

SCHOOL OF MATH AND SCIENCE STANDARD OPERATING PROCEDURE 5104.8C

From: Dean, School of Mathematics and Science

Subj: SAFETY OPERATION AND PROCEDURES FOR THE PANalytical X'Pert PW-3040-PRO
MPD SERIES X-RAY DIFFRACTOMETER (SERIAL NUMBER 201866)

Ref:

- (a) PANalytical Pro Users Guide Safety Manual, 2005
- (b) NAVMED P-5055 "Radiation Health Protection Manual"
- (c) NAVSEA S0420-AA-RAD-010 (RAD-010) "Radiological Affairs Support Program Manual"
- (d) X'Pert PRO MPD X-ray Diffraction System, 2009

Encl: (1) Facility Description
(2) Facility Radiation Safety Evaluation
(3) General Controls and Practices
(4) General Operating Instructions
(5) Emergency Procedures
(6) Radiation Monitoring
(7) Operator Qualification Requirements
(8) Records – Sheets and Logs

1. Purpose. This instruction contains the procedures to be followed for the safe operation of the PANalytical X'Pert PW-3040-PRO MPD Series X-ray diffractometer, serial number 201866.

2. Cancellation. This instruction updates MATHSCIDIVINST 5104.B.

3. Applicability. This SOP shall be used to govern operation of PANalytical X'Pert PW-3040-PRO MPD Series X-ray diffractometer located in Chauvenet Hall CH-044.

4. Discussion. The PANalytical X'Pert PW-3040-PRO MPD Series diffractometer uses x-rays to determine crystalline or amorphous character of materials made in the condensed matter laboratories. The primary purpose of the diffractometer is to give midshipmen practical experience in the physics description of solids. The laboratory is also used to support midshipman and faculty research. This instruction establishes the responsibilities of individuals involved with the safe operation and maintenance of the diffractometer.

5. Responsibilities

a. The Laboratory Manager shall:

- (1) ensure that the PANalytical diffractometer is operated only in accordance with this SOP.
- (2) ensure and certify that operators are familiar with this SOP and with references (a) through (c) and are able to operate the diffractometer safely.
- (3) maintain the facility records in the facility binder and postings on the facility walls and door.
- (4) conduct periodic surveillance checks of the facility to ensure that hazard areas are posted.
- (5) complete a quarterly Audit of the facility, records and postings, and maintain the record in the

facility binder.

(6) complete a quarterly Surveillance of an Authorized User and maintain the record in the facility binder.

(7) ensure that required Usage Log and Radiation Survey Logs are properly maintained.

(8) investigate and report alleged or suspected incidents of overexposure to the Radiation Health Officer via the Radiation Safety Committee.

(9) keep RSO informed of any issues related to management of the PANalytical laboratory, to include recommending (to the RSO) the removal of Authorized User qualification as needed.

(10) endorse qualified users to the RSC to be approved Authorized Users.

b. Authorized Users

(1) Machine sources at USNA are only to be operated by Authorized Users, or by qualified operators under the direct supervision of an Authorized User. In order to qualify as an Authorized User, the individual must meet the following conditions:

(a) the individual shall be a DoD employee or student or shall have an official appointment to the USNA faculty/staff.

(b) the individual shall have been designated as an Authorized User by the Radiation Safety Officer.

(c) the individual shall have attended the training program provided by the Radiation Safety Office. The training will be commensurate to the type of risk they are exposed at the workplace.

(d) the individual shall have received on job training from the Laboratory Manager covering the specific procedures and attendant hazards associated with the work to be performed in accordance with this SOP.

(e) students taking the SP442 course and SP495/SP496 research courses will not normally be classified as Authorized Users.

(2) Authorized Users are not required to be classified as Radiation Workers. All personnel shall strive to control their radiation exposure in accordance with the "ALARA principle - As Low As Reasonably Achievable".

(3) The Authorized User shall:

(a) be responsible for all aspects of diffractometer operation, including the radiation safety of everyone in and around the diffractometer room.

(b) ensure that they are a qualified Authorized User, as defined in Enclosure (7) of this SOP, before attempting to operate the diffractometer.

(c) ensure that they are familiar with the contents of this SOP, its enclosures and the relevant parts of the references.

- (d) operate the diffractometer only in strict accordance with this SOP.
- (e) complete and record all checks required on the Usage Log sheet before starting the diffractometer.
- (f) in the event of an abnormal shutdown or equipment malfunction, stop all operations, make a note on the Usage Log and contact the Lab Manager.
- (g) ensure that the diffractometer lab facility is securely locked and that the computer is screen locked when an Authorized User is not present.
- (h) log off the computer and return the key to the lockbox when diffractometer operations are completed.
- (i) ensure that radiation surveys are current prior to commencing routine operations.

c. Other Personnel

(1) Other personnel, midshipmen and guests that have access to CH-044 are allowed access to the laboratory room housing the diffractometer to use other equipment. Other personnel in CH-044 not working with or who do not have authority to operate the diffractometer shall not utilize the computer connected to the diffractometer, the diffractometer itself, the water chiller, or the sample preparation area when the machine is turned on.

(2) Other personnel entering the laboratory room housing the diffractometer, CH-044, to use the diffractometer shall:

- (a) Be accompanied by an Authorized User when the machine is turned on.
- (b) Only operate the instrument under the guidance of an Authorized User.
- (c) Control their radiation exposure in accordance with the "ALARA principle – As Low As Reasonably Achievable".
- (d) Follow all directions of the Authorized User.
- (e) Follow all provisions of this SOP.

d. The Radiation Health Officer shall:

- (1) establish standards of radiological health requirements.
- (2) specify and provide personnel monitoring or dosimetry devices.
- (3) maintain records of personnel exposure.
- (4) certify that all users who require TLDs have satisfactorily completed the required Radiation Physical examination before issuing TLDs.

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(5) investigate alleged or suspected incidents of overexposure and prepare the necessary reports required by the Bureau of Medicine and Surgery and the Navy Radiological Affairs Support Office, Yorktown, Virginia.

e. The Radiation Safety Officer

(1) The Radiation Safety Officer shall:

(a) Have authority to terminate immediately any operation that he/she finds to be unsafe or which is not in accordance with this or other applicable instructions.

(b) Ensure that area monitoring surveys are conducted and recorded every 6 months and prior to commencing routine operations after equipment maintenance or modification.

(c) Conduct initial radiation safety training of operators as required by reference (c), provide appropriate annual refresher training and certify the satisfactory completion of such training.

(d) Provide support as necessary to ensure compliance with higher authorities.

(e) Provide immediate assistance should any radiological safety incident occur.

f. The RSC shall:

(1) approve users submitted by the lab manager as Authorized Users. This may be performed during quarterly meetings or on an ad hoc basis.

6. Review. This instruction will be in effect for the entire time that the PANalytical X'Pert PW-3040-PRO MPD Series X-ray diffractometer is installed at the U.S. Naval Academy. The SOP will be reviewed on an annual basis by the Lab Manager and the RSO to ensure that the procedures are current and in full compliance with reference (c). Operators must review this SOP on an annual basis. This instruction must undergo complete review every five years by the Naval Academy Radiation Safety Committee.

7. Approvals. This SOP and all references contained therein have been reviewed and approved by the US Naval Academy Radiation Safety Committee. Operation or maintenance of the diffractometer in any manner or in reference to any procedure not described herein must first be approved by the RSO. Approval of such requested operation, procedure, or both will be temporary and will not constitute general approval by the RSO of the requested operation or procedure.

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FACILITY DESCRIPTION

The PANalytical X'pert PW-3040-PRO MPD Series X-ray diffractometer is an instrument that uses a monochromatic X-ray source and a solid-state detector to determine lattice and crystal structure, atomic spacing, and phase identification of both powder and solid materials.

The primary purpose of this X-ray diffractometer is to be used in the Solid-State Physics class (SP442) to study and determine the lattice structure of chemical compounds. The secondary purpose is to give the Physics Department a research grade X-ray diffractometer for both midshipmen and faculty/staff research projects.

The diffractometer, sample preparation area, water chiller, and the computer for data acquisition and analysis are all located in Chauvenet Hall, CH-044, as illustrated in Fig. 1 and Fig. 2. This room is surrounded by one classroom (CH-045) on the North-West side, a research lab on the South-East side (CH-042), two research labs and one classroom on the South-West side (CH-025) (CH-021) (CH-017), and a hallway on the North-East side. Chauvenet CH-044 is on the Zero-deck or ground floor. Below CH-044 is the basement or steam tunnels, and above CH-044 on the first deck are two classrooms (CH-156) (CH-157). The interior walls are non-load bearing and are made of standard gypsum block and gypsum board for fire-resistance.



Fig. 1. The PANalytical X-ray diffractometer is located in Chauvenet Hall CH-044, a 30-foot by 40-foot room in the middle of the Chauvenet Hall Zero-deck.

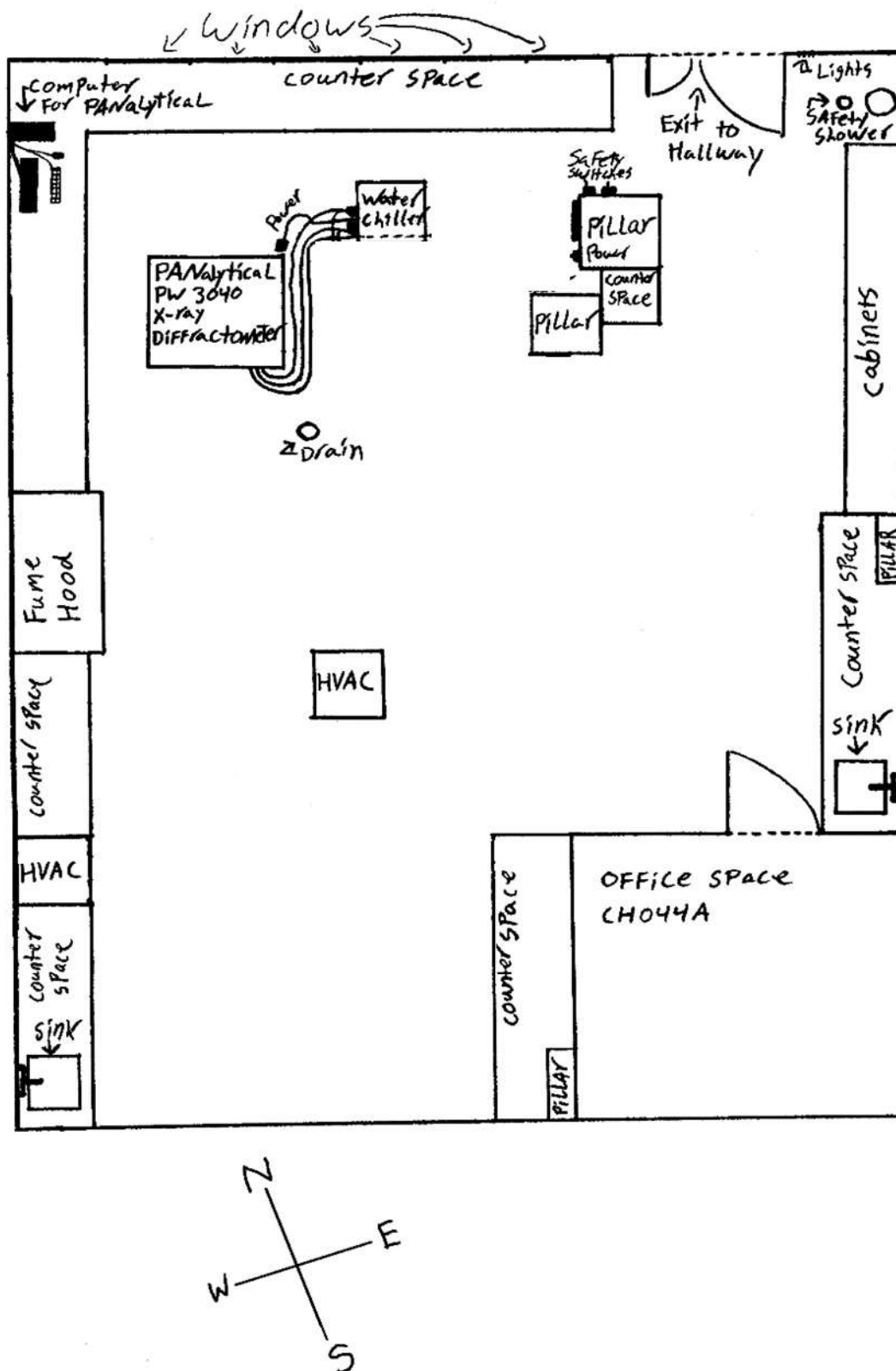


Fig. 2. Location of diffractometer equipment in Chauvenet Hall CH-044.
The relative orientation of the diagram is specified.

Enclosure (1)

Entrance to Chauvenet CH-044, the room that houses the diffractometer is available through the hallway door. The hallway door for CH-044 is normally kept locked when the room is unattended.

Chauvenet CH-044 has one standard floor drain into the sanitary sewer system. There is one safety shower and eye wash station next to the door. There are 6 windows in the room along the north-east wall and one in the hallway door. There are two sinks in the room; one located on the south-east wall and one located on the north-west wall. An office space is in the back of the room along the south-east and south-west walls. The room has 2 overhanging HVAC vents, rated for a minimum of 100 fpm per vent. The room has one fume-hood rated for a minimum of 100 fpm and houses acids, perchlorates, and solvents.

Chauvenet CH-044 is used as a class room to hold the Solid-State Physics class (SP442) along with other experimental research equipment and a Denton sputter coater. Personnel needing to use other equipment housed in CH-044 that have key access to the room have open access to CH-044 at any time.

Volatile radioactive materials are not used in Chauvenet CH-044. The diffractometer does not generate any radioactive material.

Doors are posted with emergency contact information. There is no radiological hazard when the diffractometer is off. Warning and status lights on the diffractometer, showing operational status, can be seen from the windows along the hallway wall.

PANalytical X'Pert PW-3040-Pro MPD Series Diffractometer

The PANalytical X'Pert PW-3040-PRO Multi-Purpose-Diffractometer Series X-ray diffractometer, serial # 201866, is a single source, high powered, high resolution, multi-functional X-ray diffractometer designed for collecting X-ray diffraction data on powders and solid samples.



Figure 3. View of the diffractometer hutch.

Experimental Area

The working environment, which is the upper part of the PW-3040 instrument enclosure, is constructed from steel. Those parts of the enclosure in the direct X-ray beam are 12 mm thick. Access to the inner part of the enclosure is via two fully interlocking sliding doors at the front of this instrument enclosure. The windows in the sliding doors are made of lead glass with an X-ray absorption equivalent to 1 mm of lead. The construction conforms to the most stringent X-ray safety standards; the absorbed dose equivalent rate is less than 1 $\mu\text{Sv/h}$ at 10 cm distance from the outside surface of the enclosure. See Figure 3.

The X'pert PRO PW-3040 instrument is equipped with a high stability 3 kW high-tension generator, and a water-cooled high-powered ceramic X-ray tube with a copper anode as its source, producing Cu K-alpha, (1.5405 Å) radiation. The max operating parameters for the x-ray tube are (60kV or 60 mA) with a maximum output of 3000 Watts total. The instrument collects data from the solid-state PIXcel-1D detector. The PIXcel-1D detector has an extremely high dynamic range, high count rate linearity and small strip size, resulting in fast and high quality data collection. The beam is tightly collimated and focused using modern X-ray optics: the user has the option of either manual optics or semi-automatic programmable optics. The beam can be adjusted to be as wide as 30mm by 30mm or as small as 5mm by 5mm across the sample. This instrument has a theta-theta axis goniometer in the vertical position with a max resolution of 0.005°; the X-ray tube and incident beam optics ride on the left arm of the goniometer and the detector and diffractive beam optics ride on the right arm of the goniometer. The sample sits horizontal in the equatorial plane in either a standard flat-clip bracket or in an automated reflection-transmission spinner bracket. See Figure 4. To prevent exposure of personnel to the X-ray beam, interlocks keep the beam from entering the sample chamber unless the enclosure is secure. The diffractometer is controlled using the local computer via RS232 cable.

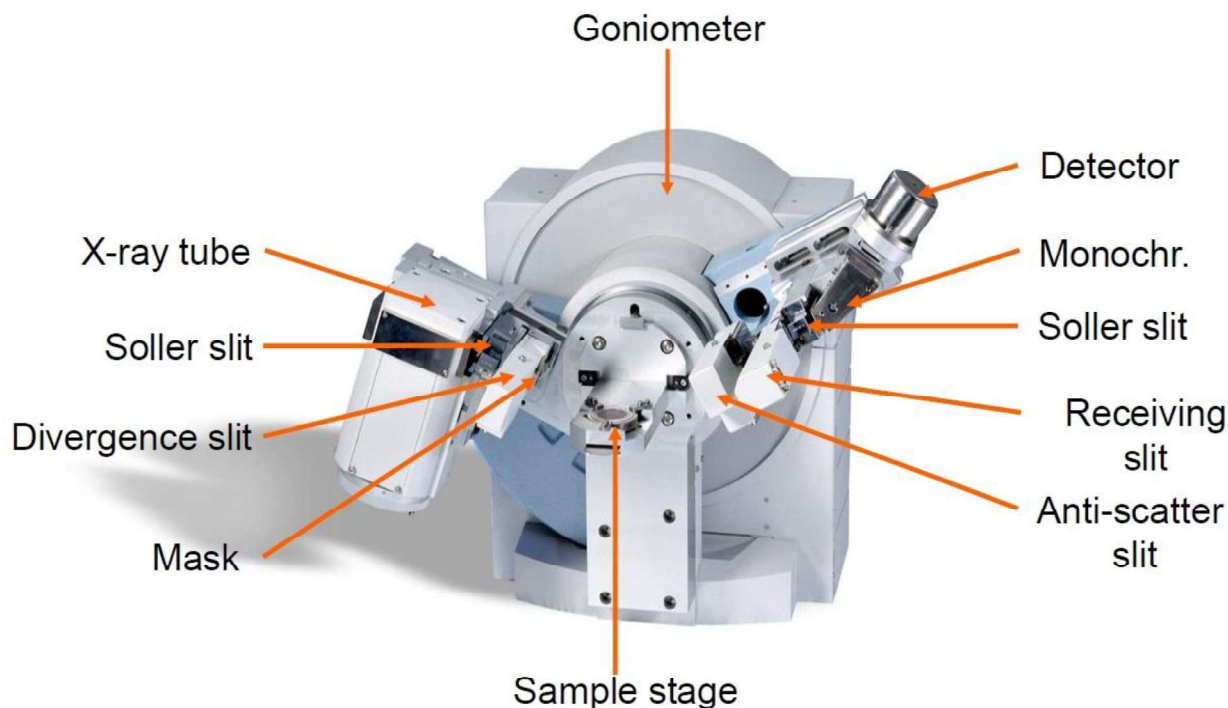


Fig. 4. The goniometer and components.

FACILITY RADIATION SAFETY EVALUATION

Hazard Discussion

The PANalytical PW-3040-Pro Series X-ray Diffractometer is classified as an enclosed beam X-ray facility diffractometer. Safety requirements for closed-beam, stationary X-ray equipment are listed in Section 13.1 of Reference (d), RAD-010 Rev 2. Issues concerning radiation safety that pertain to the diffractometer are:

1. The radiation field outside the hutch is $\ll 0.1$ mrem/hr and not observable with Navy-approved RADIACs. As a result, the following requirements have been met:
 - a. The dose rate from high voltage rectifiers shall not exceed 2 mrem/hr.
 - b. Under normal operation, an operator would not be able to experience a dose of 0.25 mrem/hr.
 - c. The beam stop assembly prevents readings greater than 0.25 mrem/hr outside of the hutch.
 - d. In the surrounding area of the room, the radiation field is less than 0.25 mrem/hr or 10 mrem/week.
2. The diffractometer complies with all of the requirements of a closed beam system.
 - a. The radiation source, sample and detector are enclosed in a chamber (the hutch).
 - b. The hutch is protected by a fail-safe interlock system.
 - c. Operators are prohibited by the fail-safe interlock system from entering the hutch while the system is producing X-rays. The hutch is locked during operation and can only be opened by pressing the Shutter Close button. Pushing this button closes the shutter, which then releases the locking mechanism on the hutch.
 - d. The beam cannot operate when the hutch is open while the interlock system is in place.
 - e. Operators are considered to be non-radiation workers and are not required to wear personnel thermoluminescent dosimetry devices.
3. The diffractometer is only to be serviced by personnel who have been trained and authorized by the Laboratory Manager or service personnel from PANalytical. A Radiation Survey must be performed after any alignment procedure has been performed.
4. The diffractometer does not activate material and, as a result, does not produce any airborne radioactivity.

GENERAL CONTROLS AND PRACTICES

Physical Controls for Chauvenet CH-044

Security:

1. The room housing the diffractometer is a Controlled Area and is normally locked when unattended. Access shall be controlled by locking the door to the room for security and safety reasons.
2. The diffractometer main power is controlled by a switch behind the back panel. Removal of the back panel is required to flip the switch.
3. The diffractometer High-Tension is enabled by a keyed switch on the front panel.
4. The key shall be removed from the switch and placed in its stored location when the diffractometer has been powered down.
5. The user account on the computer that controls the diffractometer is password protected to prevent access by unauthorized users. This user account shall be screen locked whenever an Authorized User is not present while the diffractometer is on.
6. The software that controls the diffractometer is password protected to prevent use by unauthorized users.

Warning Devices:

1. Lighted warning signs.

(a) The diffractometer hutch is equipped with lighted warning sign that operate automatically when the diffractometer controls are set to produce radiation. See Figure 5.



Fig. 5. Front Control / Display Panel – power on

(b) The warning sign “X-RAYS ON” lamp at the top of the hutch is part of a warning and interlock system that are required to be operational in order to produce X-rays. See Figure 6.



Fig. 6. Warning lights on top of the hutch.

(c) A yellow LED next to tube housing's shutter assembly illuminates when the shutter is open.

(d) Shutter open LCD indicator on front panel to indicate to the user when the shutter is open

2. Other signs.

(a) A sign is posted on the diffractometer hutch bearing the conventional yellow and magenta radiation symbol and the words: "CAUTION: RADIATION. THIS EQUIPMENT PRODUCES RADIATION WHEN ENERGIZED. TO BE OPERATED ONLY BY QUALIFIED PERSONNEL".

(b) Entrances to Chauvenet Hall CH-044 are posted with yellow and magenta signs bearing the words "CAUTION: RADIATION. THIS EQUIPMENT PRODUCES RADIATION WHEN ENERGIZED".

Interlocks:

There are two basic safety loops in X'Pert PRO MPD systems. Both of these loops must indicate a "safe" status before the system will allow High Tension to be applied to the X-ray tube.

The Upper part of the instrument enclosure is the X-ray enclosure. All access panels and doors to this enclosure, make contact with microswitches when they are closed. These microswitches are a series safety loop and microswitches are doubled, forming a second independent safety system. A yellow LED next to the tube housing's shutter assembly, illuminates when the shutter is open. If the safety circuit is broken during operation, a relay on the shutter assembly will automatically close the shutter.

The HT safety loop protects the system by automatically closing the shutter on the tube housing and shutting down or preventing the application of HT if the circuit is not completed.

Interlocks on PANalytical X'pert PW-3040 PRO MPD Series X-ray Diffractometer include:

- (1) Front Glass Sliding Doors
- (2) Rear Hinged Doors
- (3) "X-rays On" Warning Lamp
- (4) Shutter Housing Assembly LED
- (5) Tube Housing Assembly/ PreFix incident beam optical module
- (6) High-Tension Cable
- (7) High-Tension Key
- (8) Water Flow Rate

GENERAL OPERATING INSTRUCTIONS PANalytical PW-3040-Pro Series X-ray Diffractometer

These procedures represent a basic outline for safely operating the diffractometer. The operating instructions below provide general overview of the procedures for starting and stopping the diffractometer, but do not serve as a substitute for understanding the equipment. More detailed data collection procedures can be found in the PANalytical Pro Users Guide Safety Manual, Reference (a). Only qualified operators / authorized users are allowed to operate the diffractometer or other personnel under direct supervision of an authorized user.

As Authorized User you are completely responsible for this instrument being used in a safe manner. If you leave CH-044 during a scan you must screen lock the computer (select 'Switch User' to do so) and lock the room door.

1. Obtain the key to the diffractometer from the storage location.
2. Record the operator's name, starting time, date, sample name, and verify all checks in the Usage Log. Enclosure (8) contains a copy of the usage log sheet.
3. Personnel Thermoluminescent Dosimetry devices are not required. A radiation survey of the diffractometer, is only required to be performed every 6 months, after service, and whenever an issue with the beam alignment is suspected.
4. Verify that the unit is in "Stand by." If the "Stand by" indicator lamp is lit up on the control and display panel then electricity is being supplied to the diffractometer. Skip to number 6.
5. If the "Stand by" indicator lamp is not on, contact the Laboratory Manager or Authorized User. (There are three power breakers in place that need to be checked).
6. Turn the water chiller switch to the "ON" position located on the front of the water chiller unit.
7. Open the diffractometer unit's sliding glass doors and position the sample to be investigated with the diffractometer.
8. Close the unit's sliding glass doors.
9. Insert the High-Tension key into the unit's High-Tension key slot, turn to the "ON" position.
10. Press the "Power On" button on the front control panel.
 - a. If everything works properly
 - (1) The HT indicator lamp should light up on the display panel.
 - (2) The Standby indicator lamp will turn off.
 - (3) The Power On indicator lamp will turn on.
 - (4) The system will start an initialization procedure.

- (5) The HT generator will turn on and ramp up to 30 kV and 10 mA.
 - (6) The X-ray On lamp on top of the enclosure will light.
 - (7) The tube and detector will be moved to their starting positions.
 - (8) The diffractometer is now ready.
- b. If the above actions do not happen, look at the front display panel > Shutter Open
- (1) If there are three dots in the Shutter Open LED section, then the enclosure doors are not properly closed or the HT key is not turned.
 - (2) If the doors and the HT key are ok, and the system did not start up normally you will need to contact Laboratory Manager or Authorized User Technician.
11. On the computer, open software (Data Collector) and enter User (name) and Password.
12. In Data Collector, select *Instrument* tab > *Connect*.
- a. Select the sample stage you are using: PW3071/xx Bracket or Spinner PW3064.
 - b. Click “OK” for the (yellow) configuration message.
 - c. Click “yes” for fine calibration message.
13. If the diffractometer has been off for at least four days, it is advised to start the fast tube breeding procedure. If the diffractometer has been turned off for one month or longer, it is advised to start the normal tube breeding procedure.
14. In Data Collector, Click on the Instrument Settings tab.
- a. In the Hierarchal tree, double-click on any of the blue settings on the Generator branch.
 - (1) A new Instrument Settings window will pop up that will allow you to change all setting in that category.
 - b. In the new window that opens, click on the X-ray tab.
 - (1) If you need to breed the tube, click on the “Breed” button.
 - (a) Select fast or normal.
 - (2) In small increments increase the kV and mA to the desired level. Change voltage first then current. Click apply after each increment change.
 - (a) Normal power levels: (Tension = 35 kV, Current = 30 mA) 35% power
(Tension = 45 kV, Current = 40 mA) 60% power
(Tension = 50 kV, Current = 45 mA) 75% power

- (b) Full power level: (Tension = 60 kV, Current = 50 mA) 100% power
- (3) Click on the “Shutter Open” box and click “Apply.”
 - (a) The door will lock and the shutter will open.
- 15. In Data Collector, click on the “Shutter Open” box and click "Apply".
 - a. The shutter will close and the door will un-lock.
 - b. Click ok and the Instrument Settings pop-up window goes away.
- 16. In Data Collector, Click on the “Incident Beam Optics” tab.
 - a. In the Hierarchal tree, double-click on any of the blue setting on the Incident beam path branch
 - (1) A new Incident Beam Optics window will pop up.
 - (2) Compare what is selected on the software to what is in the machine in the incident beam path.
 - (3) If you change any of the incident beam optics on the machine, change it in the software.
 - (4) Click “Apply” to set any changes and then click “ok” to close the pop-up window.
- 17. In Data Collector, Click on the “Diffracted Beam Optics” tab.
 - a. In the Hierarchal tree, double-click on any of the blue setting on the Diffracted beam path branch.
 - (1) A new Diffracted Beam Optics window will pop up.
 - (2) Compare what is selected on the software to what is in the machine in the diffracted beam path.
 - (3) If you change any of the diffracted beam optics on the machine, change it in the software.
 - (4) Click apply to set any changes and then click ok to close the pop-up window.
- 18. It is advised to switch the system on with the generator operating at 75% of the maximum tube rating for between four to eight hours before you start to perform measurements. This will ensure that the temperature and various components have stabilized.
- 19. Carry out the experiment using Data Collector.
 - a. Create new program or use existing program.
 - b. Use reference (a) for a more detailed setup procedures.
- 20. If another experiment is to be performed, carry out steps 8 and 9 and then perform step 16.

21. When the experiment is complete:
 - a. Remove the sample from the chamber.
 - b. In Data Collector, Click on the “Instrument Settings” tab.
 - (1) In the Hierarchal tree, double-click on any of the blue settings on the Generator branch.
 - (a) A new Instrument Settings window will pop up.
 - (2) In the new window that opens, click on the “X-ray” tab.
 - (3) In small increments decrease the mA and kV to the desired level. (Current then voltage)
 - (a) Shut down power level: (Current = 5 mA, Tension = 15 kV)
 - (4) Unclick “Generator On” to switch the generator off.
 - (a) The X-RAY ON lamp on top of the enclosure will go out.
 - (5) Click “ok” to close the window.
 - c. Disconnect the machine in Data Collector, under the “Instrument” tab.
 - d. Close software to log off.
 - e. Turn the HT key to off and remove the key from the diffractometer.
 - f. Push the “Stand by” button on the machine.
 - (1) The Power On indicator lamp will turn off.
 - (2) The Stand by indicator lamp will turn on.
22. Return the HT key to the storage location.
23. Record the ending time and date of the experiment in the Usage Log.
24. Wait at least 5 minutes before turning off the cooling water.
25. Upon leaving the lab, ensure that the lab doors are locked.
26. To avoid equipment, damage due to power cycling the tube, the diffractometer may remain energized during the week provided that unauthorized equipment use is prevented by both.
 - (1) password protection on the computer control system and (2) locking the doors to the lab.
 - a. Notify the Laboratory Manager.
 - b. Turn the power to the idle mode of (40 kV and 10 mA.) 13.3% power level
 - c. In Data Collector software log off and close program.

- d. Close out the software and select to close software and leave the machine on.
- e. Record the ending time and date in the Usage Log.
- f. Record in the Usage Log that the machine has been left on in the idle mode, time, and date.
- g. Lock the door to the lab on the way out.

Maintenance Procedures

The PANalytical requires regular maintenance to maintain it in optimal condition. General maintenance items are provided in the following table and are performed by Authorized Users.

General Maintenance Items	
<i>Every two months</i>	
Prefix surface	
<i>Every Six Months</i>	
x-ray tube filters and o-rings	
Main cooling water filters	
Enclosure dust fan filter	
Water: PH, conductivity, algae growth	
Goniometer lubrication	
<i>As Needed</i>	
Spinner changer	
Cleaning enclosure windows	
Removing excess dust	
High-Tension connection contacts	

Other maintenance and repair procedures impacting the production of x-rays will be performed by an Authorized User or PANalytical personnel under the direction of the laboratory manager after obtaining the consent of the RSO. Training of Authorized Users will be done by the Laboratory Manager or by PANalytical personnel.

Procedures impacting the radiation production and safety features of the PANalytical are:

1. repairs and maintenance of automatic shutters
2. repairs and maintenance of interlock switches and circuitry
3. beam alignments.

A radiation survey must be performed after any alignment procedure has been performed.

EMERGENCY PROCEDURES

In the event of an emergency requiring a quick shutdown to minimize risk of human injury, the following procedures shall be followed.

1. Shut down the instrument, including the X-ray generator, by pushing the “Stand by” button on the display panel.
2. For a complete emergency hard shut down, PULL the safety switch lever DOWN that is on the pillar next to the door (LPOB9 BKR 21 & 23).
3. Leave the X-ray facility, shutting and locking the door behind you.
4. Notify the X-ray Diffraction Lab Manager and RSO.

Notify other emergency contacts, such as fire or police departments, if appropriate.

RADIATION MONITORING

Personnel Radiation Monitoring Equipment:

Operators of the PANalytical X'Pert PW-3040-Pro MPD series X-ray diffractometer are Non-Radiation workers and do not require personnel dosimetry.

Equipment Radiation Monitoring Equipment:

1. USNA radiation safety policy requires a radiation survey to be performed semi-annually. This is stricter than the RAD-010 13.1.4.2.a. minimum requirement of once every 12 months. (Semi-annual means to perform once in the first 6 months of the calendar year and once in the second 6 months of the calendar year.).
2. In addition, a radiations survey and an inspection and test of protective devices (i.e. interlocks) must be performed according to RAD-010 13.1.4.2:
 - a. Semiannually (USNA policy)
 - b. Immediately after any changes that could adversely affect radiation protection (e.g. maintenance or repair, shock, earthquake, relocation of equipment, radiation shielding modifications, etc). The RSO may review and accept the post maintenance survey conducted by the vendor.
 - c. Following any maintenance requiring the disassembly or removal of a local component in the system.
 - d. During maintenance and alignment if the procedures require the presence of a primary x-ray beam when any local component in the system is disassembled or removed.
 - e. Anytime a visual inspection of the local components in the system reveals an abnormal condition.
 - f. Whenever recommended by the manufacturer.
3. Surveys will be performed with an Ion Chamber Survey Meter (RADIAC), suitable for X-ray radiation. Only RASO approved meters shall be used to measure radiation levels needed to satisfy legal and health related documentation requirements.
4. RADIAC's must be in calibration and source-checked prior to use.
5. The survey will be performed and documented by an Authorized User, and reviewed by the RSO or ARSO.
6. Full requirements and the sheet used for surveys is found in Enclosure (8).

Permanent Area Radiation Monitors:

A TLD may be placed close to the radiation hutch to serve as an area monitor. There is no requirement for having area radiation monitors under the Naval Nuclear Materials Permit Broad Scope License. Having a thermoluminescent dosimeter placed close to the radiation hutch provides long term documentation useful for evaluating annual exposure rates.

OPERATOR QUALIFICATION REQUIREMENTS

A qualification program shall include, at a minimum:

- a. Radiation health requirements in accordance with Reference (c).
- b. Radiation safety requirements in accordance with References (a), (b), and (d).
- c. Knowledge of operating procedures in accordance with Reference (a).
- d. A full understanding of this instruction, with the instruction reviewed annually and documented on the Machine Log sheet.
- e. Operators must be Authorized Users as approved by the branch manager (Physics department chair), division manager (Dean of Mathematics and Science), USNA Radiation Safety Committee, and Radiation Safety Officer.

A qualified operator shall be trained and certified by the X-ray Diffraction Laboratory Manager. The list of qualified operators shall be maintained by the X-ray Diffraction Laboratory Manager using the sheet in this instruction. Any qualified operator, who does not operate the instrument within the span of one year, must be requalified. Midshipmen shall not routinely be qualified as operators. Only under the circumstance in which a midshipman requires use of the instrument on a continuous and recurring basis shall he or she be certified as a qualified operator. Qualification sheets are found in Enclosure (8).

RECORDS – SHEETS and LOGS

Usage Log

1. Each use of the diffractometer shall be documented by the Operator in the Usage Log maintained for that purpose at the diffractometer control console. Data recorded shall include the following items.
 - a. The instrument name and location of the lab.
 - b. Laboratory Manager's name and office phone.
 - c. User name and signature.
 - d. Date/time on and Date/time off.
 - e. Estimate of Exposure time.
 - f. Machine settings (operating parameters: kV and mA).
 - g. Experiment name or sample type under Remarks.
 - h. Verification that the interlock check is current.
 - i. Verification that radiation surveys are current.
 - j. Verification of operator annual review of SOP.
2. Verification of operator annual review of SOP.
3. The Usage Log sheet is shown on page 2 of Enclosure (8).

Radiation Survey Log:

1. A complete radiation survey, with the diffractometer running under typical conditions, shall be conducted every six months or whenever maintenance has been performed which might result in an increase in radiation levels. Data recorded shall include the following.
 - a. The date and time of the survey.
 - b. The name of the surveyor.
 - c. Date of last survey and interlock check.
 - d. Verification of the interlock check.
 - e. Survey meter used including: Model #, Serial #, Calibration and Due date.
 - f. System parameters including: Anode, kVp and mA.
 - g. Diagram showing survey points.
 - h. Readings (mR/hr) at the survey points.
 - i. RSO or ARSO signature.
2. This radiation survey satisfies the USNA requirement for semi-annual radiation protection surveys and interlock checks. (Semi-annual means to perform once in the first 6 months of the calendar year and once in the second 6 months of the calendar year.)
3. The sheet for the radiation survey is shown on page 3 of Enclosure (8).

Operator Qualification Requirements: This sheet is found on page 4 of Enclosure (8).

Lists of Qualified Operators: This sheet is found on page 5 of Enclosure (8).

PANalytical PW-3040-PRO SERIES X-RAY DIFFRACTOMETER SN 201866

Machine Usage Log

Laboratory Manager & Office Phone: _____ Laboratory: Chauvenet-044

User and AU Name & Signature	Date/time on	Date/time off	Estimate Exposure Time	Machine Settings (kV, mA, etc)	Target Material & Other Remarks	Verified 6- mth interlock check within periodicity (initial)	Verified 6- mth radiation check survey within periodicity (initial)	Verified Users are current in training & reviewing SOP (initial)

RSO Review: _____

RADIATION SURVEY
PANalytical PW-3040-PRO SERIES X-RAY DIFFRACTOMETER
S/N 201866, Chauvenet-044
(Semi-annual means to perform once in the first 6 months of the calendar year
and once in the second 6 months of the calendar year.)

Date _____ Time _____ Performed by _____

Date of last: Facility Review, Re-Authorization & SOP Update (5 yrs) _____
Radiation Survey (semiannual) _____
Interlock Check (semiannual) _____

Interlock verified (initials) _____

Survey Meter

Model # _____
Serial # _____
Calibration Date _____
Due Date _____

System Parameters

Anode _____
Focus _____
Tube# _____
kV _____
mA _____

Survey points are 5 cm from the enclosure. Results must be less than 0.25 mR/hr.

	_____ mR/hr	_____ mR/hr	_____ mR/hr			_____ mR/hr	_____ mR/hr	
	x	x	x					
_____ mR/hr	x	RADIATION ENCLOSURE				x	_____ mR/hr	
_____ mR/hr	x					x	_____ mR/hr	
_____ mR/hr	x					x	_____ mR/hr	
	x	x	x					
	_____ mR/hr	_____ mR/hr	_____ mR/hr					

Computer Console: _____ mR/hr

RSO Review: _____

OPERATOR QUALIFICATION REQUIREMENTS
PANALYTICAL PW-3040-PRO SERIES X-RAY DIFFRACTOMETER

Name: _____

1. Familiar with radiation health requirements of NAVMED P-5055 and RAD-010.

Laboratory Manager/Date

2. Trained by the Laboratory Manager on the identification of radiation safety hazards, warning systems and safety precautions associated with the instrument.

Laboratory Manager/Date

3. Understands the guidelines in this SOP and safely operates the PANalytical PW-3040-PRO Series X-Ray Diffractometer.

Laboratory Manager/Date

4. Qualified.

Laboratory Manager/Date

5. Branch Manager Certification: Physics Department Chair.

Department Chair/Date

6. Division Manager Certification: Dean, School of Math and Science.

Dean Math and Sci/Date

7. Designated Authorized User by USNA Radiation Safety Committee.

RSC/Date

8. RSO Review.

RSO/Date

QUALIFIED OPERATORS
PANALYTICAL PW-3040-PRO SERIES X-RAY DIFFRACTOMETER
S/N 201866, Chauvenet-044

Name	Date Qualified & SOP Review	Laboratory Manager Signature

RSO review: _____

Non-Radiation Worker Training Documentation

DATE(S): _____ LENGTH OF TIME: _____

INSTRUCTOR(S): _____ RSO APPROVAL: _____

GROUP TRAINED: _____

2.8.6.6 Training may be provided as lectures, demonstrations, video, or self-study and should emphasize the use of site-specific RASP radiation sources.

MATERIALS USED (i.e. Filenames, Storage Location, etc): _____

The NAVSEA04 document RAD-010 provides definitions and limits: 2.8.6 “Non-radiation Workers require access to posted radiation areas to perform specific functions and receive ionizing radiation exposure incidental to performing normal work tasks versus handling of radioactive material or using radiation producing machines. Emergency personnel, firefighters, medical, and security personnel are considered non-radiation workers.” 2.3.3 “Radioactive material and/or other sources of ionizing radiation exposure shall not be used in such a manner as to cause any non-radiation worker, as defined in Section 2.8.6, to exceed a TEDE of 500 millirem (mrem) (5 millisievert (mSv)) per year considering occupancy factors and source usage.”

Training Topics for Non-Radiation Workers are specified in the document RAD-010 2.8.6.3

Instructor Verification (pls initial)	Topic
	a. Sources of radiation in areas they may frequent.
	b. Potential hazards associated with radiation sources in areas they may enter.
	c. Biological effects and risks associated with exposure to ionizing radiation from internal or external sources.
	d. Use and meaning of radiation warning signs and barriers.
	e. Procedures to avoid and reduce radiation exposures
	f. Personnel dosimetry, bioassay and/or internal monitoring requirements
	g. Biological effects of exposure to the embryo/fetus if the person will receive greater than 100 mrem per year.
	h. Each individual is responsible for tracking his/her own radiation exposure and exposure reports that are provided by the command.
	<i>Additional Site-specific topics:</i>
	<i>for Emergency Response Personnel only:</i> emergency exposure limits and the importance of medical treatment over contamination concerns.

LIST OF ATTENDEES (legible, continue on reverse side)

