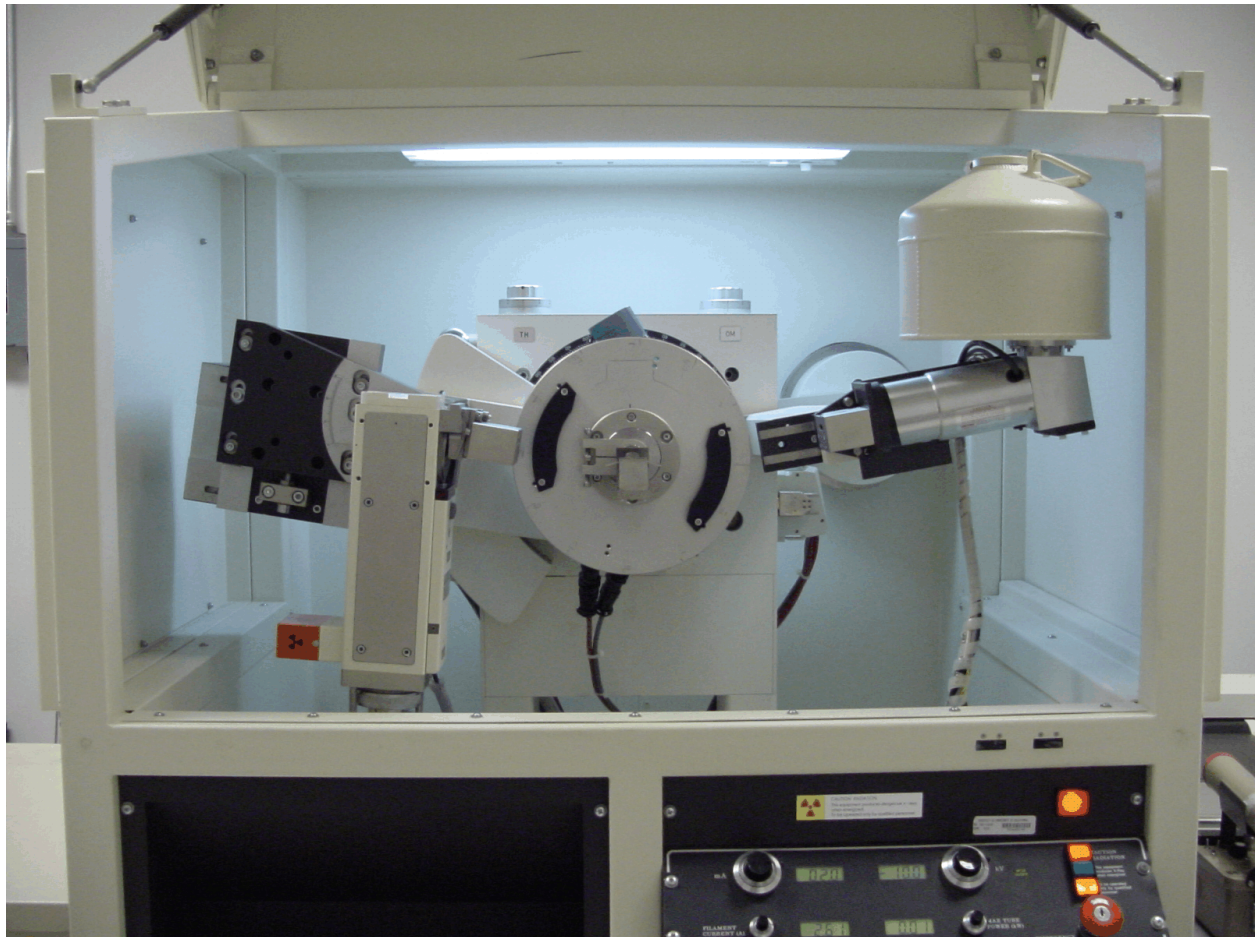


# User's Manual

Scintag XDS 2000 X-Ray Diffractometer

*PCDMS Version*



Materials Science Central Facilities  
Department of Chemical Engineering and Materials Science  
University of California, Davis

December 31, 2001



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Mike Meier

Materials Science Central Facilities  
Department of Chemical Engineering and Materials Science  
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### Cover

The cover image shows three common crystal structures and their associated diffraction patterns. From top to bottom the structures are: aluminum oxide (corundum), silicon (diamond cubic), and sodium chloride (halite). These images were generated using CaRIne Crystallography.

### Revision History

September 1996	Original version
September 24, 1997	Updated and revised for use in EMS-132L
September 1998	Minor revisions
May 17, 2000	Updated to include changes in recharge policy
September 25, 2000	Minor correction in "Standard Settings" chapter
December 31, 2001	Minor corrections



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# Part 1. Introduction

## Welcome

The Division of Materials Science is pleased to offer its students and researchers at U.C. Davis a state-of-the-art x-ray diffractometer. It is one of the most in demand instruments in Central Facilities and in its six years in operation it has been used by over 100 different researchers. In addition, it is an integral part of two undergraduate materials science laboratory courses and is available for any other course taught at UC Davis.

Our x-ray diffractometer features a  $\theta/2\theta$  goniometer, copper and chromium x-ray sources, a solid state germanium detector and easy-to-use software, a thin-film attachment, a high-temperature chamber and the JCPDS database on CDROM. While primarily used for routine phase identification a number of users have also used it to measure crystallite size (Sherrer, Warren-Averbach), percent crystallinity, retained austenite and residual stresses and even to do some preliminary work on measuring the stress in thin films.

## Responsible Persons

The principle investigator who purchased, licensed and installed the instrument and who is ultimately responsible for it and how it is used is:

Professor Shackelford  
752-0566  
jfshakelford@ucdavis.edu

The manager, responsible for the day-to-day operation, maintenance, training and scheduling use of this instrument, is:

Mike Meier  
752-5166  
mlmeier@ucdavis.edu

If you have any questions about this instrument, this manual or would like to use the diffractometer please contact either Professor Shackelford or Mike Meier.

## Training

Anyone who wishes to use the x-ray diffractometer must first be properly trained in x-ray safety, proper operation of the diffractometer, and general workplace safety for people working in the x-ray diffraction laboratory. This is a four-step process:

1. Take the x-ray safety training course which is offered by Environmental Health and Safety.
2. Amend the license so that your name is formally added to the list of authorized users of this specific x-ray producing machine. At this point you will receive your personal dosimetry.
3. Check and if necessary update your training on workplace safety and laboratory safety issues, including cryogenics safety. Some of this will be reviewed during your training session. The cryogenics safety course can be taken online at [www.matsci.ucdavis.edu/olst](http://www.matsci.ucdavis.edu/olst).

4. Get training covering the instrument's configuration, basic operation, and additional training on the safety features and procedures of this instrument.

Only when this training is complete you can schedule time on the instrument.

### **Reservations**

Authorized users can reserve time on the diffractometer by contacting the manager via phone, e-mail or in person. The preferred method is email, but in the near future we will have an on-line calendar which users can use to request time on the instrument.

### **Usage Rates**

This instrument is available all year round: days, nights and weekends. During the summer months it is booked 100+ percent of the normal working week so making reservations at least 3 days in advance may be necessary. During the academic year making reservations in advance is not normally necessary since the instrument is in use only 30-50 percent of the time.

### **Recharge Policy**

In early 1999 we established a recharge mechanism to help the department recover the cost of operating the x-ray diffractometer and supporting its users. As many people know this diffractometer has been in operation since late 1993 and during this time it has been very busy. It has become such an essential part of many of our research and teaching programs that one wonders how we every got along without one. These initial years have also provided us with time to learn more about x-ray diffraction and to establish a pattern of use which allowed us to assess the feasibility of establishing a recharge, something that was talked about when the instrument first arrived.

The recharge rates have been established in accordance with all of the applicable campus Policy and Procedures and have been approved by the Office of Planning and Budget. Four rates have been established. They are:

- |                                  |            |   |
|----------------------------------|------------|---|
| 1. Campus User Rate              | \$15/hour  | UCD student or researcher operates the diffractometer                             |
| 2. Campus Service Rate           | \$60/hour  | CHMS staff performs the experiments and analyses for UCD students and researchers |
| 3. Off-Campus Institutional Rate | \$100/hour | Other campus's and state agencies. Staff will operate the diffractometer          |
| 4. Off-Campus Commercial Rate    | \$150/hour | Analyses performed for private persons or companies, when appropriate             |

(For comparison, commercial rates in the Bay Area range from \$200 to \$300 per hour.)

In case you are not familiar with campus recharge policies, there are two issues which pretty much sum up the purpose and the practice of recharging. First, there are a number of restrictions on which costs we can recover. For instance, the costs of upgrades, staff time for billing and accounting, phones and office supplies, any cost already included in the overhead taken from Federal grants is not allowed. Second, recharge rates are set so that we do not make a profit or take a loss. Our books

will be reviewed regularly and the rates adjusted as needed. Clearly, recharging is a cost recovery mechanism, not a money making mechanism. Any costs recovered will free up a portion of the departmental operating budget for other projects.

### **This Manual**

With over 40 researchers users at any one time, increasingly demanding safety policies and over 30 additional students using this instrument in the course of their undergraduate studies this manual has become a vital part of our training sessions and each researcher's operation of the diffractometer. It includes written safety and training procedures, basic operating procedures, instrument specifications and configuration details plus other articles the user should find useful.

Each authorized user will be given a copy of this manual at the time of their training. A copy will also be available in the laboratory. Additional copies can be obtained by asking the instrument's manager.



# Part 2. Training

## Obtaining Authorization to Operate the Diffractometer

This article describes briefly the mechanism by which radiation-producing machines on this campus are licensed and how an individual can become an authorized user of these machines.

### The Regulatory Chain

The regulatory chain governing the use of ionizing radiation begins with the Nuclear Regulatory Commission, extends down through the state government and to the U.C. Davis campus which has a Broadscope license. The Health Physics Section of Environmental Health and Safety administers this license, ensuring that all radiation-producing machines, uses of radioisotopes and authorized personnel are in compliance with campus, state and federal regulations. Regarding radiation producing machines, every installation on campus which uses machines which produce ionizing radiation is issued a Machine Use Authorization (MUA). This is issued at the request of the principal investigator and specifies the type and amount of radiation and the names of all personnel who are authorized to use the equipment.

### Obtaining Campus Authorization

To become an authorized user of any radiation-producing machine on the UCD campus or at the UCD Medical Center you must:

1. Take EH&S's analytical x-ray safety course. If you have taken this course in the past you can opt to take a written open book test instead.
2. Submit a "Statement of Experience" form to EH&S.
3. Amend the MUA so that you become an authorized user. This involves filling out a simple form which must be signed by the principal investigator who is named on the MUA.

Slightly different rules apply to the following three situations:

1. Persons who are authorized users at other MUAs on this campus  
Previous EH&S safety training and the "Statement of Experience" form are valid and the personal dosimetry which has already been issued can be used. The MUA of the new instrument, however, must be amended.
2. Persons who have significant experience using similar types of equipment at off-campus sites  
There is a 30 day grace period before having to take the safety course or the exam. You must, however, submit a "Statement of Experience" form to EH&S, take their online exam, and amend the MUA before you begin using the equipment.
3. Visitors, including any person from off campus  
Visitors may accompany an authorized user who will operate the equipment. The visitor may not operate the equipment. Visitors who wish to operate the equipment must first become authorized users following the procedure described above.

#### 4. Service Technicians

Service repair people can operate the diffractometer while performing calibrations or repairs. They are expected to have their own dosimetry and to follow our safety and operational rules.

After becoming authorized to use any one radiation-producing machine EH&S will issue your personal dosimetry. At this point you must receive additional training on the operation of the specific machines you will use. This training is given by the P.I. or manager of that machine and not by Environmental Health and Safety.

### **Obtaining Instrument Authorization**

After you are on the license to operate this diffractometer and have received your dosimetry you must do two other things:

1. Submit a "Permission to Recharge" form which specifies accounts to recharge and is signed by principle investigator.
2. Provide copies of your safety training records, including general workplace safety, evacuation procedures, and cryogenics safety. If you will be working with isotopes, carcinogens, tissues or other regulated materials you must also provide copies of these training records.
3. Schedule training on this diffractometer. Training is done on a one-on-one basis and requires 2 to 4 hours depending on the user's experience. During this training the user will receive instruction on all aspects of working in the diffractometer laboratory, operation of the diffractometer itself, and the basics of data collection and analysis.

At the conclusion of this training you will be asked to sign a form acknowledging receipt of this site-specific training.

### **Other Details**

The following are a few additional notes regarding the licensing and authorization process which you might find useful:

1. Users of electron microscopes are not required to become authorized users in the manner described above. It is the responsibility of the principal investigator to provide all training and to keep a log as required in the Illness and Injury Prevention Program.
2. Analytical x-ray classes are offered approximately every 60 days. Telephone Environmental Health and Safety to reserve a spot in the next "Analytical X-Ray Safety Course".
3. Personal dosimetry for students, staff and faculty of this campus is free of charge while there is a small charge for dosimetry which is issued to visitors from off-campus. Dosimetry is exchanged and evaluated quarterly. You will be billed if you lose your dosimetry. Also, you will receive a copy of the results, which by the way, are part of your medical records.
4. You must renew your training with EH&S every three years by retaking the analytical x-ray safety class or by taking and passing a written exam. You may also be asked to take refresher

course for each of the instruments you use.



## **Training Procedure for Diffractometer Operators**

This training procedure summarizes the training a person receives from the manager of Materials Science's X-Ray Analysis Laboratory. This training is given to all users of x-ray producing equipment in this laboratory, regardless of past experience with similar or even identical equipment on or off the UC Davis campus. In addition, no more than three persons will receive training in a given session, although additional persons, present only as observers, may be present.

### **Verify Campus Authorization to Use This Equipment**

Training will not be given to persons who have not already been authorized by EH&S to use the equipment he/she is receiving training on. Ideally the instructor will already have copies of the required forms which have been processed by EH&S. If this is not the case and the trainee states that he/she is authorized to use this equipment then training may proceed with the understanding that this will be verified with EH&S. In either case, make sure that the trainee understands the authorization process by doing the following:

1. Ask if the trainee has taken EH&S's Analytical X-Ray Safety Course recently.
2. Ask if the trainee has submitted a statement of experience form to EH&S.
3. Ask if the trainee has submitted an "Amendment of RUA" form to EH&S which adds them to the list of users for this equipment.
4. Verify that personal hand and full body dosimetry has been issued and is in the possession of the trainee. If not, then the training session cannot continue.
5. Verify that all other safety training requirements have been met.

### **Additional Training on this Diffractometer**

The trainee must complete the following training to the satisfaction of the instructor and the equipment's manager. The following topics are covered in this training:

1. Administration
  - a. The department and P.I. who is ultimately responsible for this equipment.
  - b. The manager who is responsible for day-to-day operation, training, maintenance and scheduling the use of this equipment.
  - c. General policy on use of this equipment, including issues such as consumable supplies, recharges, conduct and how, when and where to report problems of any nature.
2. Safety
  - a. The safety "environment" on campus and in this laboratory. How there are two sides to this, one being regulatory in nature, designed to prevent injury by forbidding unsafe practices, and the other more service oriented where training, information and oversight help create and maintain a safe working environment and are also available to assist in any safety-related problem.
  - b. The people and organizations to contact for safety-related problems. Emphasize that the user not hesitate to ask any question or express any concern regarding personal safety and the safety of the equipment.

- c. The safety features of the equipment.
- d. The importance of following established procedures when using the equipment.
- 3. Using a Hand Held Radiation Survey Meter
  - a. Review the basic specifications and capabilities of the hand held radiation survey meter which the trainee will be using when operating the equipment.
  - b. Show the trainee how to turn on the meter, how to make sure it is working properly, and how they should configure it when using it to check the instrument for leaks.
  - c. Show the trainee how to use the radiation survey meter to check the instrument for radiation leaks.
- 4. Introduction to the Equipment
  - a. The nature and function of the equipment.
  - b. The basic configuration of the equipment, identifying all sub-systems and describing what each does and how they are interconnected.
- 5. Operating Procedure
  - a. Using the written start-up procedure in the Safety Protocol for this equipment, go through the startup, sample change and shutdown procedures with the trainee. At each step in these procedures describe the reason why things are done in a particular way, the consequences of operator errors and how to recover from them, and practical advice which will help the trainee be successful in future work.
  - b. All through this session the trainee must operate all of the controls.
  - c. Show the trainee the features of the equipment and resources in the laboratory which the trainee can use to solve most of the types of problems that he/she is likely to encounter.
- 6. Emergency Procedures
  - a. Review the emergency procedures.
  - b. Point out the location of the notice which lists the telephone numbers of the safety personnel responsible for the laboratory.
  - c. Show the trainee the phone, the fire extinguishers and the first aid kits. Review the policies and procedures concerning this equipment.
  - d. Show the trainee the “Chemical Hygiene Plan” for the laboratory, pointing out the sections on laboratory hazards and emergency procedures.
- 7. Security
  - a. Explain how to properly secure the equipment and the laboratory at the end of a working session.
  - b. Emphasize the importance of controlling access to the instrument.
- 8. Policy on Visitors
  - a. Explain the policy regarding visitors.

### **Completion of Training**

Training will be considered complete when the trainee can demonstrate reasonable knowledge of the administrative, safety and security issues and can independently and confidently operate the equipment. At this point the trainee fills out a registration form which the equipment’s manager will keep in his/her files. This registration form identifies the trainee, the trainee’s supervisor and documents verification of receipt of EH&S training and training on a particular instrument.

# Part 3. Safety

## Safety in the Central Facilities Laboratories

All of the laboratories in Central Facilities are in compliance with UC Davis's Illness and Injury Prevention Program. This program requires that each individual laboratory establish its own comprehensive safety program. These programs address issues such as: responsible persons, methods of purchasing, using and storing hazardous chemicals, working with radiation, biological agents, carcinogens and hazardous machines, training and records of this training and hazards communications. The safety program for each of central facilities' laboratories are described in detail in the red "Chemical Hygiene Plan" binders which can be found in each laboratory. An overview of these plans covering all of central facilities is summarized below.

### Responsible Persons

Just as the P.I. is responsible for everything that happens in his/her laboratories the manager of central facilities must answer for everything which happens in central facility's laboratories. This person must not only provide a safe work place but make sure that the work is done in a safe manner. In addition, everyone who works in a laboratory on this campus is considered to be a safety officer in the laboratories they work in. They have an obligation to do their part to make and keep this campus a safe working environment. This would encompass not only one's own actions but also speaking up or taking the appropriate action when a dangerous or potentially dangerous situation presents itself. One's responsibilities regarding one's own actions can be summed up in this department's basic rule: "If you use it you are responsible for it."

### Hazards Communications

All hazards present in each laboratory are listed in each laboratory's red "Chemical Hygiene Plan" binder. In addition, signs are posted in the laboratories and on or close to equipment where hazards exist. Finally, departmental and campus resources exist where a person can go to learn more about the hazards they encounter in the workplace. Refer to the "Chemical Hygiene Plan" for more information on these resources.

### Hazardous Chemicals in the Laboratories

**Use:** Proper safe working practices should be followed at all times. Care should be taken to protect yourself, your colleagues, the equipment in the laboratories and the building, even if this means making an extra effort to set up an experiment, to carry it out and to clean up afterwards.

**Labeling:** All chemicals brought into these laboratories must be properly labeled and must also bear the owner's name and the date it was purchased.

**Storage:** A selection of common chemicals will be kept on hand in these laboratories and everyone is free to use them but not to remove them from the laboratory. Any chemicals brought into central facilities or mixed in this laboratory must be removed as soon as the work of the day is completed. Users cannot store their specimens, chemicals or chemical waste in these laboratories.

## **Personal Protective Equipment**

Each laboratory is equipped with the types of personal protective equipment which is appropriate for the work normally done in that laboratory. In general, this consists of one or two aprons, pairs of chemical resistance and/or heat resistant gloves, goggles and face shields. Users are encouraged to supply their own personal protective equipment and larger groups of people (classes) will certainly have to provide their own.

## **Training**

Everyone who works in these laboratories is required to attend the Department of Chemical Engineering and Materials Science's annual safety seminar or its equivalent if one is from another department. In addition, participation in a safety orientation for the central laboratories is required. Records of this training and any other specialized training for use of equipment in these laboratories will be kept on file in the red "Chemical Hygiene" binders.

## **Enforcement of the Safety Rules**

Enforcement is only necessary when someone persists in working in a manner which is hazardous to either themselves, those around them, the equipment in the laboratory and even the building. Rarely is enforcement necessary but when it is it may take the form of a reprimand, temporary suspension or even termination of employment. In addition, departmental safety officers have the authority to close any laboratory where serious hazards exist.

Unsafe activities in Materials Science's Central Laboratories can not be tolerated. The liabilities and the costs of repairing the facilities are simply too great, not to mention the horror of seeing our colleagues injured. If gentle reminders or additional training are not sufficient to end the unsafe practice then the offending person will be temporarily banned from the laboratory. Flagrant or persistent disregard for the safety of the laboratory and persons in it will result in permanent banishment from all of the central laboratories.

# Operating Procedure (Safety Protocol) for the Scintag X-Ray Diffractometer

This safety protocol describes briefly but in detail how to prepare the diffractometer for use and how to shut it down. Any person who is at all familiar with this type of equipment should be able to read this and successfully turn the system on and off. This protocol, however, is meant to be used as a quick reference for trained, qualified users and for emergency situations where such a person is not present. It was not intended for use as operating instructions. Proper operating procedures can only be learned by having received training from either the principle investigator (P.I.) or the laboratory's manager.

## Prerequisites

Do **NOT** operate this equipment unless you have satisfied all of the following prerequisites:

1. You have the express permission of Materials Science Central Facilities staff.
2. You have satisfied all of the requirements established by Environmental Health and Safety (EH&S) for being an authorized user of this x-ray diffractometer.
3. You have satisfied all other workplace and laboratory safety training requirements.
4. You have received the additional training on the use of this particular instrument and have signed off on this training.
5. You know how to properly use the hand held radiation survey meter.

## Start-Up Procedure

The following is the basic procedure for turning on the Scintag x-ray diffractometer. It assumes that the instrument is initially in its standard shutdown state. Step 5 is necessary only if the instrument had been turned completely off. If the instrument was left in its standby state (x-ray power on but at low power) then you can skip steps 5, 6, 9, 10, 11 and 12. These steps are denoted *Optional/Inspect*.

1. Log on to the PC. The logon process will ask you to enter a recharge account and other information about your session.
2. Put on your dosimetry. Wear the full body badge on your shirt and the ring on your dominant hand.
3. Turn on the printers.
4. If you are using the VAX-based PCDMS software, turn on the two printers and the PC's monitor. (The MicroVAX and the PC are never turned off.)
5. ***Optional/Inspect*** – Turn on the chiller. Make sure that the process cooling water is turned on. (Red handles in the vertical position.) and that process cooling water is flowing (The inlet and

outlet pressures are different.).

6. **Optional/Inspect** – The power to the detector and microprocessor should already be on. If it isn't then press the square red button on the right rear corner of the diffractometer cabinet.
7. Check the bias voltage for the detector. It should read -1000 volts. If it reads 0 volts then the detector has warmed up (all of the liquid nitrogen had boiled off) and you will have to add liquid nitrogen and wait for the detector to cool down to its operating temperature. Once the detector has reached its operating temperature the bias voltage will automatically return to -1000 volts. This normally takes 15-20 minutes. After another 20 minutes or so the red LED on the bottom of the detector will go out, indicating that the detector is ready for use.
8. Check the liquid nitrogen level in the detector dewar. Add some if the level is too low to last through the rest of the day. Be careful not to add so much that it is poured out onto your specimen when during an experiment the detector is raised to higher angles.
9. **Optional/Inspect** – Adjust the "Emergency Shutdown" button on the high-voltage power supply to the up position. You'll need the key to do this.
10. **Optional/Inspect** – Push the amber "Control Power" button to turn on the control panel of the high-voltage power supply. The lights and LCD displays should come on.
11. **Optional/Inspect** – Press and hold the green "X-Ray Off" button while checking the values in the mA and kV settings. They should be 2.0 mA and -10 kV. Make any necessary adjustments now. Do not change the settings for "Filament Current" or "Power". These are safety limits and must not be changed by the average operator.
12. **Optional/Inspect** – Push the red "X-Ray On" button to energize the x-ray tube. The tube kV and mA settings should soon come up to -10 kV and 2 mA. Allow 5 minutes before proceeding with steps 14 and 15.
13. Use the hand-held radiation survey meter to make sure that there is no leakage of x-rays from the x-ray tube. Keep this meter accessible throughout your session, checking the tube (shutter closed, cabinet open) and cabinet (shutter open, cabinet closed (during scans)) periodically for leakage.
14. Enter the required information in the tube's log.
15. Increase the kV to the x-ray tube to a setting no higher than that given in the tube's log book.
16. Increase the mA to the x-ray tube to a setting not higher than that given in the tube's log book.
17. Re-check the shutter for the leakage of x-rays.
18. Enter information related to this new power setting in the tube's log.
19. Make sure the proper slits are installed, the proper filter is selected. Refer to the Standard

Operating Parameters article.

20. Start up the PCDMS or DMSNT software.
21. Verify that the goniometer is properly calibrated. Refer to Goniometer Check Procedure article.
22. Wait 10-15 minutes after completing step 15 for the tube to stabilize before starting a scan. This will allow the system, particularly the x-ray tube, time to stabilize and will ensure the most accurate results.

### **Specimen Change Procedure**

The following is the basic procedure for changing a specimen. The emphasis here is on the safe method of changing a specimen. Please also take care to prepare and mount your specimen correctly so that the results of your scan are accurate.

1. Make sure the shutter to the x-ray tube is closed. If either set of red lights on the x-ray tube are on then the shutter is open. If it is open then shut it by going to the manual page (F7), general (0) and pressing F1 to toggle the shutter closed. You will hear a solid click when the shutter closes and the red lights on the x-ray tube will go out.
2. Open the door of the cabinet.
3. Using the hand held radiation survey meter, check the shutter for the leakage of x-rays.
4. Remove and/or replace the specimen with a new one.
5. Close the door of the cabinet.
6. Press the red "Interlock Reset" button.
7. Now it is safe to open the shutter again or to start a new scan.

### **Shutdown Procedure**

The following is the basic procedure for putting the diffractometer in one of two states: standby, or completely off. To shut the system down completely execute every step in this procedure. To put the system in standby, execute every step except 4 through 7 and step 15. (These steps are denoted Optional/Inspect) Normally the system is returned to the standby state but if the system is not going to be used for more than two days it may be shut down completely.

1. Decrease the tube's mA setting to 2 mA.
2. Decrease the tube's kV setting to -10 kV. Allow 5 minutes for the tube to cool down before proceeding with step 4 below.

3. Enter information related to these new power settings in the tube's log.
4. **Optional/Inspect** – Push the green "X-Ray Off" button to remove the high-voltage power from the tube.
5. **Optional/Inspect** – Note the time the power to the tube was turned off in the tube's log.
6. **Optional/Inspect** – Push the amber "Control Power" button to turn off the high-voltage power supply.
7. **Optional/Inspect** – Press the "Emergency Shutoff" button. It should stay in the down position.
8. Turn off the hand-held radiation survey meter.
9. Return the tube and detector each to  $10^\circ$  ( $2\theta=20^\circ$ ).
10. Replace the standard slits and return the filter to its standard position.
11. Check the liquid nitrogen level in the detector dewar. Refill it if the level is so low that all of the liquid nitrogen will have boiled off before the next person uses the system.
12. Turn off the light in the diffractometer cabinet.
13. Close the door to the diffractometer's cabinet and press the "Interlock Reset" button so that the light goes out.
14. Do not turn off the power to the detector and microprocessor. (The red square switch on the right rear of the cabinet should stay lit.)
15. **Optional/Inspect** – Turn off the chiller. Leave the process cooling water on. (Red handles in the vertical position.)
16. Exit from the PCDMS or DMSNT program and log out. If you are running the PCDMS software on the MicroVAX you will also have to log out from the MicroVAX. This will return you to the PC's DOSSHELL menu program. Do not shut down DOSSHELL.
17. Turn off the printers and monitors.
18. Do not turn off the PCs or the MicroVAX.
19. Return your dosimetry to the place designated for their storage.
20. Leave the loaner key on the table in the diffractometer room, next to the computer.
21. Turn off the lights and lock up when you leave.
22. Look around the room one more time to make sure everything is neat and tidy and ready for

the next user.



## Using a Hand Held Radiation Survey Meter

A hand held radiation survey meter is provided to allow the operator to perform routine and periodic inspections for radiation leaks. The operator should inspect the instrument at the beginning of each and several times during the session. This article describes how to perform this inspection.

### About Hand Held Radiation Survey Meters

The radiation survey meter is composed of two parts: the monitor and the probe. The monitor houses the electronics, the batteries, the controls and the meter which displays the reading. This meter might be calibrated in milliroentgens per hour (mR/hr) or counts per minute (cpm). The probe is a wand which is attached to the meter via a coax cable. Probes differ in shape and in the type of radiation (alpha, beta or gamma) they were designed to detect. Please note the type of probe you are using so that you can be sure you are using it correctly.

### Radiation Units

Radiation units and the effect of radiation on persons is described in detail in your Radiation Safety Manual and in Safety Net number 15. A few details relevant to this article are summarized below.

The following table provides a quick reminder on how we describe the amount of radiation measured.

<u>Unit</u>	<u>Abbr.</u>	<u>Explanation</u>
Roentgen	R	Radiation exposure in air as ionizations per unit mass of air. (1 R = $2.58 \times 10^{-4}$ Coulomb/kg air)
Radiation Adsorbed Dose	rad	The amount of radiation deposited per unit mass of the absorbing material. (1 rad = 0.01 J/kg)
Roentgen Equivalent Man	rem	A measure of the biological effect of radiation. For x-rays, 1 rad = 1 rem.

Some common sources of radiation exposures and the dose equivalents are:

Coast-to-coast airline flight	3 mrem
Natural background radiation in the U.S.	150-300 mrem/yr
Chest radiograph	15-65 mrem/view
Full body CT scan (20 slices)	3000-6000 mrem

Typical allowable and measured doses for radiation workers at UC Davis are:

<10 mrem/month	Typical radiation dose received by radiation workers at UCD
2500 mrem	UC Davis maximum allowed dose per year, whole body
5000 mrem	Federal maximum allowed dose per year, whole body

### Inspection Procedure

During the inspection you will be placing the detector in all areas where radiation leaks might be found. Start the inspection at some distance from the x-ray source and work your way towards the source. In general, you will start by inspecting the instrument's cabinet and shielding for leaks followed by inspecting the area near the x-ray tube, checking for leaks around the tube's chamber

and shutter. The basic procedure is:

1. Turn the main selector switch to battery test. The dial should indicate a good charge on the battery. If not, replace the batteries immediately.
2. Turn the main selector switch to the most sensitive range.
3. Turn the audio off/on switch to on.
4. Note the level of background radiation.
5. Hold the meter in one hand and the probe in the other
6. Slowly snake the probe around in the area being surveyed. If you are using the beta-probe then make sure that the probe is turned perpendicular to the field so that ionization can occur. Listen to and watch the meter for changes in the count rate.
7. A reading significantly above background indicates a serious leak and should be reported to the instrument's manager.

# **Emergency Procedures for the Scintag X-Ray Diffractometer**

This article describes exactly what to do in the event that a hazardous situation develops and you find it necessary to quickly shut down the x-ray diffractometer. Possible emergency situations are discussed below as are the various shutdown procedures.

## **Definition of an Emergency**

For the purposes of this article we define an emergency as any situation where prompt specific action must be taken to:

- C Prevent injury to operators and/or people nearby
- C Prevent damage to the equipment
- C Prevent damage to the building.

## **Possible Emergency Situations**

After reviewing the system, taking into account the various levels of experience of our operators, allowing for non-standard uses of the instrument (i.e. high-temperature diffraction) and the types of things which can go wrong even in a properly maintained system it was possible to make up a short list of possible emergency situations. While no list can cover all possible emergency situations it should give the operator an idea of the types of hazards to be aware of.

- C Cooling water leaks which spray water either into the goniometer cabinet or the electronics.
- C Certain or suspected radiation exposure (possibly due to defective shutter or warning lights).
- C Evidence (sight, sound or smell) of arcing in and around the x-ray tube or along the high-tension cable.
- C Evidence (sight, sound or smell) of arcing in and around the detector.
- C Any evidence of fire or excessive heat (sight, sound or smell) in or around the instrument.
- C Emergency evacuation of the building.
- C Problems encountered during the use of the high-temperature attachment which may cause damage to the attachment, the diffractometer, or worse.
- C Rapid liquid nitrogen boil-off, indicating a problem with the detector or the dewar and posing a possible suffocation hazard.
- C Catastrophic failure of any component or subsystem.
- C Serious leaks in the process cooling water system.

## **Emergency Response**

Your exact response to an emergency situation will depend on the nature of the emergency, your familiarity with the equipment and your state of mind. An immediate evacuation might be necessary, or you might shut down part of the system or all of the system. Correct emergency responses for the typical laboratory emergency have been covered in the departmental safety training. You will have to assess the severity of the emergency you face and make your own decision based on your safety and operator training and your knowledge of the shutdown procedures described in this article. The one thing you should always do, however, is to let the appropriate people know when you have dealt with an emergency situation.

## **Emergency Shutdown Procedures**

The following procedures describe how to perform an emergency shutdown of the system. By performing all of them the whole system, except the computers and their peripherals, will be shut down. You may or may not have to shut down the whole system but if you do please shut down each sub-system in the order given below.

- 1. X-Ray Tube**                      Press the large round red button on the console of the high-voltage power supply. This will immediately shut off the power to the x-ray tube. Afterwards you will need a key to release this emergency interlock.
  
- 2. Detector**                        Press the large square red button on the rear of the cabinet. This will immediately shut down the detector electronics and the microprocessor.
  
- 3. Haskris Chiller**                Simply turn off the chiller using the switch on the front of the chiller. Flow of water to the x-ray tube and the high-temperature stage will stop immediately.
  
- 4. Process Cooling Water**       Turn off the process cooling water by turning the both red handles on the valves so that they are perpendicular to the direction of the pipes. Flow of water to and from the chiller will stop immediately.
  
- 5. Breaker Boxes**                In addition, one can turn off power to the system via the breaker boxes on the wall. If you use this method shut down the system in the following order: “XRD: HIGH VOLTAGE”, “XRD: LOW VOLTAGE” and then “XRD: CHILLER”. Finally, turn off the process cooling water.

## **Notification of Emergency Personnel and Responsible Persons**

If you ever perform an emergency shutdown to any part of the system you must contact at least one of the following persons or agencies:

- |                                 |  |
|---------------------------------|--|
| C The instrument’s manager      | Always   |
| C Departmental safety personnel | Nearly always, but especially whenever people or additional facilities are endangered, advisable |

whenever the emergency appears to be due to negligence, improper installation, operation or maintenance or the situation is likely to occur again, and encouraged when you have questions or concerns about your safety.

- C Environmental Health and Safety Any radiation exposure or suspected exposure.
- C Facilities Services (2-1655) Any building-oriented emergency, such as leaking pipes, problems with the ventilation system, lights and power, etc.
- C Campus Emergency Response (911) Whenever someone is seriously injured and needs immediate medical attention and whenever you are not able to perform an emergency shutdown or the emergency shutdown does not eliminate the immediate emergency situation.

The names and telephone numbers of people to contact in the event of an emergency are posted on the door leading out of the laboratory.



# Liquid Nitrogen Safety

This article provides an overview of the hazards associated with and the proper procedures for working with liquid nitrogen.

## Principle Hazards

There are four hazards associated with liquid nitrogen. They are:

1. High Pressure Gas      The large expansion ratio from liquid to gas (692 to 1) can produce high pressures if the container does not have adequate venting or pressure relief.
2. Contact with Materials      Some materials become brittle when cooled to the temperature of a cryogenic liquid. (Liquid nitrogen boils at 77K.) For instance, most polymers and ferritic steels. In addition, materials exposed to extreme cold for long periods of time or have undergone periodic warming should be inspected for cracking and crazing.
3. Contact with Personnel      The extremely low temperatures of cryogenic liquids can cause burns similar to those caused by extreme heat. Eyes are especially vulnerable to this type of injury.
4. Inadequate Ventilation      Boil-off of a cryogenic liquid can present hazards similar to those encountered when handling pressurized gas: displacement of air leading to suffocation, explosion hazards, corrosion, poisoning. (Fortunately nitrogen is neither corrosive nor poisonous.)

Users and persons near liquid nitrogen reservoirs should be constantly aware of these hazards and should take the necessary precautions to keep a hazard from becoming an injury.

## Handling Procedures

The hazards listed above require that special procedures be followed when using liquid nitrogen and when constructing equipment which will handle liquid nitrogen.

1. Piping or transfer lines should always be constructed so as to avoid trapping liquid in the line. Evaporation can lead to a pressure build-up and possibly an explosion of the line. If it is not possible to empty all of the lines then install safety relief valves and rupture discs.
2. Personnel should avoid wearing anything capable of trapping or holding spilled liquid close to their skin. An impervious apron or coat, cuffless trousers and high-topped shoes are recommended. Wear safety glasses or better yet full face protection. Remove all watches and jewelry. When gloves are worn they should be impervious and sufficiently large to be easily tossed off the hand in case of a spill.
3. Vent storage containers into a well ventilated area. Avoid the build-up of the gas in confined working areas.

### **Storage/Transport**

Some materials can be damaged or their properties altered by exposure to extreme cold. Use only approved containers for storing and transporting cryogenic liquids.

### **Disposal**

Since liquid nitrogen is not toxic one can dispose of it by letting it boil off, taking care that a hazardous build-up of gas does not occur. Do not pour cryogenic liquids down the drain. The water in the trap might freeze and crack the pipes or the liquid might boil violently, propelling water and waste back into the room.

### **Special Note on Suffocation Hazards**

The “feeling of suffocation” is actually triggered by abnormally high levels of carbon dioxide in the lungs. One may experience this when working in an enclosed unventilated room. One will not, however, experience this when working in a ventilated room which is being rapidly filled with a gas such as nitrogen or argon. Your only warning of eminent suffocation might be dizziness. Please be careful when using liquid nitrogen in any enclosed space.

## **Policy on Visitors in the X-Ray Analysis Laboratory**

Often one or more persons may accompany the operator of x-ray producing equipment for the purpose of guiding or advising the operator in a procedure, observing the experiment, or for personal reasons. Being as they are close to this instrument while it is operating these people should be provided the same type of radiation exposure monitoring as our authorized users. While not required by the University we decided to do this on the recommendation of the Office of Environmental Health and Safety and for the benefit of our visitors. Monitoring consists of issuing temporary personal full-body dosimetry and keeping a log of these visits. The policy and procedure pertaining to visitors is described in this article.

### **Definition of Visitor**

A visitor is any person other than authorized users who is present while the x-ray producing equipment is operating. While this definition would include persons who are present for only a few minutes (part of a tour group, dropping off specimens, custodians, etc.) this policy will apply only to persons who stay long enough to observe or participate in the experiments which are being conducted, especially if present when there is a possibility of x-ray exposure, i.e. during the sample-change procedure.

### **Restrictions on Visitors**

There are but three simple restrictions:

1. The visitor must stay clear of the x-ray source at all times.
2. The visitor may not operate any of the equipment.

Other than these two restrictions, the visitor is free to assist in preparation of samples, analysis of the results, etc.



# Part 4. Operating Procedures and Notes

## About the Scintag X-Ray Diffractometer

This article contains all the information that a user or potential user of the system would need to know when using the x-ray diffractometer. It lists the model and manufacturer of the instrument and its more important specifications and features.

### Instrument

Model	XDS 2000
Manufacturer	Scintag, Inc., 707 Kifer Road, Sunnyvale, CA 94086, (408) 737-7200
Date Installed	November 9, 1993

### X-Ray Source

Tube	Copper, 2.2 kW, 60 kV max, line focus (0.4 x 12 mm) Chromium, 1.7 kW, 60 kV max, line focus (0.4 x 12 mm)
Filters	Integrated $\lambda$ -filter wheel with Ni, Mn, V, Fe, Zr and open positions
Power Supply	4 kW switching, 0-60 kV (negative polarity), 0-80 mA, 14Vac and 5A for the filament, 0.005% line and load regulation and 0.01%/8 hours long term stability. Minimum operating settings are 9 kV and 2.0 mA.
Optics	Soller slits and divergence and scatter slits: 1, 2, 3, 4 and 6 mm

### Detector

Type	Germanium solid state
Active Area	80 mm <sup>2</sup>
Window	Beryllium, 0.05 mm thick
Resolution	195 eV (FWHM at 5.9 eV, <sup>55</sup> Fe, 6: s amplifier time constant)
Cooling	Liquid nitrogen, 3 liter dewar, 72 hours static holding time
Optics	Collimator block utilizing scatter and reference slits: 0.5, 0.3, 0.2, 0.1, 0.07, 0.05, dot-dot

### Goniometer

Type:	32/2 (22/2 and 2/2)
22 Range	-2 to 140 degrees 22
T Range	-2 to 90 degrees (incident angle)
2 Range	-2 to 70 degrees (detector angle)
Speed	Data collection: 0.1 to 120 degrees 22 per minute Maximum slew: 1500 degrees/minute
Step Resolution	0.000315 degrees per motor step
Peak Resolution	0.025 degrees 22
Goniometer Radius	160 to 300 mm

### Computer Control/Peripherals

Computer	DEC MicroVAX, 4 MB DRAM, 120 MB hard drive (DKA300), 1.44 MB floppy disk drive (DKA500), VMS 5.5 operating system and Multinet
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network software.  
 Console PC emulating a Tektronix 4200 graphics terminal  
 Network MicroVAX: xrd-vax.engr.ucdavis.edu  
 PC: xrd-pc.engr.ucdavis.edu

## **Cabinet**

General Heavy duty construction, completely encloses the goniometer, x-ray source and detector, large access door, leaded plexiglass viewing window  
 Interlocks Failsafe interlocks close the shutter to the x-ray tube when the cabinet's door is opened.

## **Special**

High-Temperature Temperatures up to 1500C in air, inert gas or vacuum depending on the specimen and the temperature. Powder (20 : m) specimens are ideal but thin sheets can be analyzed.  
 Thin-film attachment Allows for grazing incidence angle (0.1-2.0 degrees 2 $\theta$ ) analysis of thin films.  
 PC-PDF 2 The JC-PDF organic and inorganic databases on CDROM.

## **Software**

Title PC-DMS version 2.0 running on the MicroVAX. A Windows NT version is expected in September 1995.  
 Data Collection Step or continuous (normal, 2-theta, omega) scans, rocking curves, residual stress, retained austenite, signal enhancement, deadtime correction.  
 Data Analysis  $k_{\alpha 2}$  correction, background stripping, fft filtering, boxcar smoothing and peak shifting. Peak finder, peak area, reference standards, Warren-Averbach and Sherrer methods.  
 Database JCPDS, user databases, curves simulator, search-match, chemical search.  
 Crystallography Manual indexing, lattice refinement, Nelson-Riley, auto-indexing (Treror), Reitveld analysis, single crystal to powder simulator.  
 Quantitative I/I<sub>c</sub> quantitative analysis, total crystallinity, bulk silica, airborne silica.  
 Metallography Residual stress, stress parameters, retained austenite, subsurface residual stress.  
 Graphics Labeling and overlapping of data sets, overlaying of powder files 3-D displays, residual stress display, retained austenite display, profile fitting, rocking curve display.  
 Other Real-time display of data collection, data collection as a foreground or background process, batch-mode data collection and data processing, report generation, color printouts of the results, file conversion utility.

## Standard Operating Parameters

This article lists the standard configuration of the system and the operating parameters for typical data collection procedures. Users should always check the instrument's configuration before starting their work and when finished should always return it to the configuration listed below.

### X-Ray Source

Voltage: 45 kV  
Current: Copper tube: 40 mA, chromium tube: 35 mA  
Filter: None (depends on the sample and the tube used)

### Slits

Tube: Divergence=2 mm, scatter=4 mm  
Detector: Scatter=0.5 mm, reference=0.2 mm

### Detector

Bias: 1000 volts  
Range: 10,000 cps

### Data Collection

Step Size: 0.02 degrees 2 $\theta$  (equal to the reference slit divided by 10)  
Preset time: 0.5 to 2.5 seconds (typical range)



# A Typical Session on the Scintag X-Ray Diffractometer

## *PCDMS Version*

This article describes a typical session on the x-ray diffractometer where someone wants to perform a routine scan and use the search-match capabilities of the system to identify their sample.

(The numbers and such in parentheses refer to numbers and function keys on the keyboard of the PC. Capitalized words are usually either commands to enter, keys on the keyboard, or functions and programs which appear on the screen.)

## **Preliminary**

### **1. Reference Materials**

It is very helpful to have on hand copies of the powder diffraction files for your specimens. Use the ICDD PDF database to search for these cards and to print the ones which you think you will need. While you probably won't want to print out all of the files which match your search specifications it would be a good idea to print the list of these files. Simply having the list of card file numbers handy can be very helpful.

### **2. Start Up the System**

Start up the system in the usual manner. Refer to the safety protocol as needed.

### **3. Start the PC-DMS Software**

Log on to the MicroVAX and start the PC-DMS software by typing PCDMS and pressing the ENTER key.

### **4. Goniometer Check**

It is very important that you make sure that the goniometer settings and the PC-DMS software are in agreement. If the previous user had made any of a number of mistakes or if there had been a power failure then the goniometer will have to be recalibrated. To be safe, the goniometer calibration should be performed at the start of each session. Use the goniometer check procedure described in another section of this manual.

## **Perform a Scan**

### **5. Load a Specimen**

Prepare a specimen which is flat on the side which is to be exposed to the beam and load it into the specimen holder. Make sure that the specimen is flat and in the plane of the specimen holder. If the specimen is too high or too low then the positions of your peaks will be shifted. If the specimen is rotated then the peak shape will be distorted.

### **6. Perform a Scan**

From the DIFF page (F3) select either STEP SCAN (0) or CONTINUOUS SCAN (1). (In general, STEP SCAN is preferred.) Enter the following information:

File Name	The root name of the file to receive the data. PC-DMS will assign an extension.
Scan Type	NORMAL (T=2, T+2=22)
Specimen	Enter a brief description of your specimen.
22-Start	The 22 position where you want to start scanning (Refer to your PDF printout.)

22-End	The 22 position where you want to stop scanning (Refer to your PDF printout.)
Multi-Range	This can save you a lot of time on slow scans.
Step Size	Normally 0.02, equal to the receiving slit size in mm divided by 10.
Preset Time	How long to spend recording counts at each step.
Scaling	Scaling of the y-axis of the display.

Recheck all parameters on the screen, recheck the installation of the specimen, make sure the “Interlock Reset” light is off, then start the scan (F8). While “EXECUTE?” is displayed across the top of the screen you can press the SPACE bar to run data collection as a background job, press ENTER or simply wait a few seconds and the job will run in the foreground (default).

### Warning

If running the data collection as a background job do not do anything with the computer until the data collection has actually begun. Doing so seems to confuse the communications between the computer and the diffractometer and will cause erratic behavior of the system.

## Analyze the Data

### 7. Data Corrections

Go to the PROGRAM menu (F4) then the LEVEL 1 menu (0) and select BACKGROUND CORRECTION (0). Here you can specify fft filtering, boxcar smoothing,  $k_2$  correction and background stripping. Normally we do not filter or smooth the data but we do perform  $k_2$  the correction. Background stripping will shift the data towards the baseline. (In the process it will usually remove all signs of the presence of an amorphous phase, if one is present.) Make sure the file name and extension are correct and execute the program (F8). The results will be stored in a net intensity file (extension = NI). The original data file (extension = RD) is not modified.

### 8. Data Analysis (Peak Finder)

Go to the PROGRAM menu (F4) then the LEVEL 1 menu (0) and select PEAK FINDER (1). This program will locate and analyze the peaks in your pattern and will construct a table listing all peak positions, corresponding d-spacings, peak intensities, FWHM, areas, etc. This data can be used by the SEARCH-MATCH program, the NORMAL DISPLAY program, and others. It is also very useful just to have it in your files.

To use this program you must specify whether or not you want to use smoothing and you must set the threshold level (ESD Multiplier) for which any data below this level will no be considered a possible peak. Normally, the default values are adequate. Make sure the file name and extension are correct, toggle the PRINT control at the bottom of the screen so that the results are sent to the printer and then execute this program (F8). The results will be printed and also stored in an ASCII file whose extension is PKN.

Note that the results of this program are nominal as the data are fit using a generic polynomial. To obtain more precise fits use the PROFILE FITTING program (GRAPHICS page (F5), PROFILE FITTING (5)).

## 9. Identify the Sample

Go to the PROGRAM menu (F4), then the SEARCH MATCH menu (2) and select the SEARCH MATCH program (2). Make sure that the file name and extension are correct, the PRINT option is selected and then execute this program (F8) using the default parameters. The results are saved in a file which can be used by the RESULTS DISPLAY program. (You don't have to use the defaults but they will work for the typical situation. There are three screens of parameters one can change to narrow the search a bit and also tighten the criteria for a match.)

If no matches are found you should check the periodic table (PROGRAM (F4), SEARCH MATCH (2) and PERIODIC TABLE (3)) to make sure someone hasn't turned off elements in the periodic table. If too many matches were found you can specify your pattern matches more than 3 peaks in the database. You can also specify a higher probability factor.

## **Output the Data**

### **10. Graph the Results**

Go to the Graphics menu (F5) and select the RESULTS DISPLAY program (4). Make sure the file name and extension are correct, adjust the scaling if desired, and execute this program (F8). A graph of the data will be displayed in the top half of the screen along with a menu bar along the top of the screen. Select AUTO from the menu and sketches of the powder files in the list generated by the SEARCH MATCH program will appear. Select DELETE from the menu bar do delete and which you do not consider a reasonable match. The remaining sketches will scroll up to take the deleted sketch's place and next sketch in the list will appear at the bottom of the screen. Continue through the list in this manner, selecting PROJECT to overlay any of these sketches onto the data. Select COPY to print the screen and finally END to exit from this program.

### **11. Print the Results**

If you need to print out the peak file using the PEAK LISTER program (PROGRAM (F4), LEVEL I (0), PEAK LISTER (2)). Specify the name of the peak file, normally the same as the data file, and then select GO (F8). Be sure to toggle the PRINT option (CTRL-F4) if you want the output to go to the printer.

### **12. ASCII Conversion**

Go to the PROGRAM menu (F4) then LEVEL 1 (0) and select the FILE CONVERT program (4). Enter the file name of the data file and extension you want to convert and then execute this program (F8). This program will create ASCII versions (ARD and ANI) of the binary data files (RD and NI). It can also convert from ASCII to binary and binary to GSAS. The ASCII versions can be imported into many of the spreadsheet and graphics programs which run on PCs and Macs.

## **Conclusion**

### **12. Shut Down the System**

Shut down the system in the normal manner. Refer to the safety protocol as needed. Make sure that everything is turned off and that the laboratory is left in perfect condition for the next user.

### **13. Exit from the PC-DMS Program**

To exit PC-DMS and log off from the MicroVAX press shift-F8.

### **14. Transfer Files to a Floppy Disk**

Your files can be transferred to another computer using an ftp utility or to the MicroVAX's floppy disk drive using the procedure described in another article in this manual.

### **Batch Mode**

It is possible to set up a batch job where steps 6-9 can be done automatically. This can save you time and is very useful when you run the same procedure on a number of samples. Go to the BATCH page (F6) to set up the batch job. Save the batch file and then go to the STATUS page to execute it. Before executing it toggle the LOG FILE to PRINT/SAVE so that you get a printout of the results as well as a copy of this report on disk.

## **Goniometer Check Procedure**

It is not unusual to find differences in the actual position of the goniometer and the positions currently in the memory of the microprocessor and in the PCDMS program. Several things can cause this. We have found that if one aborts a scan then in almost every case it will be necessary to recalibrate the goniometer. If the system had been completely shut down or there had been a power failure the microprocessor will lose its memory of the positions of the various motors. This can be remedied by recalibrating the goniometer. The calibration procedure itself is very simple and involves little more than rotating the motors by no more than one revolution, entering the new angles and then loading these new, correct values into the PCDMS software and into the microprocessor. The procedures for determining if the calibration is necessary and how to perform the calibration are described in this article.

### **When One Should Check The Goniometer**

The goniometer should always be checked whenever:

- C You first start your session on the diffractometer
- C You abort a scan
- C The power to the microprocessor is lost
- C At the end of your session on the diffractometer.

It is very important that these checks be made since your results may look fine until you start to analyze them and you would have wasted valuable time and perhaps even a specimen. Then again, they might look fine and in fact be wrong.

### **Verify the Current Position with PCDMS**

This test checks to see that the PCDMS software has the correct values for the positions of the various moving parts of the goniometer. Go to the MANUAL (F7) and then the GENERAL (0) pages. Note the angles indicated in blue on the screen and compare these to the angles indicated on the goniometer. If they are different then you will have to recalibrate the goniometer.

### **Verify the Current Position with the Microprocessor**

This test checks to see that the microprocessor has the correct values for the positions of the various moving parts of the goniometer. While still on the MANUAL/GENERAL page:

- C Select the DR-MOT (F3) and enter a value for 22 (i.e., 20 or 40, something that when divided by 2 is easily read from the dials on the goniometer).
- C Select GO (F8) to reposition the goniometer to this angle.
- C The angles T and 2 should now both be equal to half of the angle you specified for 22. If not, then you will have to recalibrate the goniometer.
- C If the goniometer fails this verification it has also probably attempted to move past their limits and a collision error will be reported on the screen. If this is the case then you will have to back the arms off a bit before performing the calibration. To do this simply turn the silver TH and OM knobs on the top of the goniometer frame until the arms have moved back a few degrees.

## **Calibration Procedure**

This procedure will allow you to re-establish agreement between the actual goniometer position and the positions that PCDMS and the microprocessor have in their memories. While still on the MANUAL/GENERAL page:

- C Select CALIB (SHIFT-F1) to start the calibration procedure.
- C The calibration starts by moving each of the active components of the goniometer until it's index mark is found. This movement will never be more than 1 degree and is applied to each motor in sequence.
- C After each motor's index has been found you will be asked to enter a value for each motor which is active. (The motor for the angle N is normally turned off.)
- C Carefully read these values directly from the goniometer, enter them and then press F8 to load them into both the PCDMS and microprocessor memories.
- C Check the results by driving 22 to some convenient angle and checking the goniometer again to make sure it moved to the correct position.

## Useful VMS Commands

This document describes several VMS commands which the user of the Scintag diffractometer will find useful. These should be sufficient for the user to maintain the files in their own directory.

### File and Directory Specifications

The VMS file specification strategy is practically identical to that used by DOS. A file name consists of the name (10 characters max, 8 in DOS), an extension (3 characters max) and a version number. (File names are not case sensitive.) For example:

test.txt;10                      A file named TEST which has the extension TXT and is version 10 of this file.

Version numbers are not used by DOS, but are very useful. For instance, if you create a new a text file named “test” it would be specified as test.txt;1. If this file were edited then version 2 of this file, called test.txt;2, would be created and version 1 would not be overwritten. VMS usually assigns the version number, although the user can change it using the *rename* command.

Examples of file names are:

\*.\*;\*                      All files, regardless of name, extension or version number.

\*.ANI;1                      All files whose extensions are ANI and are version 1.

test.txt                      The version number need not always be specified. Often applications will simply use the highest version of a file.

VMS’s directory specification strategy is also very similar to that used by DOS except that the syntax is different. First of all, the name of a directory is enclosed in brackets [] but the rules for naming a directory are the same as for a file name (10 characters max, not case sensitive). Additional characters are used to specify the directory structure. Here are several examples:

[000000]                      Root directory of the current drive.

[guest]                      A directory named GUEST.

[users.smith]                      A directory named SMITH which is contained in a directory named USERS.

[-.ems132l]                      A directory names EMS132L which is located in the next lower branch of the directory tree.

A full device/directory/file specification might look like this:

ddif1root:[ems132l.group2]quartz.rd;3

where ddif1root: is the name of a drive partition but could be any logical device.

## Changing Directories

Changing directories in VMS is similar to changing directories in DOS except that the syntax is different. Instead of using the DOS *chdir* command, abbreviated *ch*, you use the VMS *set default* command, abbreviated *set def*. As for the names of the directories themselves the VMS files specification strategy is similar to that used by DOS but the syntax is different.

<u>DOS</u>	<u>VMS</u>	<u>Description</u>
c:\ cd d:\	\$set def ddiflroot:[000000]	Change to the root directory of the disk partition which contains the DMS users' files.
d:\ cd ..	\$set def [-]	Move back down the directory tree, towards the root directory.
d:\ cd ..\ems132l	\$set def [-.ems132l]	Change to the EMS132L directory which happens to be located in a directory one branch lower than the current directory.
d:\ cd guest	<u>\$set def [guest]</u>	<u>Change to the GUEST directory.</u>

## Directory Listing of Files

The *directory* command lists the files on the screen. Output can also be redirected to the printer or a file. Here are a few examples:

\$dir *.*	List all files in the current directory.
\$dir *.*;	List the latest versions of all files in the current directory.
\$dir [guest]*.ani	List all files in the GUEST directory which have the ANI extension.
\$dir [guest]*.* /printer	List all files in the GUEST directory on the printer.
\$dir/size=used/date=created/versions=1/protection test	Lists all files called test along with their size in blocks (512 bytes), creation data and file protection settings.

The *directory* command has a number of options which you may find useful. Use the *help* utility to find out more about them.

## Copying Files

The copy command will duplicate the specified file(s). Here are a few examples:

\$copy *.*;* [-.guest]	Copies all files to the GUEST directory.
------------------------	--

\$copy \*.\*;\* /since=today [-.guest] Copies all files created today to the GUEST directory. Any date including TODAY (default), TOMORROW and YESTERDAY can be used as dates.

\$copy Invokes the *copy* command in interactive mode. The system will prompt you for the source and destination file specifications.

## Deleting Files

The *delete* command permanently deletes the specified file. Here are some examples:

\$del \*.\*;\* Deletes all files in the current directory.

\$del guest.dir:1 Deletes the GUEST directory. This operation can only be performed on empty directories.

\$del \*.\*;\* /before=15-apr-1994 Deletes all files created before April 15, 1994. Any date including TODAY (default), TOMORROW and YESTERDAY can be used as dates.

Note that you must specify the file's version number.

## Moving/Renaming Files

The *rename* command can be used to both rename a file and to move it to another directory or device. Here are some examples:

\$ren test.txt;1 test01.txt;1 Renames the file TEST.TXT;1 to TEST01.TXT;1

\$ren test01.txt;1 [-.guest]\*.\*;\* Moves the file TEST01.TXT;1 to the GUEST directory.

\$ren \*.\*;\* [ems132l]\*.\*;\* Moves all files in the current directory to the EMS132L directory.

Note that like the *delete* command you have to explicitly specify the file version number.

## Help

Help on almost every VMS command can be obtained by typing help followed by the name of the command. Here are some examples:

\$help Invokes the help utility in an interactive mode. A list of topics for which help is available is listed. You can enter one of these. After the help text is displayed you can then ask for help on any of the command's options.

\$help directory Requests a description of the VMS *directory* (*dir*) command.

\$help directory /since Request help on the VMS *directory* command and specifically the effect of using the */since* option.

## Control Keys

The following control sequences can be very useful:

CRTL-S	Halt printing to the screen. Output to the screen will be halted until resumed by pressing CTRL-Q
CRTL-Q	Resume printing to the screen after pressing CTRL-S stopped printing.
CTRL-O	Stop printing to the screen. The current list is aborted.
CTRL-C	Stop the current process. Similar to CTRL-Break in DOS.
CTRL-Y	Stops the current program, returning you to the operating system.

## Other

Here are a few other commands which might be useful:

\$sh users	Lists the accounts which are currently logged on to the system. The <i>sh</i> command is the abbreviation for the <i>show</i> command.
\$sh users /full	List all current users and include details of their sessions.
\$stop process /id=xxx	Stops a running program. The ID xxx was listed by the <i>sh users /full</i> command
\$sh dev	List installed devices
\$sh dev dka300: /full	List the details of the hard drive, including the space used and the space free.
\$print test.txt;10	Prints the file test.txt;10 on the printer.
\$type test.txt;10	Displays the file test.txt;10 on the screen.
\$set pass	Invokes the password command in interactive mode. You will be prompted for the current and new passwords.

## **A Word or Two About High-Temperature X-Ray Diffraction**

Basic operation of the XDS 2000 x-ray diffractometer is both simple and inexpensive. High-temperature x-ray diffraction using the high-temperature chamber, however, is not simple and can be very expensive. The user who wishes to conduct high-temperature x-ray diffraction experiments should be aware of this. The special considerations and requirements of high-temperature x-ray diffraction are described in this article.

### **Setup**

It takes about a day to install and align the high-temperature chamber, connect the purge gas or the vacuum pump, and to test the heater.

### **Specimen**

Options on the quantity and form of your specimen are very limited. Ideally, the specimen should be a few grams of a fine (< 20: m) powder. If high quality results are not absolutely necessary then thin flat specimens can be used. The problems with this type of specimen are proper positioning of the specimen, obtaining good heat flow from the heater strip to the specimen, temperature gradients, and the fact that thin specimens tend to curl up when they are heated.

An expensive issue is the possibility that the specimen may react with the platinum heater strip. This heater strip, along with the attached thermocouple, costs around \$1,200 and would require better than a day to replace even if a spare were already on hand. All users of the high-temperature stage should be prepared to replace the heater strip.

Another concern is that the specimen may melt, out gas, boil or react in a way which would contaminate the chamber. The nickel sputter shield (\$300) will protect the beryllium window from sputtering but more serious contamination will require cleaning the chamber and most likely replacing the beryllium window (\$5,000). The user will be asked to bear the costs of any repairs.

### **Data Collection Rates**

Data collection rates are a factor of about 100 slower than routine ambient data collection. This is because of a 20% loss in intensity due to the beryllium window with the remaining loss in intensity due to the nickel sputter shield. In a recent series of experiments a scan rate of 0.01 degrees 2 $\theta$  was used. Data collection took over 12 hours each and maximum peak intensities, which were normally several thousand counts per second, were around 60 counts per second. The low scan rate used allowed for more time to count x-ray photons at each step in the scan. This gave us the excellent counting statistics required to get accurate peak profiles.

### **Take-Down**

Removing the high-temperature chamber and remounting the standard specimen holder takes about a half hour. Realigning the goniometer, however, takes from one half a day to 2 days, depending on how successful one is at mounting the standard specimen holder in exactly the same position as it was before installing the high-temperature stage. The alignment procedure is tedious but not difficult except that there are so many variables, making it difficult to judge which ones are out of spec<sup>2</sup>.

## Policy

For the good of all users we have established the following policy regarding the use of the high-temperature chamber:

1. Before using the chamber the user must provide a detailed description of temperatures and atmospheres that will be used along with a description of the specimen, its composition and form (i.e. powder, strip).
2. The user must describe how the specimen will respond to the high temperature and vacuum or gas atmospheres, addressing such things as out-gassing, melting, sublimation and compatibility with the material used in the construction of the specimen stage. The user might be asked to demonstrate the compatibility of the specimen and the stage materials.
3. The user must agree to replace or repair any and all parts which are damaged during their use of the high-temperature chamber, vacuum system and inert gas system. This might involve something as simple as cleaning and polishing the chamber walls to replacing the beryllium window.
4. Because of the effort required in installing the high-temperature chamber and restoring the diffractometer to its original general-purpose condition we will schedule the high-temperature experiments at a time when the demand for the diffractometer is low.

## Itemized Costs

For your information, the cost of the chamber and selected components are:

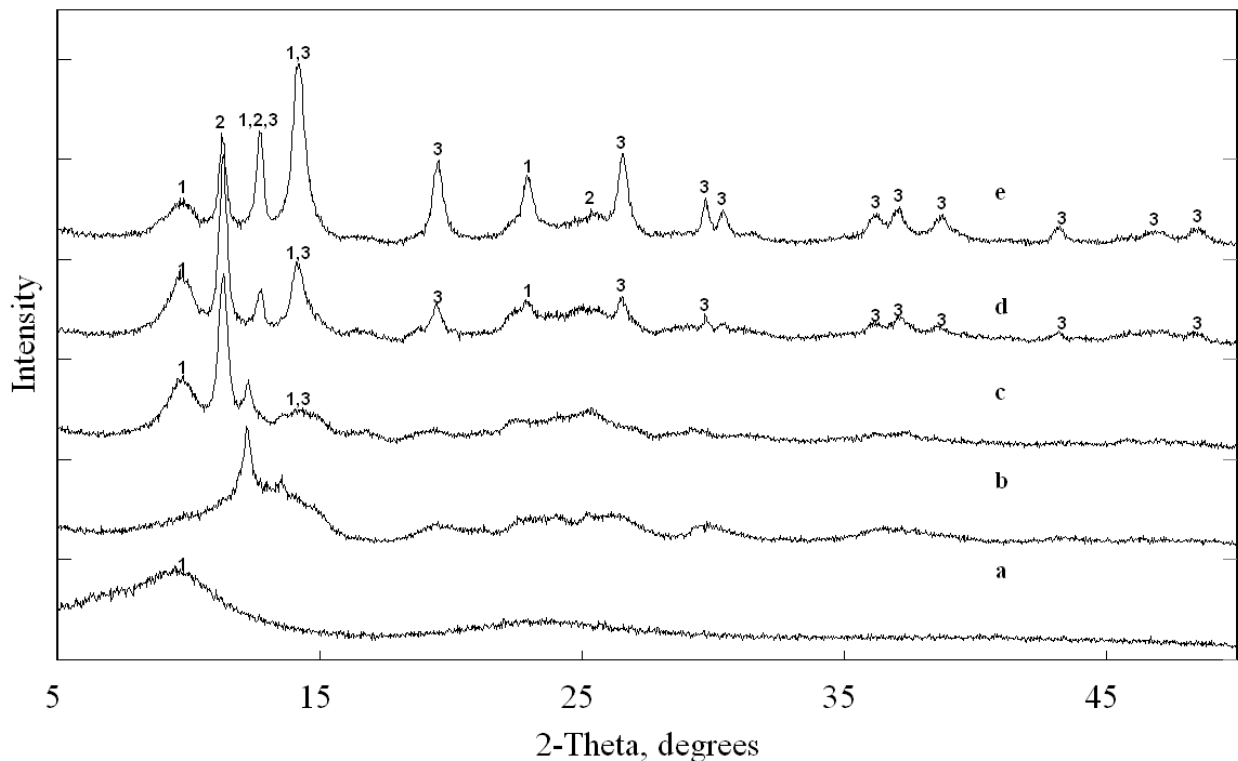
Complete high-temperature chamber:	\$34,000
Replacement beryllium window:	\$5,000 + shipping + 4 weeks
Replacement of Pt specimen stage:	\$1,200 + 1 day
Replacement of Pt-thermocouple:	\$135 + 1 day
Replacement of the nickel foil:	\$350 + 20 minutes
Setup and take-down:	1-2 days
Rerun standards:	1-2 days

All work will be recharged at the standard service rate.

## Importing PCDMS Data into a Spreadsheet

This article describes how to import ASCII data from Scintag x-ray diffractometer and generate a graph. It includes a description of Scintag's data format followed by the procedure for building a Quattro Pro spreadsheet which involves importing several data files and plotting them all on the same graph (see figure 1). (Microsoft Excel users should have no problem translating these instructions into the equivalent Excel commands.) It also shows how to use an offset so that the curves do not fall on top of each other and it shows how to label individual peaks. Familiarity with the spreadsheet programs is assumed. Basic skills should be sufficient.

The original raw data (\*.RD) and net intensity (\*.NI) files are stored in a binary format which cannot be used by programs which run on Macs and PCs. Make sure your data files have been converted to the ASCII format (\*.ARD and \*.ANI).



**Figure 1** An example of the type of graph generated in this example. Each line is spaced vertically by 1000. The tick marks along the y axes indicate the baselines for each line.

## Format of the PCDMS Data File

The data file from a "step" or "continuous" scan consists of a header containing the description of the specimen, the time and date the scan was done and the significant parameters from the scan such as 2 $\theta$ -start and 2 $\theta$ -end, preset time, step size, wavelength and others. Below this header are three columns of numbers. The first column contains the values of 2 $\theta$  in degrees, the second column the intensity in counts per minute and in the third the standard deviation of the intensity in counts per minute. Below is a small portion of a Scintag data file, showing the header and the first 10 data

points.

```
AL/MG/R1 (94)
5.000000 70.000000 0.030000 0.90000 0.00000 1.54059994
 3200 0 0
 8 26 94
18 59 27
 4383 349 56466.
0 0 0 0 0 0 0 0 0 0 0 0
5.00000 30667. 1430.
5.03000 31600. 1451.
5.06000 29267. 1397.
5.09000 29800. 1409.
5.12000 31400. 1447.
5.15000 34400. 1514.
5.18000 31467. 1448.
5.21000 33333. 1491.
5.24000 30067. 1416.
```

## Sample Data Import/Graph Procedures

This sample procedure demonstrates how to import several data files, to display them all on the same graph and to label the peaks. To construct this spreadsheet you will need five ASCII data files (\*.ARD, \*.ANI). A printout of each peak-file will also be useful.

This example assumes that all data was collected using the same starting and ending 2 $\theta$  and the same step size or chopper increment. This special case will allow you to define one column for the 2 $\theta$  axis with the intensity data for each file in adjacent columns. It is still possible to combine data from scans using different scan parameters but be warned that the final spreadsheet will be much larger and therefore much slower.

Quattro Pro version 6.0 and above uses a tabbed notebook scheme that allows one to put different parts of the spreadsheet on different pages. This example makes use of this feature.

### 1. Create a Blank Spreadsheet

From the *File* menu select *New* to create a new, blank spreadsheet.

### 2. Create the Pages of the Notebook

Create and name the pages of the notebook. Call the first one the "Title and Graph", the next "Data for the Graph" and the third "Imported Data". The "D", "E" and other pages will be used as scratch pads.

### 3. Import a Scintag File as Text

Go to an unused notebook and import the data file as text by selecting *Text Import* from the *Notebook* menu, locating the file, and importing it.

### 4. Parse the Imported Text

Delete every row down to the first row of 2 $\theta$  data and then insert one row at the top. Now select all of the data in the that column, from the top most cell to the last cell containing the imported text.

Use *Parse* from the *Notebook* menu to convert this column of text into three columns of numbers and place the parsed data in another unused notebook page. When done, delete the original imported text.

### 5. Create the 22 Column

Copy the 22 data for this first file to column “A” the “Imported Data” page and then copy the intensity data to column “B” of the same page. Leave a couple of blank rows at the top and enter the name of the ASCII data file at the top of the column containing the intensity data. Delete the parsed data on the scratch pad page.

### 6. Import and Install the Other Files

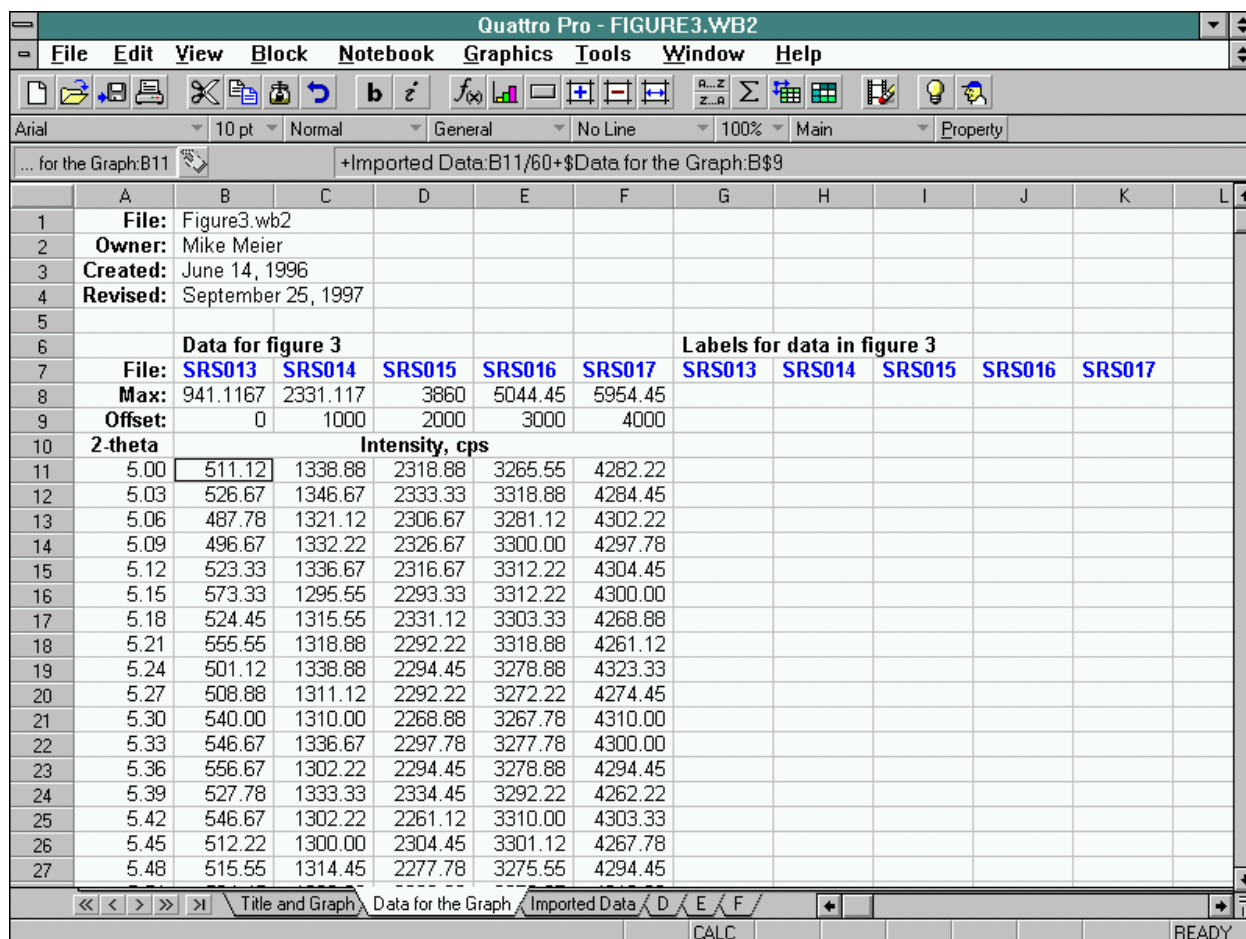
Repeat steps 3 and 4 for the next ASCII data file. After the new data is parsed copy the intensity data to the “Imported Data” page, placing it next to the other intensity data, and then delete the parsed data. Repeat this procedure for the remaining data files.

### 7. Set up the Table for the Graph

The data will be organized as one column for the x axis (22) and n columns for n data sets (diffraction intensity in counts per second). Figure 2 shows what this table will look like. To create this table:

- C Copy the first column of numbers in the “Imported Data” page to the “Data for the Graph” page. The first 22 value should be placed in cell A11.
- C Enter the file name of the first data set in cell B7.
- C Enter the initial value for the offset in cell B9. This offset will be used to shift the data up or down on the graph.
- C In cell B11 divide the data in the intensity column for the corresponding data file by 60 and add the offset in cell B9. The syntax for referring to cell B9 should be  $\$B\$9$ . This will make it so that the reference to cell B9 will not change when we replicate the equation in cell B11. The formula should look something like  
$$+\text{Imported Data}:B11/60+\$B\$9.$$
- C Repeat this procedure until cells C9, D9, etc. contain values of offsets and cells C11, D11, etc. contain formulas for the intensity data.
- C Copy the formula in cell B11 to cells C11 through cell x11 where x refers to the last column of data. Now copy cells B11-x11 to cells B12 through By where y refers to the end of the columns of data.
- C Enter into row 8 of each column the function which returns the maximum values of the data in each column ( $+\text{@max}(b11..by.)$ ). This information can be very useful when defining the scaling of the y axis of the graph.

The data table is now ready for plotting. The data in column A will define the x-axis, the data in



**Figure 2** Screen image of the “Data for Graph” notebook page in the sample spreadsheet. Note that the first cell of the intensity data is selected and that the formula in this cell is displayed in a field near the top of the screen.

column B the first y-series, C the second y-series, etc.

## 8. Create the Graph

Create an xy graph where column A contains data for the x series, column B the data for series 1, column C for series 2, etc. Set the scaling of the graph and adjust the values of the offsets to display the data the way you want it.

## 9. Label the Peaks

In the columns to the immediate right of the intensity data columns you can enter letters, numbers or other codes to identify individual peaks in your data. Your peak-finder printout can help you here. Define each of the label columns as the data labels.

## 10. Annotate the Graph

Use Quattro Pro’s annotate feature to label each curve with the number or letter you will use to refer to each of them.

## 11. Insert the Graph in the Spreadsheet

Insert the graph in the “Title and Graph” page of the spreadsheet and add the spreadsheet’s title, file

name, owner, creation and revision dates, etc. This makes an attractive printout and a convenient way to monitor your changes to the graph, its data labels, etc.

## **12. Export the Graph**

Quattro Pro 6.0 will let you export the graph as either a BMP, EPS, TIF, CGM, GIF or PCX file. Quattro Pro 7 and above supports additional graphics file formats. Figure 1 was exported from Quattro Pro 8 as a TIF file after displaying the graph on the screen as large as possible. One can also simply copy a graph to the clipboard and then paste it into a document.