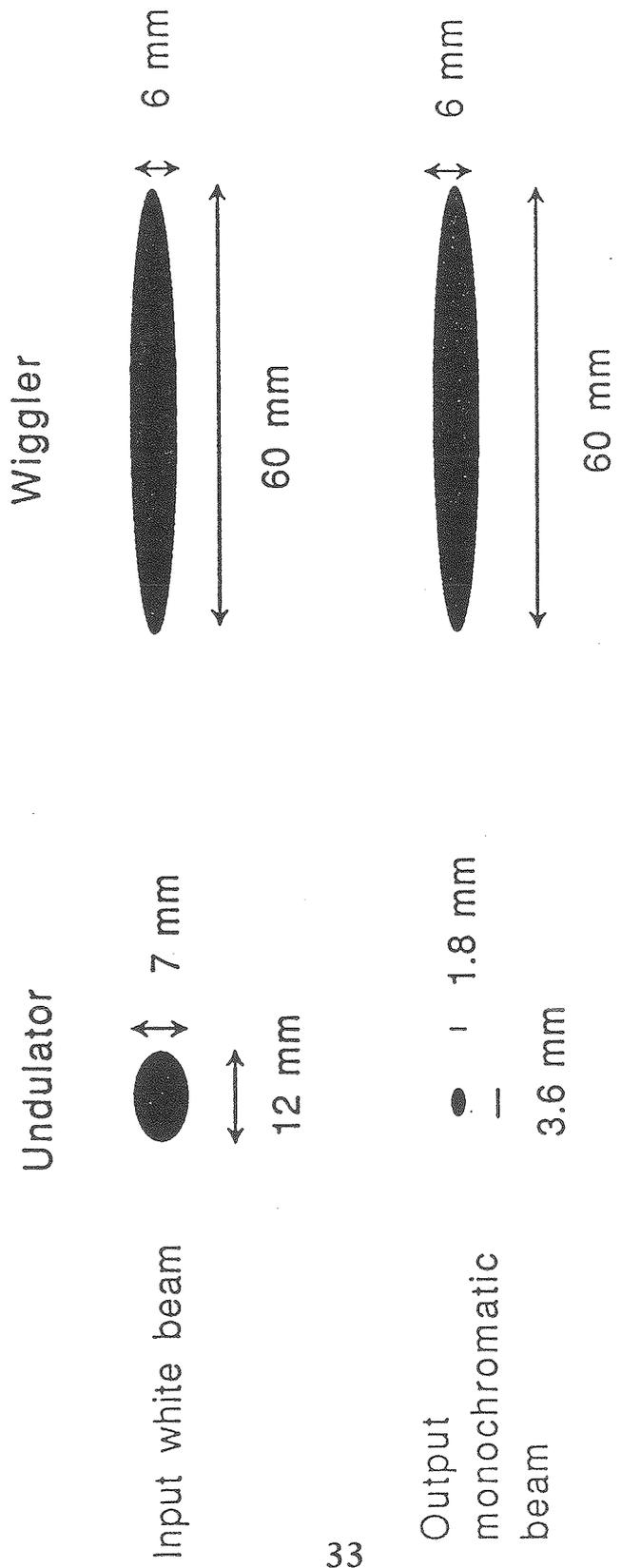


⊕ X_{TANK} : OPTION B - ENTIRE VACUUM TANK MOVES Laterally

Fig. 5(b): Schematic of inclined crystal geometry, side view.

Fig. 6: First optics enclosure (FOE) plan and elevation, showing approximate position of the double crystal monochromator.





Note: For the undulator, the output beam size can be as shown or as big as the input beam depending on the size of the input slits.

Fig. 7: Entrance and exit beam sizes of APS Undulator A and Wiggler A at 30 m from the source.

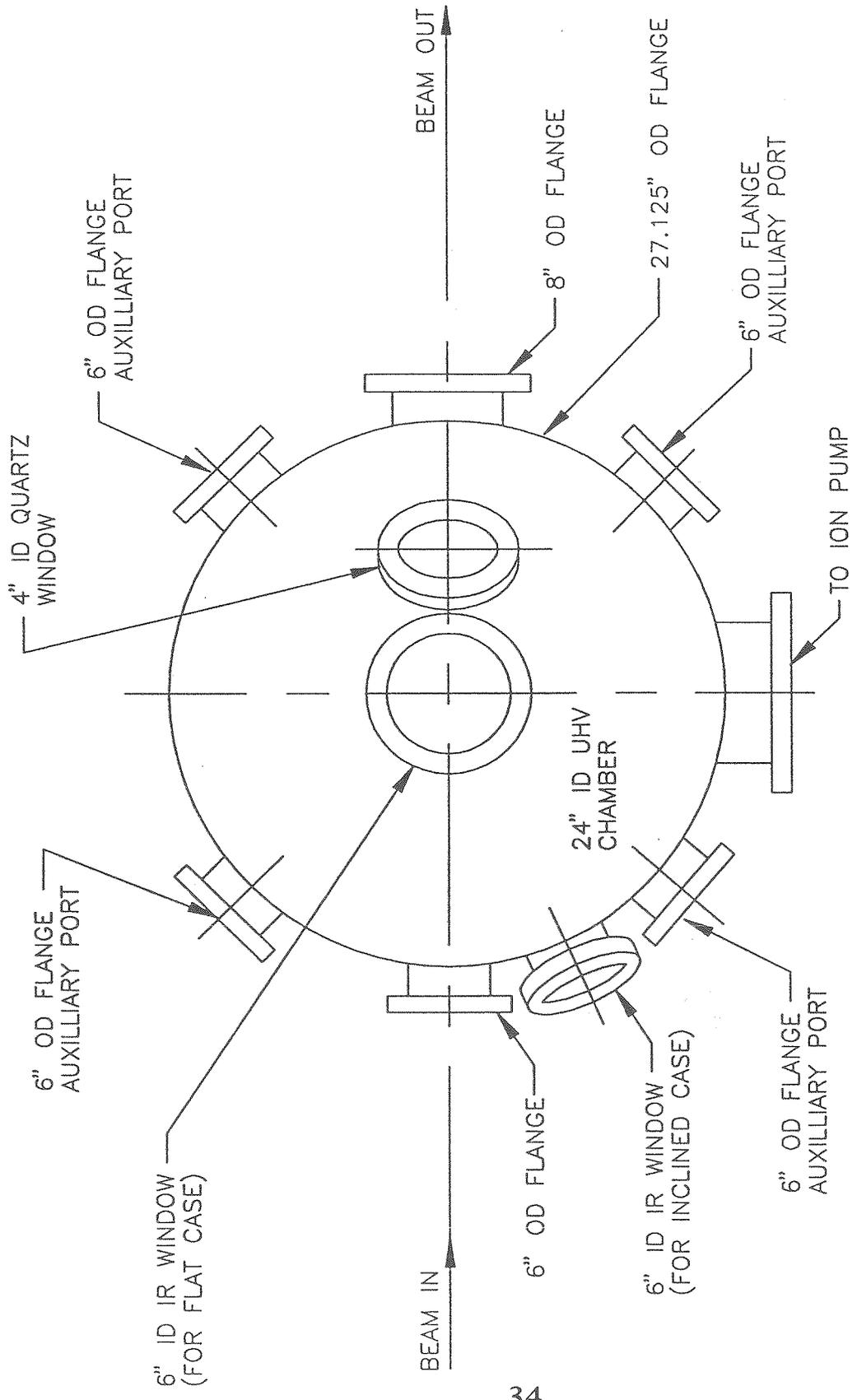


Fig. 8: Schematic of UHV chamber for double crystal monochromator.

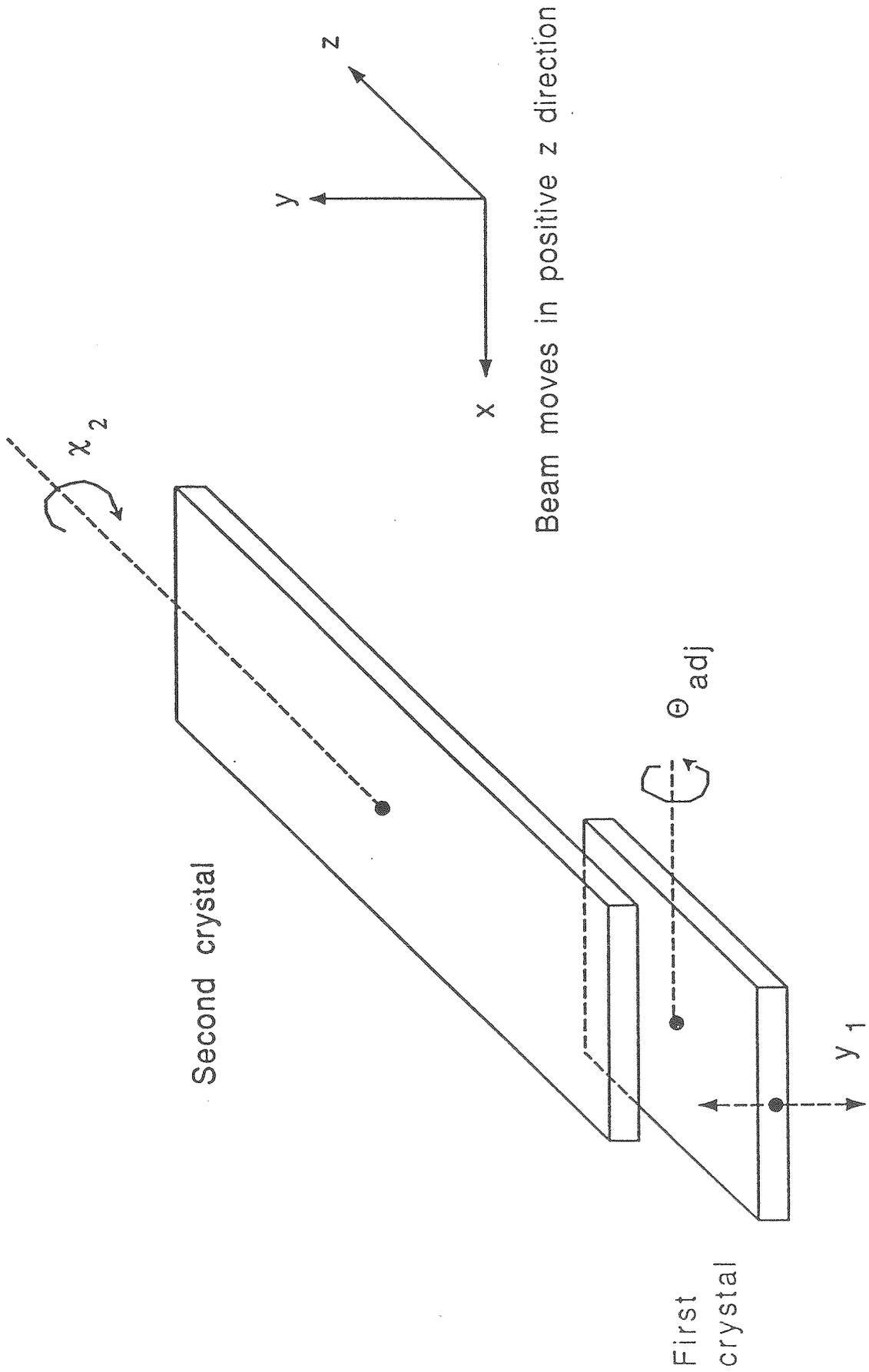


Fig. 9: Schematic of motions for the flat crystal geometry.

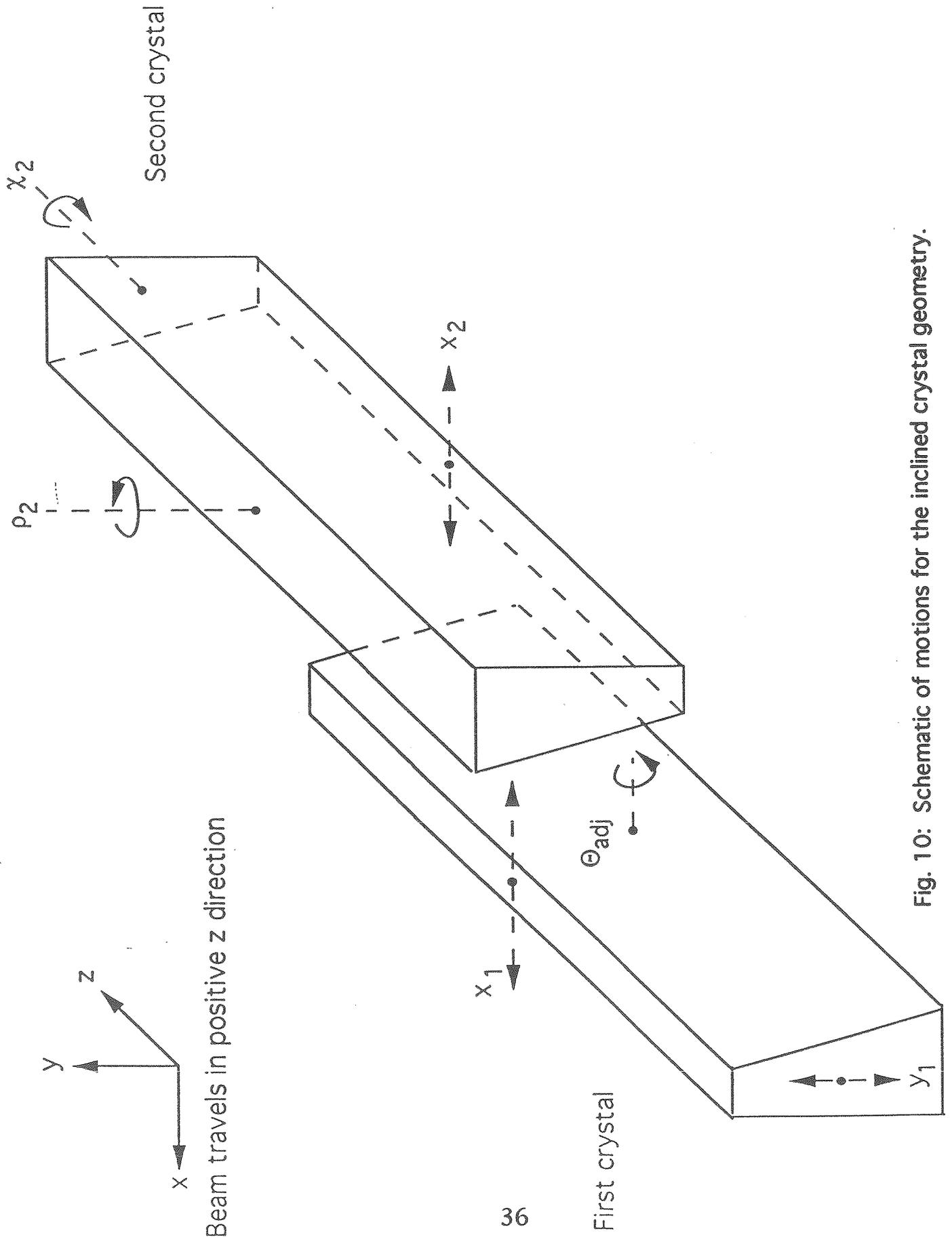


Fig. 10: Schematic of motions for the inclined crystal geometry.

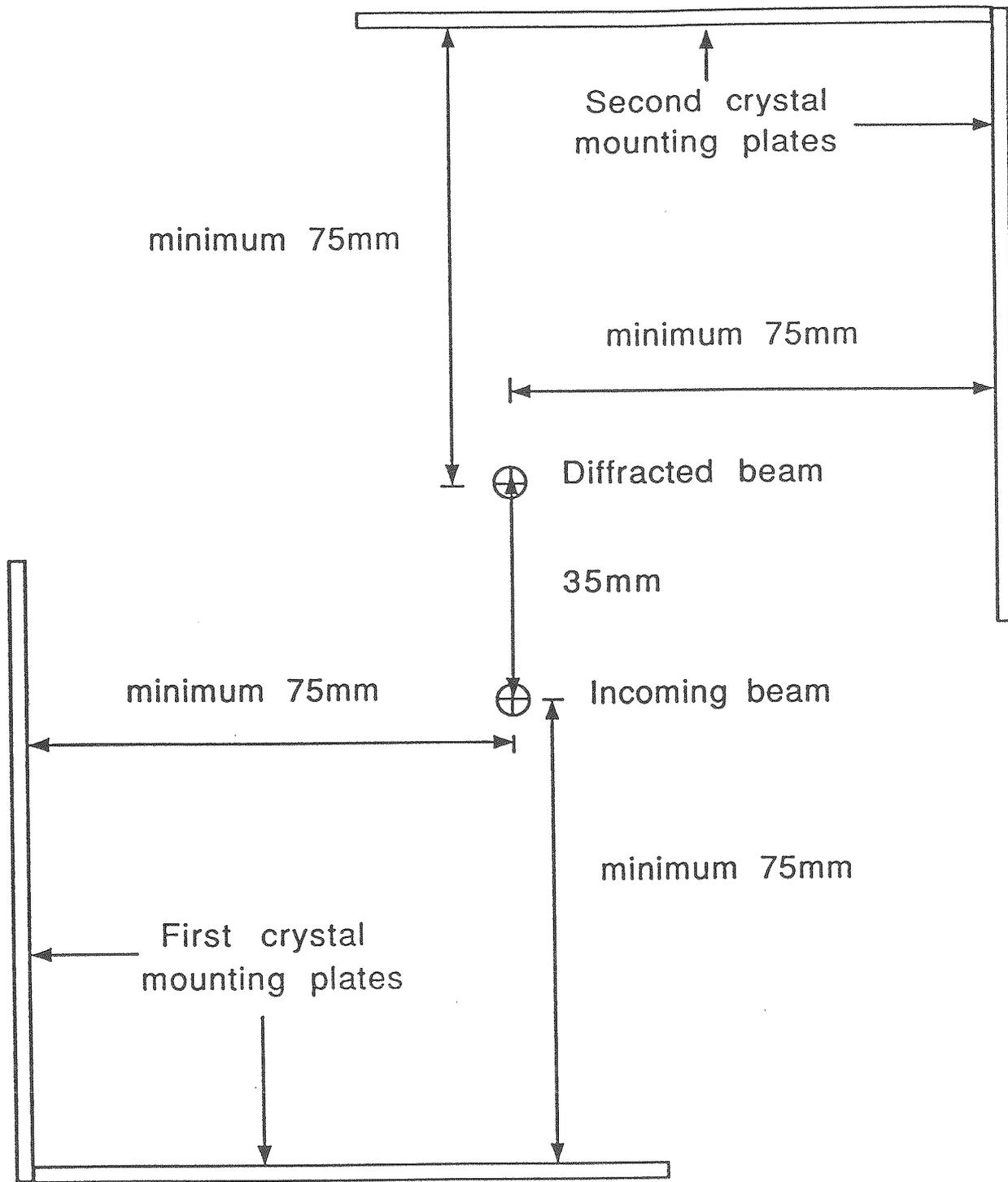
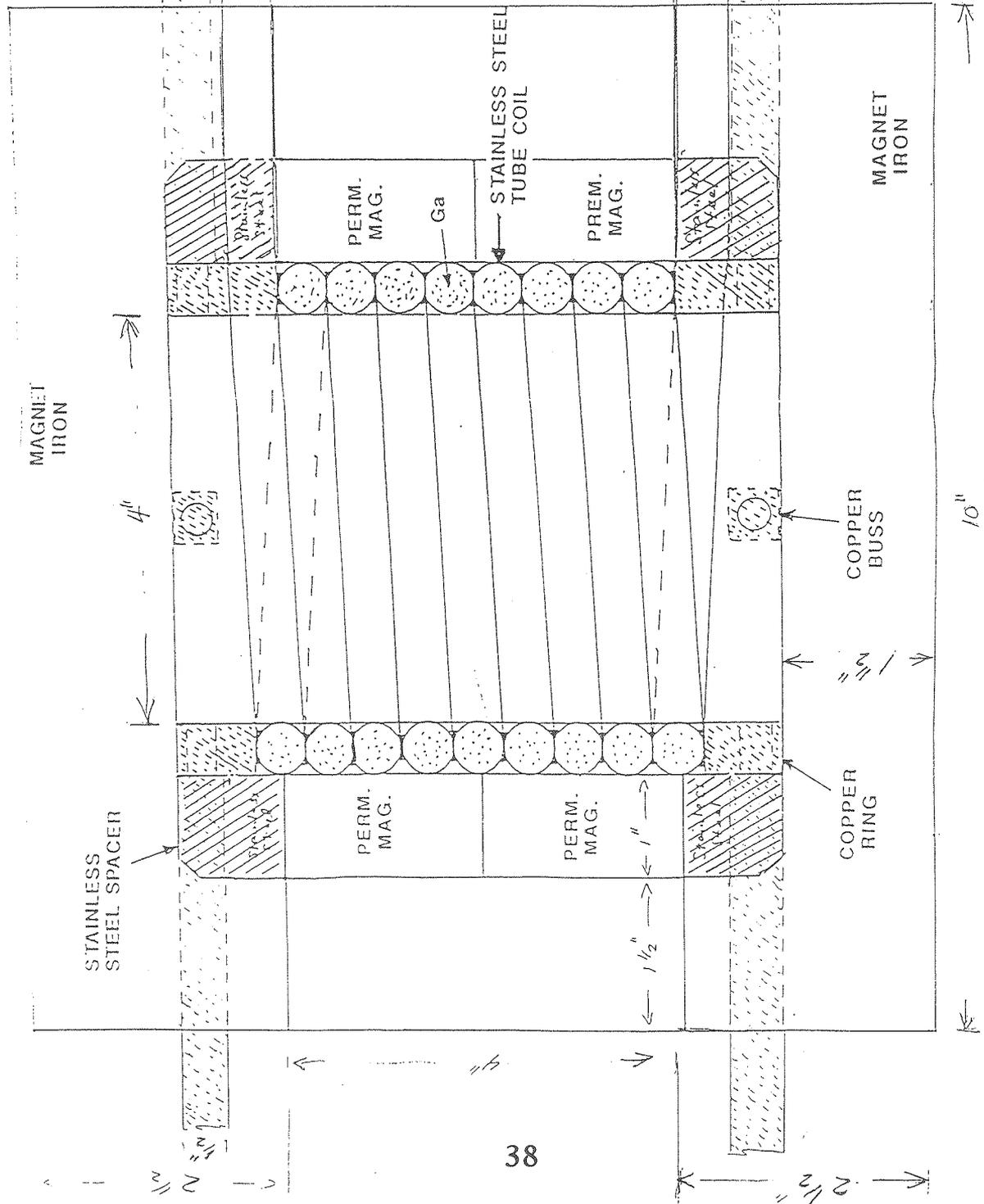


Fig. 11: Minimum clearances from beam centers to mounting plates.

NEW GALLIUM PUMP ASSEMBLY
USING PERMANENT MAGNETS
AND DC CURRENT SOURCE



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Fig. 12: Schematic drawing of DC current liquid gallium pump.

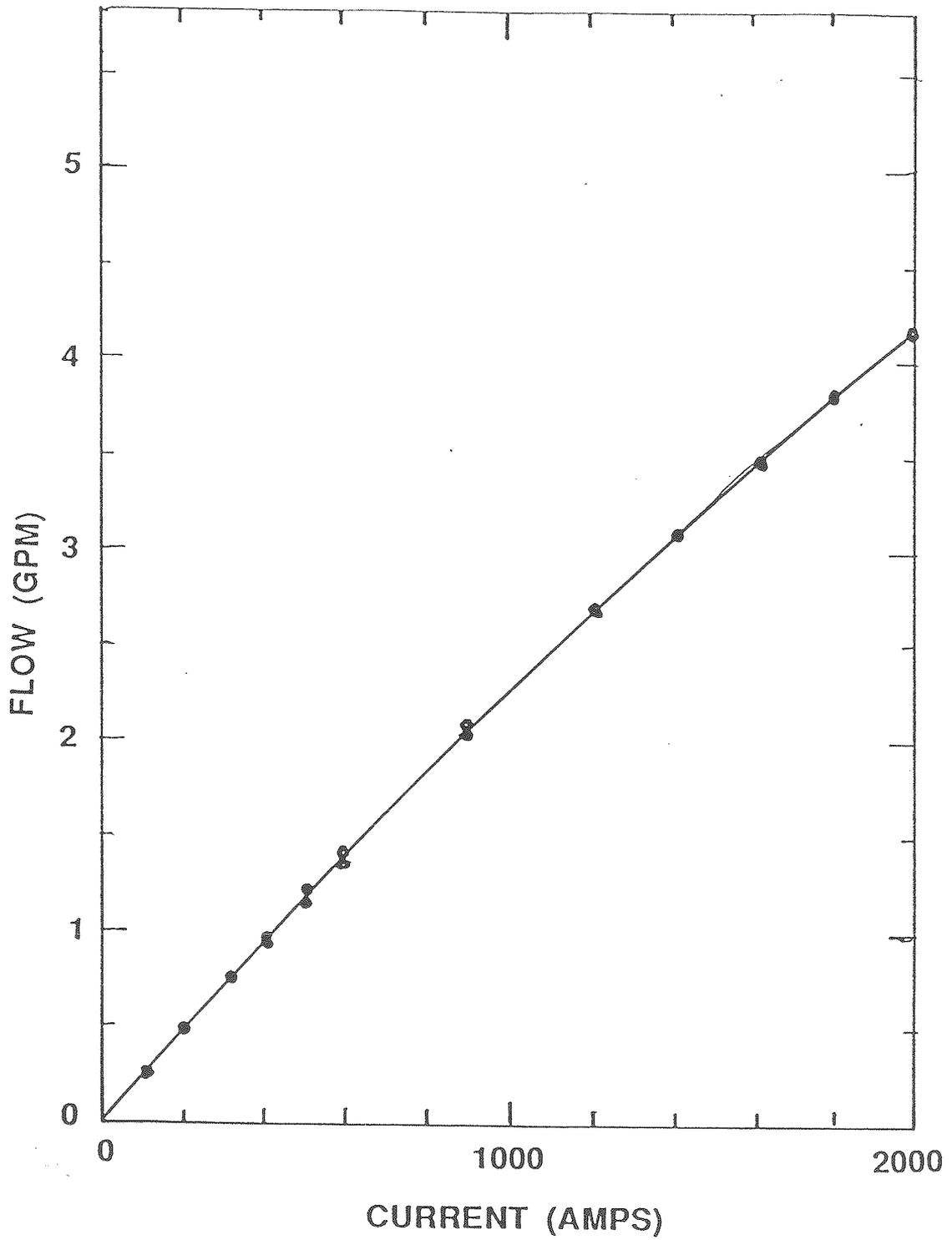


Fig. 13: Liquid gallium flow rate as a function of applied DC current in the APS prototype DC pump.

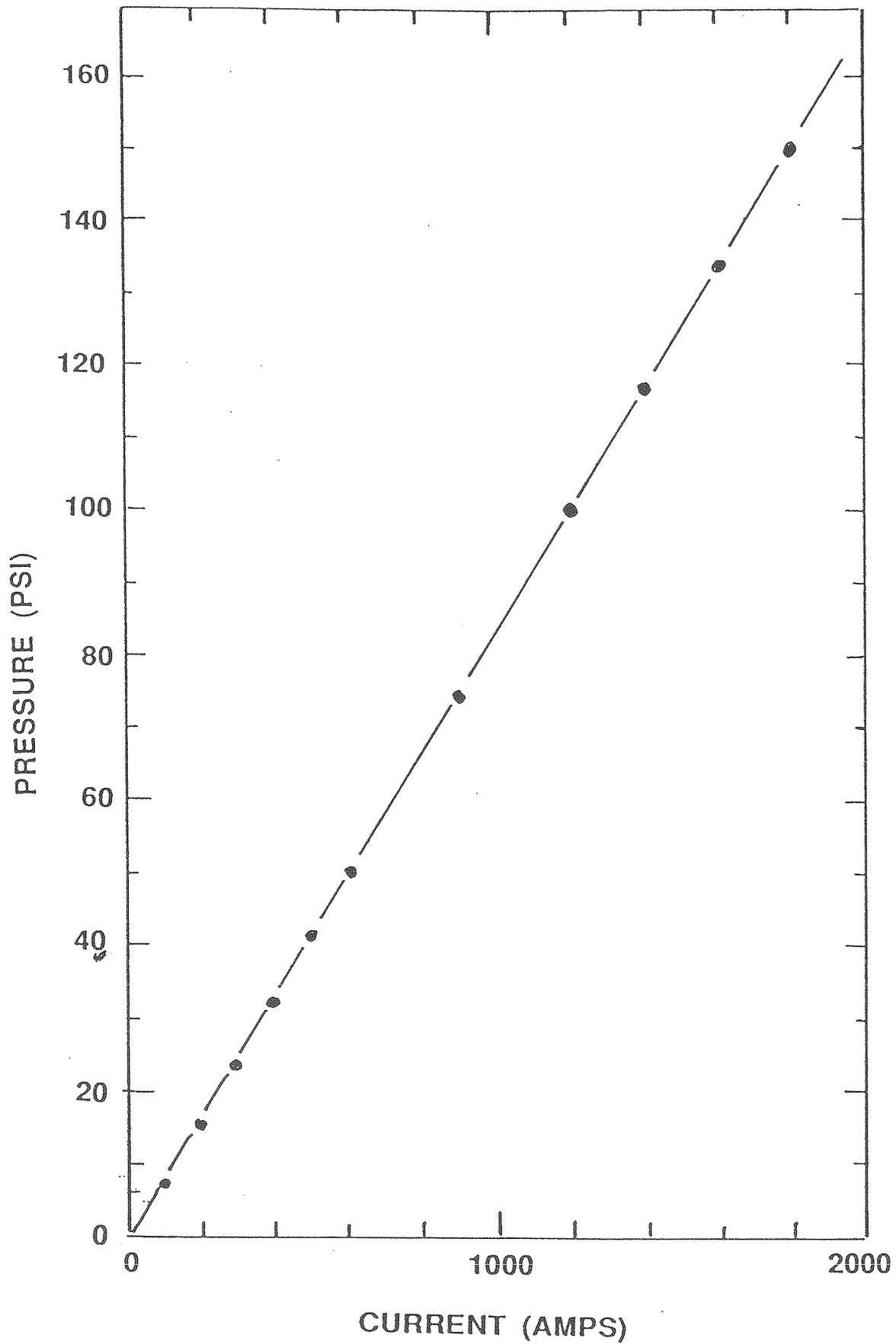


Fig. 14: Liquid gallium head pressure as a function of applied DC current in the APS prototype DC pump.

Fig. 15: Taken from reference 13. Comparison of reflectivity curves between a 85° inclination crystal and a flat crystal at 5 keV and 13.84 keV.

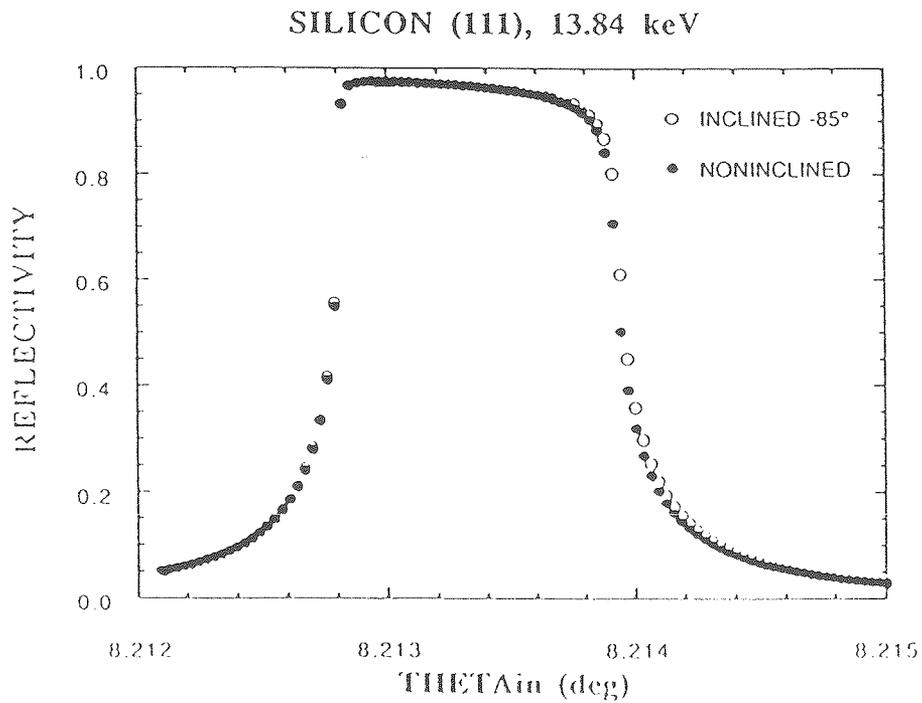
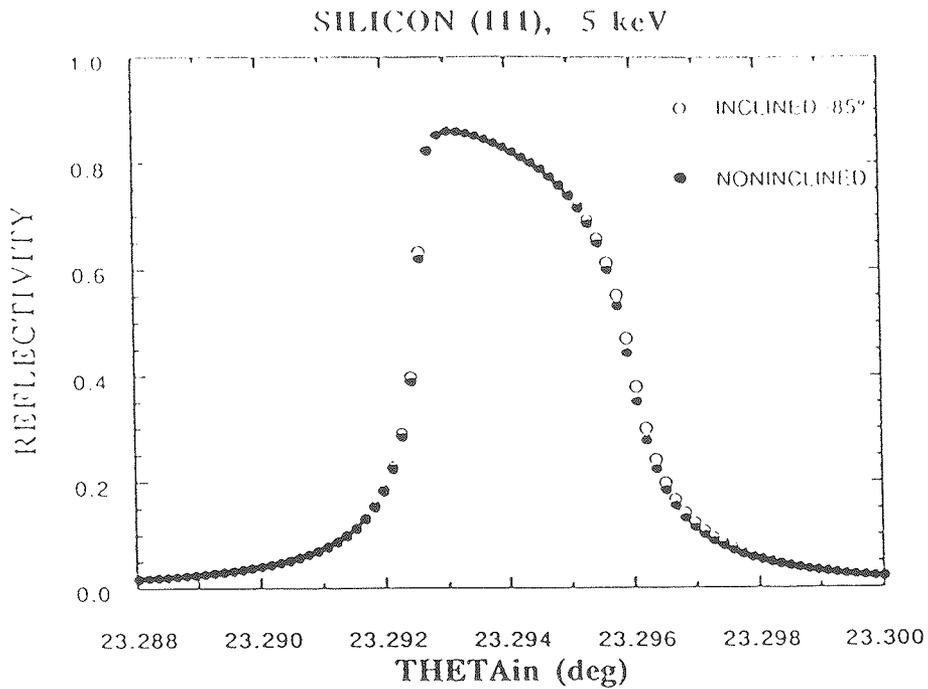


Fig. 16(a): Sketch of a 78 degree inclination Si(111) crystal for use in the 8 - 20 keV range. The footprints of the 8 keV and 20 keV cases are shown. The corners of the rectangle are cut off to avoid blocking the incoming or outgoing beams.

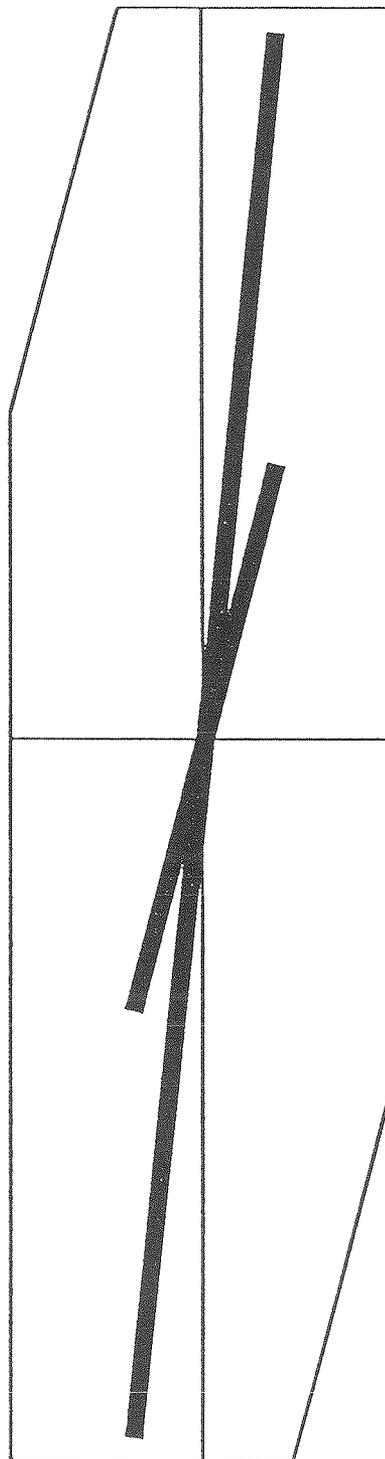


Fig. 16(b): Sketch of a 78 degree inclination Si(111) crystal for use in the 8 - 20 keV range, as a second crystal in a DCM with a 35 mm vertical offset. The footprints of the 8 keV and 20 keV cases are shown. The footprints are displaced from one another because the second crystal stays stationary during an energy scan.

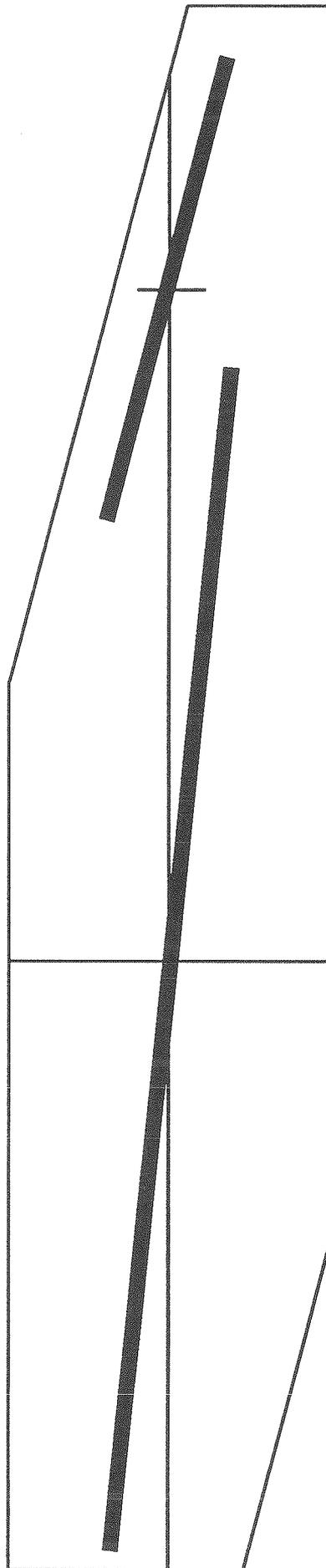


Fig. 18: Plot of the asymmetry parameter b , as a function of ρ , the rotation about the reciprocal lattice vector H . Positive is more grazing incidence. This case is for a 70.5° inclination angle at 5 keV for Si(111).

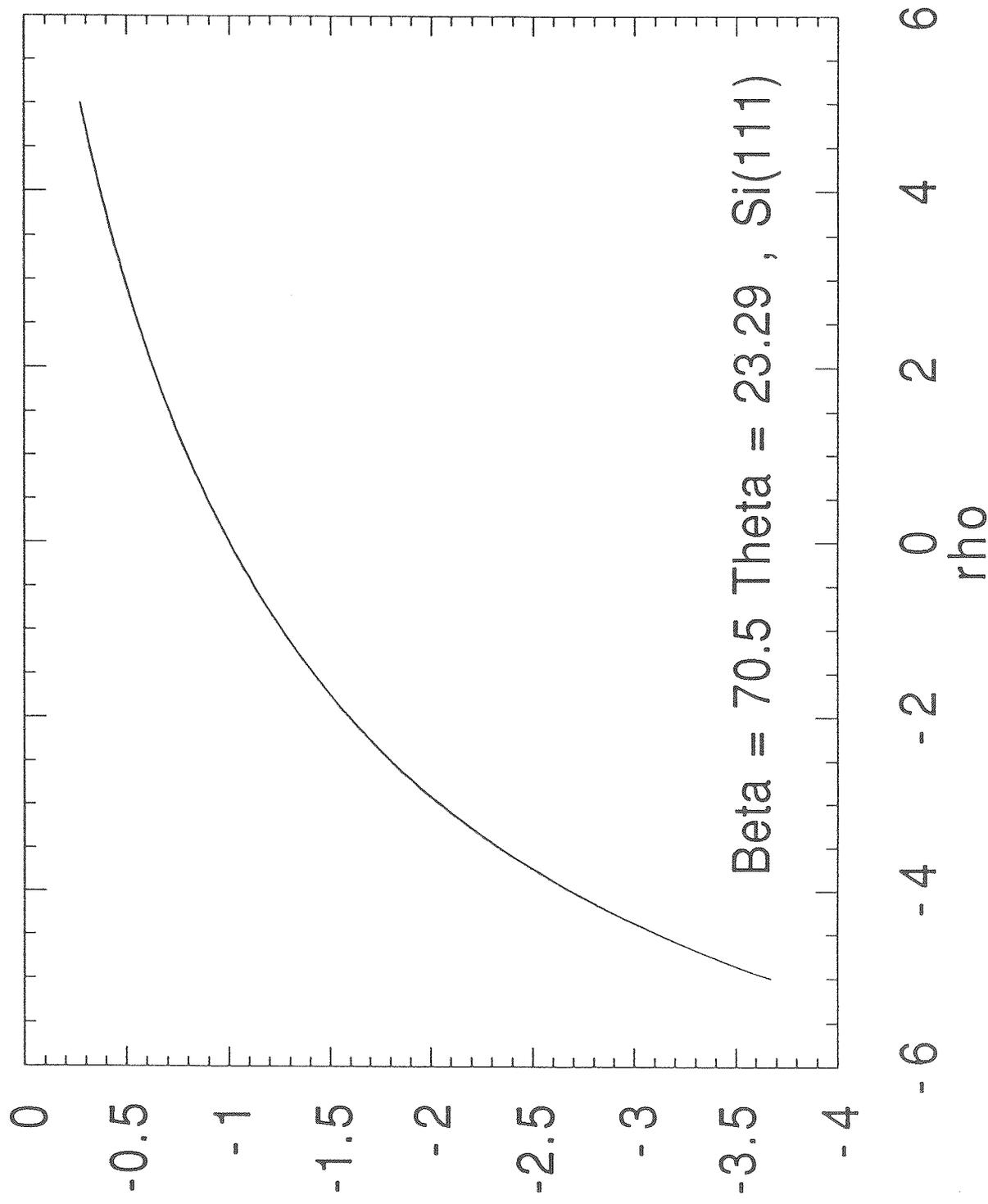
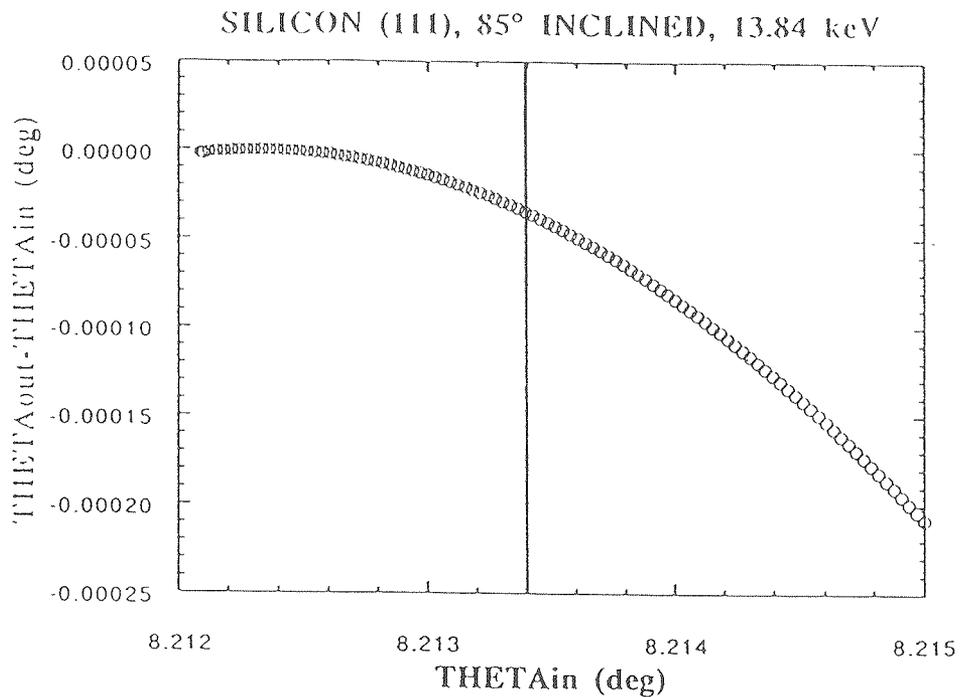
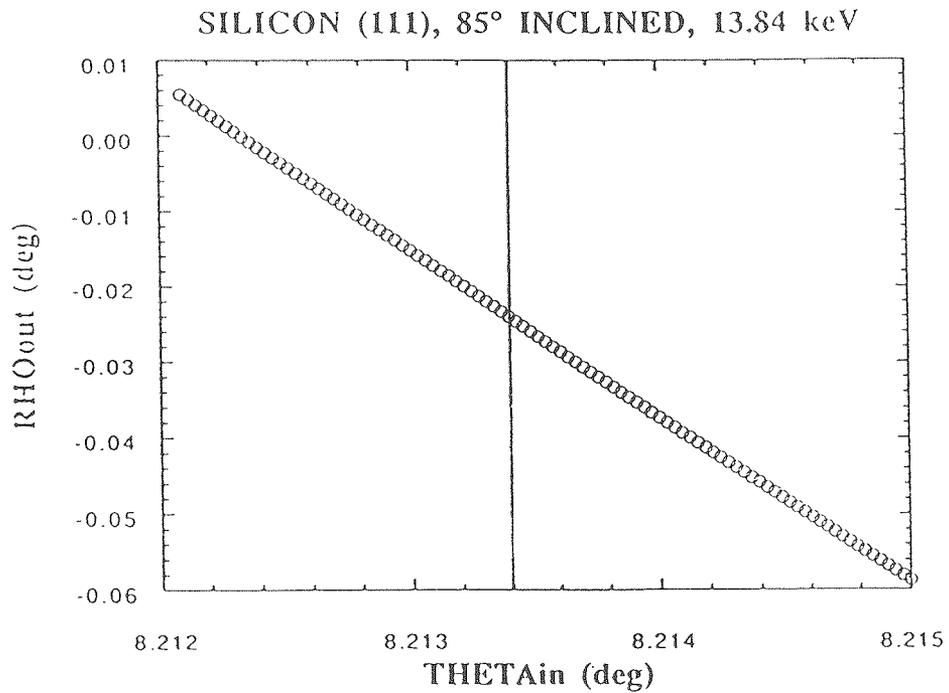


Fig. 19: Taken from reference 13. Difference between the incoming and outgoing angles of the beams, measured with respect to the (111) plane. This is for the case of an 85° inclination at 13.84 keV. The straight line denotes the center of the reflectivity curve.



4. The difference between the angles of the ingoing and outgoing beams where the angles of the beams are measured with respect to the (111) plane.

Fig. 20: Taken from reference 13. The amount of out-of-plane angular divergence as a function of the incoming beam, for the case of an 85° inclination Si(111) crystal at 13.84 keV. The straight line denotes the center of the reflectivity curve.



6. Values of ρ_{out} as a function of Θ_{in} for incident beams all having $\rho_{in}=0$.

APPENDIX A

Review of Safety-Related Precautions for the Use of Liquid Gallium Metal as a Cooling Fluid for X-ray Optics

I. INTRODUCTION

Recent experiments performed at Argonne and Cornell have established the usefulness of liquid gallium as a cooling fluid for the first optical elements in the intense photon beams that will be generated by the Advanced Photon Source [1]. Liquid gallium is so effective as a cooling fluid that it may be used with 50 % or more of the beamlines at the APS. The possibility of this type of extensive use of liquid gallium as a cooling fluid suggests that reasonable safety-related precautions for handling this material be established. The reader is advised that these precautions are recommendations as general guidelines and should not be relied on for achieving compliance with occupational safety and health regulations.

II. APS GUIDELINES

A. Precautions for Handling both Liquid and Solid Gallium Metal

1. When handling or working with either liquid or solid gallium metal, personnel should wear plastic gloves or gloves of other materials impervious to liquid gallium. A second pair of thick cotton gloves may also be needed for insulation if one is handling hot liquid gallium.
2. All personnel should wear lab coats or other protective clothing that can be removed should contamination occur.
3. Respirators with HEPA filters should be worn if airborne gallium compounds are present. Respirators should be worn when activities might suspend Ga dust in the air.
4. All personnel should wear safety glasses or goggles.
5. NIOSH-approved respirators should be worn if toxic vapors or fumes are being emitted.

B. Special Precautions for X-ray Optics Applications

1. Solid gallium metal should be melted in a plastic container placed in a water bath and heated to 50°C before the liquid gallium is added to an operating pumping system. If the system is being filled for the first time, it should be

preheated to 50°C before adding the gallium. This will insure that the gallium will fill the system properly and not leave any voids or bubbles.

2. Normal operating temperatures for gallium cooling systems are between 40°C and 60°C. Temperature excursions up to 200°C are permissible. Before operating at higher temperatures one must discern if all of the components can be operated safely at the elevated temperature. Often part of the system consists of plastic or Teflon tubing. These materials become weaker at elevated temperatures.

3. Liquid gallium will begin to interact with stainless steel at temperatures above 600°C, so operating temperatures should be kept well below this value. [2]

4. Liquid gallium does not appear to attack single crystals of silicon at temperatures of 100°C or less [3]. Some etching of silicon crystals by the In-Ga eutectic was reported in the literature for a 21 hour exposure at 350°C [4].

5. No precautions are taken against the possibility of vapor escaping from the surface of liquid or solid gallium because of its very low vapor pressure at any normally expected temperature in cooling applications. All available information suggests that the vapor pressure of liquid Gallium or the In-Ga eutectic will be less than 10^{-12} Torr at temperatures equal to or less than 300°C [4].

6. Gallium reacts readily with rolled aluminum plates by diffusing into the grain boundaries and reducing the aluminum plate to flakes. It also forms a variable mixture, dissolving the aluminum completely if enough gallium is present.

7. The In-Ga eutectic attacks Cu, Cu-Be, brass, Ag, and Mg at elevated temperatures (350°C) but not W, 304 stainless steel, Ni, C, Ge, Invar, SiC, or B. Pb, Cu, Cu-Be, Ag and brass were not affected at 20°C [4]. This would suggest that Ge crystals could be cooled with liquid gallium, however, further research is needed.

C. Housekeeping & Storage Precautions

1. Solidify liquid metal after use and place it in air-tight containers for storage. Refrigeration (cold storage) is recommended for long-term storage. Gallium melts near room temperature (29.87°C) and will coat the plastic container when liquefied resulting in a loss of material.

2. Collect all scraps or small particles of gallium and return to storage as soon as possible.

3. If a liquid spill should occur, pour cold water on the liquid until it freezes and then pick up with common housekeeping utensils.

4. Gloves should be worn during any clean-up procedure because the heat of one's hand will melt the surface of small amounts of gallium that will then interact with the moisture in the skin, coating the skin with gallium oxide.

5. Approved respirators should be worn during clean-up because small particles of gallium oxide and other compounds can become airborne and be inhaled or ingested.

D. Emergency & First Aid Procedures

Inhalation: Remove personnel to fresh air.

Eyes: Remove eye particles with cotton swab.
Flush with large quantities of water.

Skin: Wash off skin with soap and water.

Ingestion: Induce vomiting. Contact physician.

III. HAZARDS

A short summary of the potential hazards present with the use of gallium was published by A. J. McIntyre and B. J. Sherin in the September 1989 issue of *Solid State Technology* [5]. McIntyre and Sherin work in the Components Group of Hewlett-Packard Co in San Jose, CA. Their main interest and the main thrust of the article concerns the hazards associated with gallium arsenide, but they give the following evaluation of the hazards for gallium based on earlier work [6-8]:

“Elemental Ga is insoluble in water and is therefore poorly absorbed in mammals. Inorganic salts of Ga (e.g., gallium oxide, gallium trichloride, etc.) undergo hydrolysis of hydroxide, which is also insoluble and become colloidal in biological tissues. The toxicity of Ga compounds varies widely among animal species. Large animals (e.g., dogs) are 40 to 60 times more sensitive than mice and rats. GaCl₃ administered orally to rats in concentrations as high as 1000 ppm had no effect after 26 weeks because of negligible absorption. GaCl₃ was also not absorbed when inhaled in doses from 25 to 125 mg/m³ for 0.5 to 4 hrs. Severe intravenous overexposures are lethal because of kidney failure. Renal damage was similar to that seen for mercury.

Very little toxicological data is available on Ga effects in humans. Experiments, in general, on the Ga-containing drugs have shown that anorexia, nausea, vomiting, skin rashes, and depression of red and white blood cell counts can occur. Other clinical experiments on Ga-containing drugs have resulted in some bone marrow depression, dermatitis and severe itching and gastrointestinal disturbances.”

Three standard references were consulted for information on the hazards of industrial materials. The first, *Dangerous Properties of Industrial Materials*, seventh Edition, Volume II, edited by N. Irving Sax and Richard J. Lewis, Sr. [9] gives the following report from EPA TSCA Inventory:

"GALLIUM

CAS: 7440-55-3

NIOSH: LW 8610000

DOT: 2803

af: Ga aw: 69.72

PROP: A beautiful, lustrous, silvery liquid or metal or a gray solid. Mp: 29.78°C, bp: 2403°C, d (solid): 5.904 gm/cc @ 29.6°C, d (liquid): 6.1 gm/cc@ 29.8°C.

DOT Classification:	ORM-B; Label:	None, liquid;
	ORM-B; Label:	None, solid;
	Corrosive Material; Label:	Corrosive

THR: Poison by subcutaneous and intravenous routes. Corrosive; probably an eye, skin and mucous membrane irritant. It has a metallic taste, causes dermatitis and depression of bone marrow function. Potentially explosive reaction with hydrogen peroxide + hydrochloric acid. Violent or vigorous reaction with halogens. Forms an amalgam with aluminum alloys."

For the gallium compounds, in general, it gives:

"GALLIUM COMPOUNDS

THR: Preliminary investigations were done with the oxide, tartrate, benzoate, and anthranilate, which were used by some investigators in the treatment of syphilis. Amounts up to 15 mg/kg of body weight were injected intravenously and were tolerated without harm by laboratory animals. Larger doses produced hemorrhagic nephritis. In the case of gallium lactate, work done at the Naval Medical Center Research Institute showed that intravenous injections of about 40 mg/kg of body weight in rats or rabbits were lethal. Metallic gallium as well as the nitrate produced no skin injury, and subcutaneous injections of relatively large amounts could be tolerated both by rabbits and rats without evidence of injury. It has, however, been demonstrated that gallium remains in the tissues for long periods of time following intramuscular injections of soluble gallium salts. Tissue distribution experiments indicate that it behaves like bismuth and mercury in that one respect."

It also gives information on many of the other common Ga-compounds.

The second reference consulted was the *Encyclopedia of Chemical Technology*, Third Edition, Volume 11, pg 604, chapter entitled, "Gallium and Gallium Compounds." This reference [10] gave considerable information on the properties and production of gallium. Most of the production of gallium occurs as a by-product of the refining of aluminum or zinc. Its abundance in the earth's crust is similar to that of lead. Their report on the toxicology of gallium reads as follows:

“TOXICOLOGY

The toxicity of metallic gallium or gallium salts is very low. The corrosive, poisonous, or irritating nature of some of its compounds is attributable to the anions or radicals with which it is associated. Gallium metal-organics, such as $\text{Ga}(\text{CH}_3)_3$, react vigorously with air, and can be explosive. The gallium halides, except fluoride, hydrolyze in water to form the corresponding halogen acids. Gallium phosphide, arsenide, selenide and telluride react slowly with water and more vigorously with acids and bases, to liberate toxic compounds. The LD_{50} (lethal dose, 50%) of the solution for mice is ca 3-4 g/kg $\text{Ga}(\text{NO}_3)_3$. The ^{72}Ga and ^{67}Ga isotopes were studied, e.g., as citrate salts, for detection of tumors: ^{72}Ga concentrates in bone tissue and ^{67}Ga seems to have a tumor-specific affinity.”

The third reference was Sigma-Aldrich Library of Chemical Safety Data, Edition I, Ed. R.E. Lenga, (1985), revised in July 1990 [11].

GALLIUM (1990)

TOXICITY HAZARDS

RTECS No. LW8600000

Reviews, Standards, and Regulations:

NOHS 1974: HZD 34585; NIS 2; TNF 163; NOS 4; TNE 427
NOES 1983: HZD 34585; NIS 5; TNF 130; NOS 9; TNE 2871;
TFE 929

EPA TSCA Chemical Inventory, 1986
EPA TSCA Test Submission (TSCATS) Data Base, Jan. 1990

The following data are selected from the Registry of Toxic Effects of Chemical Substances (RTECS). See actual entry in RTECS for complete information.¹

HEALTH HAZARD DATA

ACUTE EFFECTS

Harmful if inhaled or swallowed.

May cause eye irritation.

May cause skin irritation.

¹ Note that RTECS can be accessed on-line through MEDCARS or the NIH/EPA Chemical Information System.

To the best of our knowledge, the chemical, physical and toxicological properties have not been thoroughly investigated.

FIRST AID

In case of contact, immediately flush eyes with copious amounts of water for at least 15 minutes.

In case of contact, immediately wash skin with soap and copious amounts of water.

If inhaled, remove to fresh air. If not breathing give artificial respiration. If breathing is difficult, give oxygen, call a physician.

APPEARANCE AND ODOR

Shiny silver solid.

EXTINGUISHING MEDIA

Dry chemical powder.

SPECIAL FIRE FIGHTING PROCEDURES

Wear self-contained breathing apparatus and protective clothing to prevent contact with skin and eyes.

UNUSUAL FIRE AND EXPLOSIONS HAZARDS

Emits toxic fumes under fire conditions.

REACTIVITY DATA

INCOMPATIBILITIES

Strong acids.

Strong oxidizing agents.

Halogens.

Strong bases.

Protect from moisture.

HAZARDOUS COMBUSTION OR DECOMPOSITION

Nature of decomposition products not known.

SPILL OR LEAK PROCEDURES

STEPS TO BE TAKEN IF MATERIAL IS RELEASED OR SPILLED:

Sweep up, place in a bag and hold for waste disposal.

WASTE DISPOSAL METHOD

Material in element state should be recovered for reuse or recycling.

Observe all federal, state, and local laws.

PRECAUTIONS TO BE TAKEN IN HANDLING AND STORAGE

Wear appropriate NIOSH/MSHA- approved respirator, chemical resistant gloves, safety goggles, other protective clothing.

Safety shower and eye bath.

Mechanical exhaust required.

Wash thoroughly after handling.

Keep tightly closed.

Moisture sensitive.

Store under nitrogen.

Refrigerate.

The following is a note added by Sigma-Aldrich:

THE ABOVE INFORMATION IS BELIEVED TO BE CORRECT BUT DOES NOT PURPORT TO BE ALL INCLUSIVE AND SHALL BE USED ONLY AS A GUIDE. SIGMA-ALDRICH SHALL NOT BE HELD LIABLE FOR ANY DAMAGE RESULTING FROM HANDLING OR FROM CONTACT WITH THE ABOVE PRODUCT. SEE REVERSE SIDE OF INVOICE OR PACKING SLIP FOR ADDITIONAL TERMS AND CONDITION OF SALE.

IV. SUPPLIER INFORMATION & MATERIAL SAFETY DATA SHEET

To date, the gallium metal has been supplied by United Mineral & Chemical Corp., 1100 Valley Brook Ave, Lyndhurst, NJ 07071. It is supplied as high purity metal (99.9999%) so no hazardous ingredients were present. The manufacturer is required to supply a Material Safety Data Sheet with each delivery). The following information comes from this data sheet:

SECTION 3 - Physical & Chemical Characteristics:

Boiling Point:	2403°C
Specific Gravity:	5.904 gm/cm³
Percent Volatile by Volume:	N.A.
Vapor Density (Air = 1):	N.A.
Evaporation Rate:	0 (due to very low vapor pressure)
Solubility in Water:	insoluble
Reactivity in Water:	none
Appearance & Color:	silvery gray metal solid, no odor
Flash Point:	N.A.
Extinguisher Media:	not required
Auto-ignition Temperature:	does not auto ignite
Special Fire Fighting Procedures:	none
Unusual Fire & Explosion Hazards:	presence of acids and high heat can evolve toxic fumes

SECTION 4 - Physical Hazards

Physical Stability:	stable, avoid temperatures above 29.78°C (it melts)
Incompatibility:	avoid contact with acids, halogens
Hazardous Decomposition Products:	none
Hazardous Polymerization:	will not occur

SECTION 5 - Health Hazards

Threshold Limit Value:	no figures
Signs & Symptoms of Exposure	
Acute Overexposure:	metallic taste, can cause dermatitis, obvious blood in stool
Chronic Overexposure:	gastrointestinal problems, skin & hemorrhagic nephritis resulting from soluble gallium salts (gallium is soluble in acids)
Chemically Listed as Carcinogen or Potential Carcinogen:	no
National Toxicology Program:	no
I.A.R.C. Monographs:	no
OSHA:	no
OSHA Permissible Exposure Limit:	no figures
ACGIH Threshold Limit Value:	no figures
Emergency & First Aid Procedures:	
Inhalation:	remove personnel to fresh air
Eyes:	remove eye particles with cotton swab; flush with large quantities of water
Skin:	wash off skin with soap and water
Ingestion:	induce vomiting; contact physician

SECTION 6 - Special Protection Information

Respiratory Protection:	wear NIOSH-approved toxic vapor respirator for gallium compound fumes.
Ventilation:	local exhaust, mechanical
Protective Gloves:	plastic

Eye Protection: glasses or safety goggles

**Other protective clothing
or equipment:** lab coat

SECTION 7 - Special Precautions & Spill / Leak Procedures

Precautions to be Taken in Handling & Storage:

Store in refrigerator. Handle with plastic gloves to prevent material contamination. Dust mask to be worn to prevent material contamination (through ingestion or inhalation).

Steps Taken in Case Material is Released or Spilled:

Solid gallium can be picked up with ordinary housekeeping utensils. Liquid gallium should have cold water spilled on it to solidify it.

Waste Disposal Methods:

Place in labeled container, which should be heat sealed in plastic and disposed of with EPA service. Consider for waste reclamation."

V. REFERENCES

1. R. K. Smither, et. al., Rev. Sci. Instrum. **60** (1989) 1486.
2. George Forster, ANL, private communication.
3. R.K. Smither, et. al., Nucl. Instrum. & Methods A **266** (1988) 517.
4. W.R. Hunter, and R.T. Williams, Nucl. Instrum. & Methods **222** (1984) 359.
5. A. J. McIntyre and B. J. Sherin, Solid State Technology **32** (1989) 119.
6. H.C. Dudley, K.E. Henry, B.F. Lindsey, Studies of the Toxic Action of Gallium," Journal of Pharmacology and Experimental Therapeutics **97** (1978) 409.
7. B. Venugopal, T.D. Luckey, "Toxicity of Group III Metals," in *Metal Toxicity in Mammals. Chemical Toxicity of Metals and Metalloids*, (Plenum Press, New York, 1978).
8. H. E. Stokinger, "The Metals," in *Patty's Industrial Hygiene and Toxicology*," 3rd Ed., Vol. **2A**, (John Wiley & Sons, New York, 1981).

9. *Dangerous Properties of Industrial Materials*, 7th Edition, Vol III, edited by N. Irving Sax and Richard J. Lewis, Sr., (Van Nostrand Reinhold, New York), p. 1793.
10. Kirk-Othmer, "Gallium and Gallium Compounds," in *Encyclopedia of Chemical Technology*, 3rd Edition, Vol. 11, edited by D. Echroth, (John Wiley & Sons, New York, 1988) p. 604.
11. "Sigma-Aldrich Library of Chemical Safety Data," Edition I, (1985) p. 987, revised in 1990.

**Appendix B - Specification Sheet for Neodymium Iron Boron
Permanent Magnets**

Magnetic Characteristics	
Peak Energy Product—(BdHd) max. $\times 10^6$	35
Residual Induction B_r —Gauss	12,100
Coercive Force H_c —Oersteds	11,300
Intrinsic Coercive Force H_{ci} —Oersteds	$\geq 14,000$
Curie Temperature— C°	300
Temperature affecting material— C°	150
Reversible temperature coefficient— $\%/C^\circ$ For magnets operating at (BH) max.	.10
Magnetizing Force H_s — KO_ϵ	> 28

Appendix C - Magnetic Iron Specifications

PRODUCT DESCRIPTION

CMI-C Electromagnetic Iron Rod is specially processed with a critical strain for optimum uniformity. Maximum magnetic properties are achieved following suggested final anneal applied to fabricated parts.

• BENEFITS

Hi-permeability, low coercivity.
Low loss provides highest force/watt input.

• EASY ANNEAL

Response of magnetic properties to short anneals of 750°C—850°C offers customer savings in heat-treating costs, as compared to expensive, high-temp decarb anneals.

• AVAILABILITY

CMI-C Rod is available out-of-stock in most common fraction diameters to 2 3/4" on a 1-2 week delivery. Larger sizes are also available. CMI-C Grade (0.02%C) is available in:

- Hot Rolled Bar Rounds
- Wire (cold heading quality)
- Square-edged Flats
- Round-cornered Squares (forging quality)

• MAGNETICS

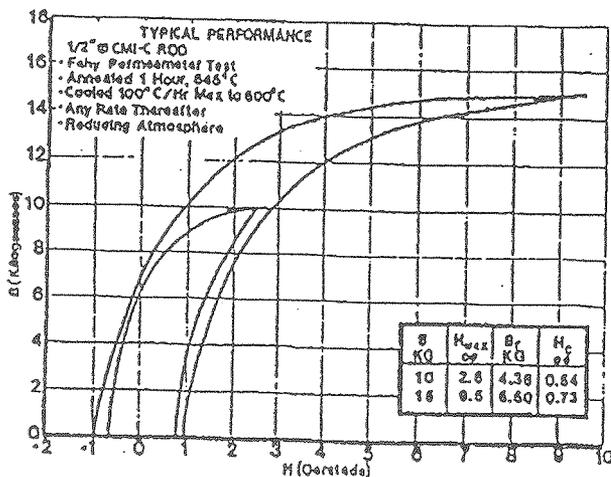
All material is specially processed for electromagnetic applications with certified magnetics. CMI products will meet the magnetic requirements of most OOD, Military and Commercial specifications. (OVER)

CHEMICAL COMPOSITION

	C	Mn	P	S	Si	Al	N
Typ.	.01	.30	.008	.01	.01	.04	.003
Max.	.02	.38	.01	.02	.06	.06	.004

TYPICAL MECHANICAL PROPERTIES

Hardness, Rb	65-85
Tensile, Yield, 0.2% Offset KPsi	85
Ultimate KPsi	87
Elongation in 4 x Dia. %	20.8
Area Reduction %	78.1
Youngs Modulus KPsi	27.7



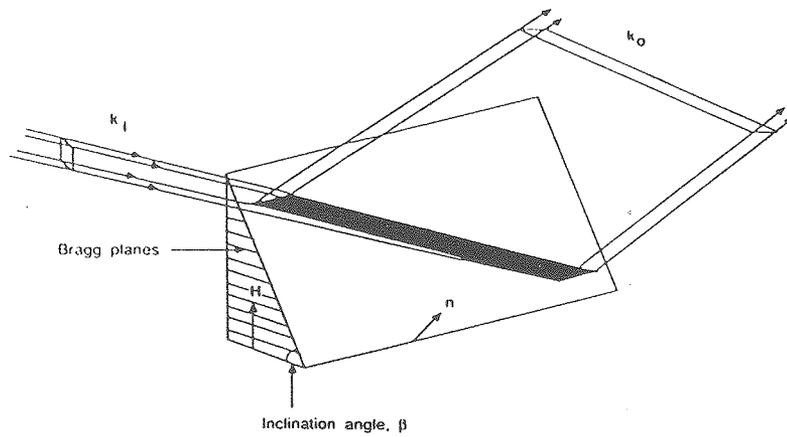
** CMI-C conforms to magnetic specifications of: ASTM A-848-87
AMS 7708
MIL-I-11695
Dwg 71 AF 45549
NARM-122

• APPLICATIONS

COLD DRAWN ROUNDS	Solenoid and relay cores, plungers, magnetic control devices.
HOT-ROLLED BAR	Generator and motor field frames, and pole pieces. Magnetic chucks, loud speakers parts, clutch plates.
FORGINGS	From hot and cold drawn rounds for magnetic recording/playback heads and flux collectors, acoustic devices and similar user requiring highest possible flux density with minimum ampere-turns and low coercive force.

Appendix D - The Inclined Crystal Geometry

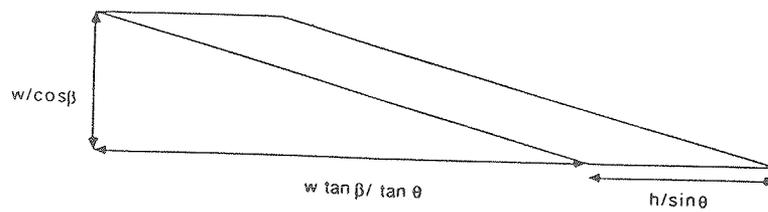
The inclined crystal geometry:



Disregarding the small beam distortion effects, the following shapes are useful for designing inclined beam optics. For an incoming beam profile:



The footprint on the first crystal is:



The beam profile after one inclined reflection is:

