XSCANS Software Users Guide

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Address comments to:

Marketing Communications Department
Bruker AXS Inc.
5465 East Cheryl Parkway
Madison, Wisconsin 53711-5373
USA
# XSCANS Users Guide

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Appendix A:  YLID Test Crystal

Appendix B: Absorption Correction Programs
1 General Information

1.1 Introduction

The XSCANS Data Collection Package is a diffractometer control system for use with the Siemens P4 and P4/RA Single Crystal Diffractometer Systems. This package provides a convenient and user-friendly environment through which you can perform all required instrument operations associated with high quality X-ray intensity data collection from single crystal samples. After the data is collected, SHELXTL can be used to solve and refine crystal structures.

XSCANS includes a variety of routines ranging from low-level hardware functions (open/close shutter, insert/remove attenuator, drive axes, etc.); instrument utilities (calibrate attenuator and dead time, tube alignment, etc.); crystallographic routines (reflection search and center algorithms, autoindexing, least squares calculation of unit cell parameters, determination of Bravais lattice and Laue class, etc.); and intensity data collection routines (THETA/2THETA, OMEGA and THETA/XTHETA scan data collection). Improved features since XSCANS v2.1 include new algorithms for data profiling, PSI scan tools, check reflections selection, extended (coupled) angular limits, new sorting utilities and an AUTOSCRIP option.

XSCANS is a screen-oriented, menu-driven system with flexible keyboard and mouse input capabilities and graphic output.
1.2 Manual Organization

This manual is written for first time users of XSCANS. It supplements detailed information contained in the XSCANS Technical Reference Manual. If you are a first-time user of XSCANS, this manual provides you with enough information to begin using the system in a very short time. If you have a limited background and experience in single crystal X-ray diffraction, consult the textbook Crystal Structure Analysis\(^1\).

We strongly recommend that you follow the example YLID data collection (Sections 4 through 23) at least once, whether you are experienced or not. This will help you to become familiar with the most commonly used XSCANS procedures.

This manual is organized in a sequential, step-by-step manner. Each step in the data collection process is explained as it is encountered. Where appropriate, references are made to specific sections of the XSCANS Technical Reference Manual.

Section 1 General Information

Describes the manual conventions, keyboard and mouse input conventions, and the P4 hardware features that must be present for XSCANS to function.

Section 2 Getting Started

Describes the procedures that are used to start XSCANS, to load and save parameter files, to edit input data, and to interpret the various portions of XSCANS screens. Procedures for paging through output and for obtaining screen dumps are described. Online help, logfiles, and program interruption is also explained.

Section 3 Basic P4 Operation

Describes how to control the basic hardware functions with XSCANS.

Sections 4–23

Describe a sample interactive data collection for the YLID test crystal supplied with your instrument. Most of the procedures in these chapters are the same as those used for unknown samples.

Section 24 AUTOMATIC Mode

Describes an automatic way to perform the same tasks as those used in interactive data collection. Although you can use AUTOMATIC mode for most samples, it is important that you understand the procedures described in Sections 4–23. AUTOMATIC mode performs the same basic procedures as used in interactive data collection.

Sections 25–30

Describes other commonly used XSCANS features, some of which are meant to be used only by your System Administrator and are not included in this guide (see Appendix C of the XSCANS Technical Reference Manual System Administration).

Appendix A YLID Test Crystal

Provides a crystallographic description of the YLID test crystal.

Appendix B Absorption Correction Programs

Describes the programs used to perform absorption corrections.

1.3 Manual Conventions

This guide adopts several format conventions to simplify explanations. The following items demonstrate how and when these various formats are used.

1.3.1 Keys

The names of keyboard keys are in **bold** type. In addition, they appear in all capital letters within the text or when used with a command. For example, the carriage return keys ("ENTER" or "RETURN" on your keyboard) are presented in this guide as follows:

**ENTER**

Since keys that perform similar functions may differ between keyboards, standard key names are used in this guide to represent equivalent keys. In this guide, **RETURN** is synonymous with **ENTER**.

When two or more keys should be pressed at the same time (such as the **CTRL** and **C** keys), both keys are set in **bold** type and separated by a slash as follows:

**CTRL/C**

1.3.2 Mouse, Trackball, Etc.

Although some users may have trackballs or other pointing devices on their system, this manual refers to all such devices as "the mouse," and assumes a right-handed, three-button mouse at all times.

1.3.3 Switches

Control switches appear in **bold** type and in capital letters. The position (or setting) of the switch is set off with quotation marks and is also in capital letters. For example:

Set the **POWER** switch to "ON."

1.3.4 Input

Anything that must be typed (input) at the terminal is presented in Courier font and in **bold**. The required input appears, indented, on its own line.

This convention helps to differentiate the command from other text appearing on the page. For example, if you must type in the command "LIST," then press **ENTER**, it appears in this guide as follows:

**LIST ENTER**

1.3.5 System Responses

System responses to your input also appear in the Courier font. For example, the message "Press Any Key When Ready" appears in this guide as follows:

**PRESS ANY KEY WHEN READY**
1.3.6 Procedures and Menu Names
Procedures and menu names are depicted as:
CONFIGURE/READ

1.3.7 Command Paths
In this guide, the following format is used to show the path or location of a command or subcommand:
GONIOMETER/SHUTTER
This means that SHUTTER is an option on the GONIOMETER menu. Paths are listed in general to specific order.

1.3.8 Precautionary Statements
There are three types of precautionary statements in this manual:

CAUTION
CAUTION: Alerts you to a condition that may result in serious equipment damage (including destruction of data).

WARNING
WARNING: Alerts you to a hazardous situation. If the warning statement is ignored, injury due to X-ray exposure or high-voltage shock may result.

Note: Notes contain information especially pertinent to the task at hand, and generally outline preferred or recommended procedures to follow in specific situations. They are in italic type.
1.4 User Input/Output Conventions

1.4.1 Keyboard and Mouse Input

Input to XSCANS is accomplished with a graphical user interface. Commands or input parameters can be selected with the keyboard or mouse. To select a command or parameter, highlight the appropriate line on the screen. A highlighted line is displayed in reverse video (onscreen, highlighted lines are displayed with black characters on a white background). Non-highlighted lines are displayed as white characters on a black background. You can move the highlighted line in one of three ways:

- Move the mouse
- Use arrows
- Type the first letter of the command or parameter; the highlighted line moves down to the next command starting with that letter.

When you enter parameter values, a cursor on the highlighted line indicates where the next typed character will appear. The cursor is displayed as a white character on a black background.

ESC and ENTER have important functions. Generally, ESC is used to go to a higher level menu when no other operation is being performed. ENTER is used to go to a lower level menu, or to execute the indicated command. If you want to change parameter values, but not execute the command, terminate the parameter input with ESC. If you wish to execute the command after all parameter values are entered, use ENTER. See Section 2.8 Editing Parameter Input.

The keyboard keys and corresponding mouse buttons have the following functions:

ESC or center mouse button: Exit from an input panel or menu without executing the command.

ENTER or left mouse button: Execute the selected command or go to the selected lower menu.

CTRL/BREAK: Interrupt the operation in progress, such as centering or data collection.

up/down arrows (or mouse movement): Move the highlighted line in an input panel or menu.

left/right arrows (or mouse movement): Move the cursor to the right or left.

INS: Toggle between character insert and character overstrike modes when editing a line in an XSCANS panel.

END: Move the cursor to the end of the highlighted line.

HOME: Move the cursor to the beginning of the highlighted line.

DEL: Delete the character at the cursor position.

BACKSPACE: Delete the character to the left of the cursor position.

CTRL/K: Delete to the end of the highlighted line beginning with the cursor position.

PGUP and PGDN: Move the highlighted line to a new page in the panel.

Characters can be entered in lowercase or uppercase letters. XSCANS interprets these characters in the same way.
1.4.2 User Output

Some routines output more information than can be displayed onscreen at one time. To view all the output, use **PGUP** and **PGDN** to page through the output. Press **ENTER** to see the VIEW/EDIT menu. See Section 13.7 of the *XSCANS Technical Reference Manual* for information on how to use these additional functions.
1.5 P4 and P4/RA Diffractometer Hardware

The Siemens P4 and P4/RA Crystallographic Systems are state-of-the-art research instruments to determine complete X-ray structure of single crystal specimens. The systems include the following hardware components (Fig. 1-1):

- X-ray source (3kW X-ray generator and sealed X-ray tube for P4, or 18kW rotating anode for P4/RA)
- Radiation safety enclosure with interlocks and warning lights
- X-ray shutter assembly
- Automatic attenuator (P4 only)
- Graphite crystal monochromator
- Incident beam collimator system
- Goniometer assembly
- Goniometer head
- Microscope for viewing the specimen (not shown)
- Detector system
- Diffracted beam collimator system
- Manual control module
- Ratemeter (not shown)
- Optional low temperature attachment (not shown)
- General Goniometer Controller System (GGCS)
- Main instrument control computer

Fig. 1-1: P4 single crystal X-ray diffractometer system
Before beginning YLID data collection, you should become familiar with some basic hardware and software functions of XSCANS and the Siemens P4 or P4/RA diffractometer systems.

2.1 Starting XSCANS

To start XSCANS, type:

XSCANS ENTER

from any directory. The Siemens logo appears, followed by the COMMANDS menu (Fig. 2-1).

XSCANS automatically loads the default parameters from a file called P4_DEF.P4P and establishes communications between the computer and the diffractometer controller.
2.2 XSCANS Screen

The main features of the XSCANS opening screen are shown in Fig. 2.1. The XSCANS title appears at the top of the screen and indicates the copyright and version number.

*Note: You must know your software version number when calling Siemens for technical support.*

The COMMANDS menu shows the available XSCANS commands. Other menus, when displayed, appear in the same part of the screen as the COMMANDS menu.

The CRYSTAL_ID name and the extended $\omega/\chi$ and $\omega-2\theta/\chi$ angular limits file prefix appear near the bottom of the screen (see the XSCANS Technical Reference Manual). The bottom line gives information about the highlighted command. The diffractometer status area, located in the lower right-hand corner of the screen, provides information on the current angles for the various goniometer axes. The shutter status is also shown in this area.
2.3 Online HELP Text

XSCANS includes online documentation for all commands and parameters. To access the online HELP documentation, move the highlight to the desired command. The corresponding HELP text appears on the last line of the screen (Fig. 2-2).

Much of the XSCANS Technical Reference Manual can be accessed with COMMANDS/HELP. To view the help files:

1. Choose HELP. The HELP INDEX menu appears (Fig. 2-2), which consists of the titles of the sections in the XSCANS Technical Reference Manual.

```
HELP INDEX
Introduction
About XSCANS
XSCANS Features
XSCANS Conventions
Files
Listing Files
Keyboard Conventions
Starting XSCANS
Commands Menu
Automatic Mode Menu
Autosave and Collection Parameters
Collect Menu
Himac Menu
Choose Checks
Edit Checks
Get Checks
Starts Menu
Scan Menu
Output Menu
```

2. Choose the title of interest.

XSCANS displays your selection (Fig. 2-3). Press PGUP or PGDN to move through the text.
HELP on Search Menu

13.14 Search
Using this option, you have access to the different XSCANS search procedures. It brings up an input panel, from which the search type is determined by parameter settings.

SEARCH parameters are:

- **Number of Reflections** (Default: 25) Defines the maximum number of reflections in an incremental, thin shell or random search.
- **Search Type** (Default: HEAVY) Defines the type of search. Seven search routines are available: HEAVY, INCREMENTAL, RANDOM, PHOTO, FUNCTIONAL, THIN shell, and AXIS.
- **Minimum 2-theta** Default: wavelength dependent) Defines the minimum 2-θ value for the incremental search. Since the

Crystal: P4_β, Extended w/x & w-2θ/x limits: p4_β

Line 11: Press left mouse button or ENTER

2-θ  0.00
    0.00
    311.21
    13.94
Shutter CLOSED

Fig. 2-3: XSCANS reflections/search HELP information

To see more viewing options, press ENTER (Fig. 2-4). See Section 13.7 of the XSCANS Technical Reference Manual for more information.

HELP on Search Menu

13.14 Search
Using this option, you have access to the different XSCANS search procedures. It brings up an input panel, from which the search type is determined by parameter settings.

SEARCH parameters are:

- **Number of Reflections** (Default: 25) Defines the maximum number of reflections in an incremental, thin shell or random search.
- **Search Type** (Default: HEAVY) Defines the type of search. Seven search routines are available: HEAVY, INCREMENTAL, RANDOM, PHOTO, FUNCTIONAL, THIN shell, and AXIS.
- **Minimum 2-theta** Default: wavelength dependent) Defines the minimum 2-θ value for the incremental search. Since the

Crystal: P4_β, Extended w/x & w-2θ/x limits: p4_β

Move down one page (PyDn key)

2-θ  0.00
    0.00
    311.21
    13.94
Shutter CLOSED

Fig. 2-4: XSCANS HELP menu VIEW/EDIT
2.4 XSCANS Files

All files written and read by XSCANS are ASCII files. This means that they can be displayed, printed and edited by standard DOS utilities and text editors, copied to other media (diskette), or transferred via network to other computers.

XSCANS files are characterized by a CRYSTAL_ID name which is normally the same for all files associated with a particular analysis. Each file also has a three character extension. For example, the filename YLID.P4P consists of a CRYSTAL_ID name, YLID, and an extension, .P4P. The extension normally determines the type of file. In this example, .P4P indicates that the file is an XSCANS parameter file. YLID.P4T is a file associated with YLID analysis and contains the raw intensity data from THETA/2THETA data collection. A complete list of extensions and the corresponding file types is in the XSCANS Technical Reference Manual.

You should copy these files to a diskette or other storage medium after each sample analysis. You should backup CRYSTAL_ID.* which includes all files associated with a particular analysis.
2.5 Parameter File

Parameter files are used to define the specific configuration of your diffractometer and all the parameters for a particular analysis. These files have the extension .P4P. The default parameter file name is P4_DEF.P4P. The default file is in the data directory and is designated in CONFIGURE. The file is always loaded from the current directory when XSCANS is started (see Appendix C of the XSCANS Technical Reference Manual).
2.6 Loading the Parameter File

To load an existing experimental parameter file, follow these steps:
1. Choose LOAD from the COMMANDS menu (Fig. 2-5).

Fig. 2-5: LOAD option

2. XSCANS displays the LOAD Options input panel (Fig. 2-6).

Fig. 2-6: LOAD Options input panel
If you know the name of the filename, enter the CRYSTAL_ID name (the extension is not needed). If you do not know the filename, press SPACEBAR followed by ENTER. A list of filenames appears (Fig. 2-7).

![XSCAN list example](image)

Fig. 2-7: XSCANS file list example

3. Choose the file you want to load. The name of the file appears in the LOAD Options panel. If the filename is correct, press ENTER.

The parameter file can also be loaded with CONFIGURE/READ (see Section 6.2 of the XSCANS Technical Reference Manual).
2.7 Saving the Parameter File

The parameter file should be saved periodically. It contains information about the centered reflections, unit cell parameters, data collection parameters, etc., which are determined during sample analysis. If you need to restart XSCANS at some point, you can recover all information if you have saved the parameter file. Follow these steps to save the current parameter file:

1. Choose SAVE from the COMMANDS menu (Fig. 2-8).

![Fig. 2-8: SAVE option](image)

2. The current filename appears in the SAVE Options input panel (Fig. 2-9).
3. Enter a new filename (only the CRYSTAL_ID name is required). If you do not know the filename, press SPACEBAR, then ENTER. A list of filenames appears (Fig. 2-7). Select the desired filename. An existing file is overwritten by the SAVE command. To save the file without overwriting an existing file, you must enter a unique filename. When you are sure you have chosen the correct filename, press ENTER.

The parameter file can also be saved with CONFIGURE/WRITE (see Section 6.3 of the XSCANS Technical Reference Manual).
2.8 Editing Parameter Input

Use the keyboard keys listed in Section 1.4.1 Keyboard and Mouse to enter and edit input parameters. Use the up/down arrows to see additional lines of input. If you must put more than one parameter on a line, separate the parameters with spaces. Terminate a line of input with ← or →, or any other keystroke that causes the highlight to change to a different line. Use ESC or ENTER only when you have finished editing.

Examples of valid lines of input into the reflection array are:

```
h 1 2 3 ↑
h -1 -2 -3 ↓
ESC
h 1 2 3 ENTER
```

An example of an invalid line of input is:

```
h 1 2 3 ESC
```

The higher level REFL_ARRAY menu is displayed when you press ESC.

Pressing ENTER causes three different things to happen, depending on the situation:

- A selected procedure starts; for example, if the LEAST SQUARES Option panel is displayed, press ENTER to execute LEAST SQUARES (see Section 10 LEAST SQUARES), or:
- A lower level menu appears (for example, when editing the reflection array, press ENTER to see the VIEW/EDIT menu), or:
- If there is no lower level menu and no procedure to be executed, press ENTER to see the next higher level menu (for example, if you press ENTER in the SCANS Options input panel, the higher level COLLECT menu appears).

For large arrays of input (reflection array, check reflection array, or crystal faces array) there are additional editing functions, such as SELECT, CUT, STORE and PASTE (see Section 13.7 of the XSCANS Technical Reference Manual). When editing any of the arrays, press ENTER to display the VIEW/EDIT menu (Fig. 2-10).
<table>
<thead>
<tr>
<th>View/Edit</th>
<th>H</th>
<th>K</th>
<th>L</th>
<th>2Theta</th>
<th>Omega</th>
<th>Phi</th>
<th>Chi</th>
<th>Incos</th>
</tr>
</thead>
<tbody>
<tr>
<td>Exit</td>
<td>0</td>
<td>0</td>
<td>2</td>
<td>9.626</td>
<td>8.299</td>
<td>298.826</td>
<td>41.474</td>
<td>723437.88</td>
</tr>
<tr>
<td>Edit</td>
<td>0</td>
<td>-1</td>
<td>2</td>
<td>13.759</td>
<td>-0.355</td>
<td>341.487</td>
<td>26.514</td>
<td>1948822.88</td>
</tr>
<tr>
<td>UpPage</td>
<td>1</td>
<td>0</td>
<td>-1</td>
<td>15.628</td>
<td>8.294</td>
<td>185.739</td>
<td>31.849</td>
<td>427358.88</td>
</tr>
<tr>
<td>DownPage</td>
<td>0</td>
<td>1</td>
<td>3</td>
<td>17.481</td>
<td>8.388</td>
<td>243.947</td>
<td>34.643</td>
<td>715757.68</td>
</tr>
<tr>
<td>Top</td>
<td>1</td>
<td>-1</td>
<td>-1</td>
<td>18.482</td>
<td>-0.337</td>
<td>68.151</td>
<td>25.897</td>
<td>989557.68</td>
</tr>
<tr>
<td>Bottom</td>
<td>1</td>
<td>0</td>
<td>-3</td>
<td>20.799</td>
<td>0.324</td>
<td>185.227</td>
<td>4.559</td>
<td>858577.70</td>
</tr>
<tr>
<td>Write</td>
<td>1</td>
<td>1</td>
<td>-2</td>
<td>28.296</td>
<td>-0.294</td>
<td>134.394</td>
<td>14.779</td>
<td>1828862.88</td>
</tr>
<tr>
<td>Read</td>
<td>0</td>
<td>-2</td>
<td>2</td>
<td>21.941</td>
<td>-0.914</td>
<td>353.884</td>
<td>15.792</td>
<td>2483557.88</td>
</tr>
<tr>
<td>Select(T7)</td>
<td>-1</td>
<td>-3</td>
<td>23.831</td>
<td>-0.853</td>
<td>77.588</td>
<td>3.459</td>
<td>1874768.88</td>
<td></td>
</tr>
<tr>
<td>C2t(F10)</td>
<td>1</td>
<td>2</td>
<td>2</td>
<td>26.573</td>
<td>-0.343</td>
<td>178.980</td>
<td>44.749</td>
<td>1319781.88</td>
</tr>
<tr>
<td>Store(F10)</td>
<td>-1</td>
<td>-2</td>
<td>28.382</td>
<td>-0.841</td>
<td>18.112</td>
<td>68.196</td>
<td>1951867.88</td>
<td></td>
</tr>
<tr>
<td>Faste(F10)</td>
<td>0</td>
<td>-1</td>
<td>-11</td>
<td>55.964</td>
<td>28.840</td>
<td>182.999</td>
<td>319.172</td>
<td>28391.76</td>
</tr>
<tr>
<td>14 ARL</td>
<td>0</td>
<td>0</td>
<td>11</td>
<td>55.965</td>
<td>28.830</td>
<td>202.999</td>
<td>48.851</td>
<td>38861.94</td>
</tr>
<tr>
<td>15 ARL</td>
<td>0</td>
<td>-1</td>
<td>11</td>
<td>55.944</td>
<td>28.823</td>
<td>130.632</td>
<td>139.922</td>
<td>267727.57</td>
</tr>
<tr>
<td>16 ARL</td>
<td>0</td>
<td>-1</td>
<td>-11</td>
<td>55.965</td>
<td>28.846</td>
<td>318.637</td>
<td>48.185</td>
<td>25401.74</td>
</tr>
<tr>
<td>17 ARL</td>
<td>1</td>
<td>-4</td>
<td>-7</td>
<td>55.085</td>
<td>27.983</td>
<td>67.557</td>
<td>346.926</td>
<td>8387.69</td>
</tr>
<tr>
<td>18 ARL</td>
<td>-1</td>
<td>4</td>
<td>7</td>
<td>55.851</td>
<td>27.927</td>
<td>247.557</td>
<td>13.874</td>
<td>9188.54</td>
</tr>
<tr>
<td>19 ARL</td>
<td>1</td>
<td>4</td>
<td>7</td>
<td>55.947</td>
<td>27.930</td>
<td>227.538</td>
<td>39.916</td>
<td>18891.88</td>
</tr>
<tr>
<td>20 ARL</td>
<td>1</td>
<td>4</td>
<td>-7</td>
<td>55.849</td>
<td>27.936</td>
<td>163.793</td>
<td>345.394</td>
<td>8430.32</td>
</tr>
</tbody>
</table>

Crystal: ylid, Extended w-x & w-20/x limits: p1_def
Move down one page (FpOn key)

2-Theta: 0.00
Omega: 0.00
Phi: 0.00
Chi: 0.00
Shutter: CLOSED

Fig. 2-10: Reflection array VIEW/EDIT menu
2.9 LOGFILE

You can create a logfile to save a detailed listing of your sample analysis progress. If you want to ask questions of Siemens personnel about XSCANS, it is useful to have the logfile which tells exactly which steps were performed.

To start logging:

1. Select LOGFILE from the COMMANDS menu (Fig. 2-11).

```
Fig. 2-11: LOGFILE option
```

2. Press ENTER.

3. Select NEWFILE (Fig. 2-12) and press ENTER.
The LOGFILE Options input panel appears (Fig. 2-13). The logfile has an extension of _LG. For other options, see Chapter 2 of the XSCANS Technical Reference Manual.
2.10 Screen Dumps

If your host computer is attached to a laser printer, press **CTRL/P** to print the displayed screen. The PRINT Options input panel appears (Fig. 2-14). The Output Print Filename is usually **SAXI$$PRINTER**.

![PRINT Options screen](image)

Fig. 2-14: PRINT Options screen

Press **ENTER** to print the current screen. If you do not have a laser printer, you can direct the output to a file. In this case, enter the filename into the Output Print Filename field and press **ENTER**. You can then transfer this file to a computer with a laser printer. For other PRINT options, see the *XSCANS Technical Reference Manual*. 
2.11 Interrupting Program Execution

If XSCANS is performing an operation (for example, centering or data collection), press
CTRL/BREAK to interrupt the operation. XSCANS automatically saves enough information to let
you restart the procedure at an appropriate place (for example, at the start of the current reflection
during data collection).
2.12 Exiting XSCANS

Follow this procedure to exit an XSCANS session and return to the DOS prompt:

1. Interrupt execution, if necessary, by pressing **CTRL/BREAK**.

2. Return to the COMMANDS menu by pressing **ESC** until it appears onscreen.

3. Save the parameter file if necessary. This is strongly recommended if you want to continue with the analysis later.

4. Select EXIT and press **ENTER** (Fig. 2-15).

![EXIT Menu](image)

*Fig. 2-15: EXIT option*

XSCANS displays this message (Fig. 2-16):

Exit: Are you sure? Press ENTER to exit, ESC to continue
5. Press **ENTER**. XSCANS displays this message (Fig. 2-17):

   **Save Parameters?** Press **ENTER** to save, **ESC** to abandon

Press **ENTER** to save the current parameter settings in the .P4P parameter file on disk. Press **ESC** to not save the parameters. In either case, the DOS prompt appears on the screen.
3 Basic P4 Operations

3.1 Goniometer Control

Many XSCANS commands control instrument hardware functions. The most basic operations are accessed through the GONIOMETER menu. To see the GONIOMETER menu, highlight the GONIOMETER command (Fig. 3-1) and press ENTER.

![Goniometer Control Menu](image)

Fig. 3-1:  GONIOMETER option

The GONIOMETER menu appears (Fig. 3-2). Its options are discussed in the following sections.
3.1.1 Shutter and Attenuator Control

SHUTTER is an open/close toggle command. Select SHUTTER when the shutter is open to close it. Select SHUTTER when the shutter is closed to open it. To use this option, select SHUTTER and press ENTER.

**Note:** The shutter opens only if the enclosure doors are closed and the interlock indicator is green.

The attenuator (if present) can be inserted or removed in a similar way by selecting ATTENUATOR.

3.1.2 Verifying and Updating Angles

Although the P4 controller is designed to keep track of the angle settings for your instrument, you should verify that the actual angular settings and the host computer are in agreement. You normally need to do this only when you have just loaded XSCANS.

To drive all axes to 0°, follow these steps:

1. Select ZERO (Fig. 3-3) and press ENTER twice.
2. Check the actual settings of the goniometer axes by reading the goniometer scales for all four axes (Fig. 3-4).

Fig. 3-4: 4-circle axes scales

3. If any of the scale readings are not zero, select UPDATE (Fig. 3-6) and press ENTER.
Fig. 3-5: UPDATE option

The GONIOMETER Options input panel appears (Fig. 3-6).

4. Input the correct angles (as read from the scales) and press ENTER.
5. Drive all the axes to 0° and verify the actual settings.
3.1.3 Driving Axes

1. Select DRIVE (Fig. 3-7) and press ENTER.

The GONIOMETER Options input panel appears (Fig. 3-6).

2. Enter the angles to which you want the diffractometer to drive and press ENTER. The diffractometer drives the axes to the indicated angles.

3.1.4 Manual Mode

To use MANUAL mode, select MANUAL from the GONIOMETER menu (Fig. 3-9) and press ENTER.
The GONIOMETER Options input panel appears (Fig. 3-9).

If these parameters are satisfactory (see Chapter 8 of the XSCANS Technical Reference Manual), press ENTER again. You can drive the diffractometer with the manual control box (Fig. 3-10). The buttons are: AXIS PRINT, PHI, CHI, 2THETA, OMEGA, SLOW FORWARD, SLOW REVERSE, FAST FORWARD and FAST REVERSE:
• Press one of these buttons to drive the selected axis in the indicated direction at the indicated speed.

**CAUTION**

CAUTION: Manual movement of the axes is not fully software-protected. Equipment damage can occur (especially glassware breakage) if you are not careful of hardware collisions.

• Release the button to stop the drive motor.
• Press the **AXIS PRINT** button to display the current goniometer angles onscreen.

(Control Box Layout A)

(Control Box Layout B)

*Fig. 3-10: Manual control box*
In MANUAL mode, you can enter four commands through the keyboard:

**S**  Toggle the shutter (open and close)

**A**  Toggle the attenuator (insert and remove)

**C**  Start a stationary count

**CTRL/G**  Exit from MANUAL mode to GONIOMETER mode.

When you are finished driving the goniometer manually, press **CTRL/G**.
3.2 Generator High Voltage and Current Settings

Generator kV and mA can be set from the control panel on the generator (see the *P4 Hardware Manual* for instructions on how to adjust these settings). For newer systems with K710D or K760D X-ray generators, kV and mA can be set from within the program (see the *XSCANS Technical Reference Manual, Section 8*).
3.3 Enclosure Doors

For safety reasons, the diffractometer cannot open the shutter unless all the access doors are closed and the interlock indicator is green. On some enclosures, you may need to push a SHUTTER RESET button. Be sure these conditions are satisfied before using any of the XSCANS automatic procedures.
4 YLID Data Collection

4.1 Introduction

Commonly used XSCANS procedures are described in the following sections using the YLID crystal supplied with your instrument. The complete chemical name for the YLID crystal is 2-dimethylsulfuranylidene-indan-1,3-dione. Appendix A YLID Test Crystal has the chemical and crystallographic data for the YLID crystal.

Even if you are experienced in using modern diffractometers, we recommend you follow these sections in detail to gain a good understanding of how XSCANS is organized. You can then efficiently collect data on your own samples.
4.2 Interactive and Automatic Methods

There are three basic methods of data collection:

**INTERACTIVE method**  Proceeds in a step-by-step fashion. At the end of each step, you can analyze the results, then make decisions concerning which steps to perform next. At the beginning of each step, you can set parameters to be used for that step.

**AUTOMATIC method**  Lets you set certain parameters at the beginning, then start the AUTOMATIC procedure. XSCANS performs the various steps and chooses intermediate parameter values. AUTOMATIC can proceed automatically through data collection, data reduction, space group determination and structure solution.

**AUTOSCRIP\_T method**  Lets you customize your own data collection procedures. For more information, see Section 4 of the XSCANS Technical Reference Manual.

Whether you use INTERACTIVE, AUTOMATIC or AUTOSCRIP\_T, we recommend that you always follow the steps outlined in Sections 5–7. After you have taken and analyzed the FULL ROTATION photograph, you can choose whether to use AUTOMATIC (Section 24), INTERACTIVE (Sections 8–23), or some combination. If you choose INTERACTIVE, you have great flexibility in deciding which steps to use. The next sections follow the flow chart diagram shown in Fig. 4-1.

*Note:* Where several procedures in the flow chart are on the same horizontal line, you normally choose just one of those procedures.

In the various procedures, we only mention those parameters you might need to modify. For a complete listing of parameters and a description of the algorithms used for each procedure, see the XSCANS Technical Reference Manual. After you have become more experienced with XSCANS, you do not need to examine all the menus and input panels listed in these sections, or to set so many parameters. You can set most of the parameters for the way you normally collect data, then save them in the P4_DEF.P4P file and the P4_DEF.RED file. Then, for each new sample, you only need to change a few parameters. See Section C.3.1 of the XSCANS Technical Reference Manual for details about saving the P4_DEF.P4P file and Section 12.7 of the XSCANS Technical Reference Manual for details about saving the P4_DEF.RED file.
Fig. 4-1: XSCANS data collection flow chart
5 Preliminary Operations

5.1 Verifying Goniometer Angle Settings

To verify that the instrument controller and host computer agree on the current angle readings (Section 3.1.2 Verifying and Updating Angles), follow these steps:

1. Start XSCANS as described in Section 2 Getting Started.

2. Highlight GONIOMETER (Fig. 3-1) and press ENTER.

3. Highlight ZERO on the GONIOMETER menu (Fig. 3-2) and press ENTER.
   The following message appears at the bottom of the screen:
   Will drive all axes to zero — Press ENTER to continue, ESC to EXIT
   Press ENTER.

4. Read the four physical angle scales (Fig. 3-3) to verify that all angles are at 0°.

5. If any of the angles are not 0°, use UPDATE on the GONIOMETER menu (Figs. 3-5 and 3-6).
   Repeat steps 2 through 4 until all angle scales read 0°.
5.2  Loading Default File Parameters

Each time a new specimen is mounted on the instrument, a new parameter file specific to the sample must be created. This is usually done by starting with the default parameter file, then making the appropriate changes to suit the sample:

1. Select LOAD from the COMMANDS menu (Fig. 2-5) and press ENTER.

2. The Output Parameters for Data Collection input panel is displayed (Fig. 2-6). Set Configuration Filename to P4_DEF and press ENTER.
5.3 Editing CRYSTAL Parameters

The CRYSTAL parameters menu lets you record the physical and chemical properties of your crystal.

1. Select CRYSTAL from the COMMANDS menu (Fig. 5-1) and press ENTER.

![CRYSTAL Options Input Panel](image)

*Fig. 5-1: CRYSTAL option*

2. Edit the parameters on the CRYSTAL Options input panel. Select a unique Parameter And Data Filename Root for each sample mounted. This is the CRYSTAL_ID name for all files generated for the sample.

   **Note:** In this manual, the CRYSTAL_ID names YLID and YLID1 are used. However, you normally use just one CRYSTAL_ID name for a given sample.

Set Descriptive Title and Chemical Formula. Enter the chemical formula as the empirical formula for the compound. For each element, use the conventional element symbol. Enter the first character as a capital letter and the second, if present, as a lower case letter (for example: C, Br, O, Li). Follow the element character(s) with the number of atoms (if greater than 1) of that chemical element in the compound. Do not include spaces in the chemical formula. The chemical formula for the YLID test crystal is \( \text{C}_{11}\text{H}_{10}\text{O}_{2}\text{S} \) (Fig. 5-2).
Fig. 5-2: CRYSTAL Options input panel

3. Enter the remainder of the crystal information (Fig. 5-3). Leave question marks for items for which you have no information.

Fig. 5-3: CRYSTAL Options input panel

4. Press ENTER.
5.4 Saving the Modified Parameter File

To write the modified parameter file to disk, select SAVE from the COMMANDS menu and press "ENTER" twice. A .P4P file with the new CRYSTAL_ID name is saved on the hard disk (see Section 2.7 Saving the Parameter File).
6 Optical Alignment

6.1 Mounting the Goniometer Head

The YLID test crystal supplied by Siemens is a pale yellow sphere approximately 0.4 mm in diameter, glued to the end of a small glass fiber. This fiber is secured in a 0.125" brass mounting pin, which in turn is mounted on a standard XYZ goniometer head (Fig. 6-1). This type of crystal mount is fairly common for air-stable materials for which room temperature measurements are to be performed.

**Note:** If your YLID crystal is mounted on a goniometer head with arcs (a eucentric goniometer head), set the arcs to zero before proceeding.

![Goniometer head detail](image)

**Fig. 6-1:** Goniometer head detail

Follow these steps to mount the goniometer head:

1. Unscrew the cover of the goniometer head storage container and remove the goniometer head.

2. Open the enclosure’s sliding glass doors and mount the goniometer head on the base of the goniometer. Be careful not to touch either the microscope or the collimator when mounting the goniometer head. YLID test crystals are difficult to replace.

**Note:** The base of the goniometer head is threaded and contains a positioning notch. When mounting the goniometer head, be sure the notch lines up with the positioning pin on the goniometer base (Fig. 6-2). The positioning pin is located at the back of the goniometer when all angles are at 0°. You may need to zero all angles before mounting the goniometer head. See Section 5.1 Verifying Goniometer Angle Settings.
3. Securely tighten the collar on the goniometer head base. Be sure not to over-tighten it.

**CAUTION**

EQUIPMENT DAMAGE: Do not use tools. Excessive force could damage the goniometer assembly.
6.2 Aligning the Sample

After the goniometer head containing the sample has been mounted on the goniometer, use the translational adjustments on the goniometer head (Fig. 6-1) to position the specimen so its center of mass is in the center of the goniometer. Follow these steps to optically align the sample (see Section 8 of the XSCANS Technical Reference Manual for more information):

1. Using the small allen wrench provided with the goniometer head, loosen the three screws that lock the goniometer head translations (Fig. 6-1).

2. Highlight OPTICAL on the GONIOMETER menu (Fig. 6-3) and press ENTER.

   ![Goniometer Interface]

   Fig. 6-3: OPTICAL option

3. Verify that the microscope base offsets (Fig. 3-9) are correct for your instrument (for example, base $\phi=30.0^\circ$ and base $\chi=330.0^\circ$ for the standard P4 diffractometer).

4. Press ENTER to initiate the OPTICAL ALIGNMENT functions.

   The OPTICAL ALIGNMENT routine activates five of the buttons on the manual control box (Fig. 3-10). In OPTICAL ALIGNMENT, the buttons have the following functions:
   
   - **A** = PHI: Toggle between positions 1 and 2
   - **B** = CHI: Toggle between positions 3 and 4
   - **C** = 2THETA: Toggle between positions 5 and 6
   - **D** = OMEGA: Toggle between positions 7 and 8
   - **T** = AXIS PRINT: Toggle switch—rotate phi by 180°
The following table gives the angle settings for the eight positions for the standard P4 system:

<table>
<thead>
<tr>
<th>BUTTON</th>
<th>POSITION</th>
<th>$\phi$</th>
<th>$\psi$</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>1</td>
<td>30°</td>
<td>330°</td>
</tr>
<tr>
<td>A</td>
<td>2</td>
<td>210°</td>
<td>330°</td>
</tr>
<tr>
<td>B</td>
<td>3</td>
<td>120°</td>
<td>330°</td>
</tr>
<tr>
<td>B</td>
<td>4</td>
<td>300°</td>
<td>330°</td>
</tr>
<tr>
<td>C</td>
<td>5</td>
<td>330°</td>
<td>150°</td>
</tr>
<tr>
<td>C</td>
<td>6</td>
<td>150°</td>
<td>150°</td>
</tr>
<tr>
<td>D</td>
<td>7</td>
<td>60°</td>
<td>150°</td>
</tr>
<tr>
<td>D</td>
<td>8</td>
<td>240°</td>
<td>150°</td>
</tr>
</tbody>
</table>

5. Press the A button, then the T button to drive to position 1.

6. Make a coarse adjustment of the X (horizontal) and Z (vertical) translations on the goniometer head to position the sample approximately in the center of the microscope. If the sample is out of focus, use the Y translation (along telescope axis) to adjust it. Observe the location of the crystal with respect to the horizontal and vertical crosshairs of the microscope. Use the crystal edges or the center of mass as a reference point.

7. Press the T button to drive to position 2. Observe the crystal’s location and compare it to the location for position 1. Adjust the X translation on the goniometer head and move between positions 1 and 2 until the sample remains in the same location with respect to the vertical crosshair at both positions.

Note: If the microscope is mis-aligned, it may not be possible to center the sample on the crosshairs in both positions. Use the crosshairs as a reference point and adjust the sample position so it is in the same position with respect to the crosshairs in both positions, even if the center of the sample does not correspond to the center of the crosshairs. Consult your system administrator to re-align the microscope so the center of the crosshairs corresponds to the center of the goniometer.

8. Press the B button, then the T button to drive to position 3. Observe the sample’s location.

9. Press the T button to drive to position 4. Observe the location of the sample and compare it to the location for position 3.

10. Adjust the Y translation on the goniometer head and toggle between positions 3 and 4 until the sample remains in the same location with respect to the vertical crosshair at both positions.

11. Press the A button, then the T button to drive to position 1. Observe the sample’s location.

12. Press the C button, then the T button to drive to position 5. Observe the sample’s location and compare it to the location for position 1. Adjust the Z translation on the goniometer head and toggle between positions 1 and 5 until the sample remains in the same location with respect to the horizontal crosshair at both positions.

13. Repeat steps 5 through 12 (if necessary) until the center of mass of the sample remains in the same place with respect to the crosshairs in all five positions. The crystal is now optically aligned and its center of mass should remain in the same place for all angle combinations.

14. Carefully tighten the locks on the goniometer head translations with a small allen wrench. After all translations have been locked, cycle through all positions to verify that the sample has not moved.

15. When the specimen alignment has been completed, type CTRL/G to exit from the OPTICAL ALIGNMENT routine.
7 Rotation Photograph

7.1 Taking a Rotation Photograph

There are three distinct tasks associated with taking a rotation photograph:
- Prepare the computer and hardware for the photograph
- Expose and develop the film
- Analyze the photograph

Follow these steps to take a rotation photograph:
1. Select PHOTO from the COMMANDS menu (Fig. 7-1) and press ENTER.

![COMANDS: X-ray Single Crystal Analysis System](image)

Fig. 7-1: PHOTO selection

The PHOTOGRAPH menu is displayed (Fig. 7-2).
2. Select ROTATION and press ENTER. The ROTATION PHOTO Options panel is displayed (Fig. 7-3).

3. Set 2Theta and Chi to 0.0°. If you are using a P4 diffractometer, set Crystal To Film distance to 19.50. If you are using a P4/RA, set Crystal To Film distance to 24.30.

4. Press ENTER. All angles are driven to 0 and the following message appears:
Mount Polaroid Cassette – Press ENTER to continue

Note: We recommend that you take all rotation photographs with a standard set of operating conditions (for example, 10 minute rotation with X-ray generator set at 50 kV and 40 mA). This lets you make qualitative comparisons of the relative diffracting power of samples. Later, generator power and data collection parameters can be adjusted to suit the sample.

5. Load the film into the film cassette:
   5a. Set the LOAD lever on the film cassette to LOAD.
   5b. Push the EXPOSE lever down (opposite the direction of the arrows).
   5c. Holding the film with “57” facing the same direction as the EXPOSE lever, insert the film into the holder (metal clip first).
   5d. Push the film all the way in until it touches the bottom of the cassette.
   5e. Pull the paper sleeve of the film up until it stops. The paper sleeve comes about 2/3 of the way out of the cassette before it catches.
   5f. Pull the EXPOSE lever up on the film holder in the direction of the arrows on the lever.

6. Mount the film cassette:
   6a. Open the enclosure cabinet doors to access the goniometer.
   6b. Loosen the thumb screw on the detector mount and slide the mount and detector back to the CLEAR mark on the 2θ arm (Fig. 7-4).

   ![Diagram](image)

   **Fig. 7-4:** Detector CLEAR position

   6c. Slide the film holder onto the detector mount (Fig. 7-5) by pushing the film cassette assembly over the V-shaped dovetail. Do not touch the beam stop or the collision switches.
6d. Slide the detector mount (with the cassette) forward to the PHOTO position (Fig. 7-6) and tighten the thumb screw.

6e. Close the enclosure’s sliding glass doors. Press the RESET button on the generator safety module. Be sure the green interlock light comes on.

7. Expose the rotation photograph:

7a. Be sure the X-ray generator is turned on and set to a normal power level (for example, 50 kV, 40 mA). Press ENTER. The shutter opens, the BEAM indicator lights and the Φ axis starts. The following message appears at the bottom of the screen:

Taking Rotation Photograph...

The photograph is exposed for the amount of time specified on the ROTATION PHOTO Options input panel.
8. Remove the film cassette:

8a. When the exposure of the rotation photograph is completed, the BEAM indicator light turns off and the following message is displayed:

Dismount Polaroid Cassette — Press ENTER to continue

8b. Open the enclosure's glass doors. Loosen the detector mount thumb screw and slide the detector back to the CLEAR position.

8c. Remove the film cassette from the detector mount by pulling up on the film cassette assembly. Do not touch the beam stop or the collision switches. Move the detector back to the POST position (Fig. 7-7).

Fig. 7-7: Detector post position

8d. Close the enclosure's sliding glass doors and press the RESET button on the generator safety module. Be sure the green interlock light comes on.

9. Develop the rotation photograph:

9a. Push the EXPOSE lever on the film cassette down. Push the paper film sleeve into the film cassette until it touches the bottom.

9b. Move the LOAD lever on the back of the film cassette to the PROCESS position.

9c. Pull the film pack out of the film holder with one smooth, firm pull and wait 20 seconds for the film to develop. Apply fixative to the film if desired.
7.2 Analyzing the Rotation Photograph

Examine the rotation photograph carefully to determine whether or not the sample is suitable for further analysis. A good rotation photograph contains sharply defined spots of varying intensities (Fig. 7-8). Careful inspection of the rotation photograph for a given sample may indicate that the sample should be rejected without further use of instrument time.

Fig. 7-8: XSCANS rotation photograph
7.3 Rotation Photo Troubleshooting

The following examples illustrate typical problems encountered on rotation photographs:

Problem: The rotation photograph is totally black with no image of the beam stop or background scattering:
- The film was not properly loaded.
- The X-ray tube was not on.
- The shutter did not open (the safety interlock system prevents the shutter from opening if any cabinet door is not closed or the RESET button has not been pressed).
Solution: If a totally black photograph is produced, carefully retake the photograph while watching for these problems.

Problem: The photograph contains the image of the beam stop, but no spots:
- The sample has fallen off the goniometer head pin.
- The sample has dissolved in the glue.
- The sample has decomposed.
- The sample is amorphous (does not diffract).
Solution: Reject the sample.

Problem: The photograph contains spots, but they appear to be split (each reflection spot on the film seems to be doubled). This is often an indication that the sample is cracked.
Solution: Proceed with caution. Look for bad profiles or problems in centering and/or indexing the reflections.

Problem: The photograph contains spots, but they appear to be fuzzy (spots are not sharp and clearly defined):
- Poor crystal quality.
- Large mosaic spread.
- Severe disorder of molecules within the crystal.
Solution: Proceed with caution. Look for bad profiles or problems in centering and/or indexing the reflections.

Problem: The photograph contains a few spots, but they are weak. A weak rotation photograph indicates that the sample does not diffract well. This can be caused by:
- Small crystal size.
- Insufficient X-ray power.
Solution: Repeat the rotation photograph with higher power and/or a longer exposure time.

Problem: The photograph contains powder rings (in addition to the reflection spots, rings are also present on the photograph). Rings on the rotation photograph indicate that there is some powder present on or in the sample. This can be caused by a number of problems:
- The crystal is heavily coated with powder (poor sample preparation).
- The crystal is in a capillary and the rings are caused by the capillary liquid.
- The crystal has decomposed into a powder.
Solution: Reject the sample.
Problem: The photograph contains moderately strong spots, but they are all near the edges of the photograph. This situation indicates that the unit cell volume is small.

Solution: Increase Minimum 2Theta and Maximum 2Theta and decrease Minimum Axial Length in SEARCH PARAMETERS if you want to use INCREMENTAL, RANDOM or HEMISPHERE search methods. If you use Cu radiation on a crystal with a small unit cell, PHOTO search method finds reflections much faster than the other search methods.
8 Locating and Centering Reflections

After you have optically aligned the sample and determined that it is suitable for analysis based on the rotation photograph, you can locate a small subset of reflections from the complete diffraction pattern for unit cell determination. These reflections must be representative of the entire diffraction pattern (both weak and strong reflections, high and low angle reflections, etc.) and their positions must be accurately determined. They form the basis for crystallographic unit cell determination and calculation of a 3x3 orientation matrix relating the positions of all reflections in the diffraction pattern to the goniometer angles on the diffractometer.

XSCANS provides several different ways to obtain a small set of reflections for use in unit cell determination. Each of these methods has advantages and disadvantages, depending on the specific sample to be analyzed. All the methods work well for the YLID crystal:

- PHOTO COORDINATE search
- INCREMENTAL search
- RANDOM search
- HEMISPHERE search

Each of the methods is described in the following sections. You can use any or all of the techniques for the YLID crystal.
8.1 Photo Coordinate Search Method

The rotation photograph provides important information which can be used to locate reflections on the diffractometer. Regardless of the specimen or its orientation on the goniometer, any full rotation photograph exhibits both horizontal and vertical symmetry planes. Every reflection produces four spots on the photograph (except for a few spots obscured by the arm of the beam stop). It is always possible to measure several sets of 2X and 2Y coordinates using the Axial Photograph overlay. These 2X and 2Y measurements, combined with a knowledge of the film-to-crystal distance, lets the instrument locate and center reflections.

In many cases, the PHOTO search method is faster to obtain an initial set of reflections, especially when Cu radiation is used and/or the sample has a small unit cell.

1. Using the Axial Photograph overlay provided with your system, measure 10 to 15 sets of 2X and 2Y values (in centimeters) from the rotation photo. To measure coordinates:

   1a. Orient the photo vertically.

   1b. Lay the Axial Photograph overlay on the photo (Fig. 8-1).

   1c. Select a reflection with good intensity in the upper left quadrant of the photo.

   1d. Place the 0,0 intersection of the overlay on the selected reflection. Measure horizontally from that point across to its mirror image in the upper-right quadrant. This is the 2X value for that reflection—write it down.

   1e. Measure vertically from the point to the mirror image in the lower-left quadrant. This is the 2Y value for the reflection—write it down.

   1f. Measure the rest of the selected reflections. The reflections should be a representative sampling of 2θ (some of the reflections should be close to the center of the photo (low 2θ) and some should be near the edge of the photo (high 2θ)). The reflections should
be a representative sampling of $\chi$ (some of the reflections should have a large 2X and a small 2Y (low $\chi$) and some should have a small 2X and a large 2Y (high $\chi$)). Choose both weak and strong reflections.

2. Select REFL\_ARRAY from the COMMANDS menu (Fig. 8-2) and press ENTER.

3. Select EDIT from the REFL\_ARRAY menu (Fig. 8-3) and press ENTER.
4. The reflection array input panel is displayed. Type P, followed by the first 2X and 2Y values. The P, 2X and 2Y entries must be separated by spaces (Fig. 8-4).

![Reflection Array Input Panel](image)

Crystal: yld, Extended w/ X & w-2θ/X limits: p1_def
Edit text: Press left mouse button or ENTER for options.

Fig. 8-4: Reflection array input panel

5. Press ↓.

6. Enter all the 2X and 2Y values this way.

7. Use the arrows and BACKSPACE to edit any errors you make while entering this information. When you are finished, the array should look like Fig. 8-5.

   Note: The 2X and 2Y values are displayed in the 2Theta and Omega fields, respectively.
8 Locate and Center Reflections

Fig. 8-5: Reflection array example

8. Press ESC.

9. Select SEARCH (Fig. 8-6) and press ENTER.

Fig. 8-6: REFL_ARRAY menu

10. Set Search Type to Photo in the SEARCH parameters input panel (Fig. 8-7).
### Search Parameters

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Number of reflections to find</td>
<td>25,000</td>
</tr>
<tr>
<td>Search type</td>
<td>PHOTO</td>
</tr>
<tr>
<td>Minimum 2-Theta</td>
<td>21.000</td>
</tr>
<tr>
<td>Minimum Chi</td>
<td>15.000</td>
</tr>
<tr>
<td>Maximum 2-Theta</td>
<td>56.010</td>
</tr>
<tr>
<td>Maximum Chi</td>
<td>36.000</td>
</tr>
<tr>
<td>Minimum Intensity</td>
<td>10000.000</td>
</tr>
<tr>
<td>Omega, 2-Theta speed</td>
<td>300.000</td>
</tr>
<tr>
<td>Phi, Chi speed</td>
<td>300.000</td>
</tr>
<tr>
<td>2-Theta Increment</td>
<td>1.500</td>
</tr>
<tr>
<td>Chi Increment</td>
<td>2.500</td>
</tr>
<tr>
<td>Phi Increment</td>
<td>1.000</td>
</tr>
<tr>
<td>Minimum axial length</td>
<td>5.000</td>
</tr>
<tr>
<td>Maximum axial length</td>
<td>35.000</td>
</tr>
</tbody>
</table>

**Crystal:** q1ld, Extended w/X & w-2θ/u limits: pt_def

**Fig. 8-7:** SEARCH parameters input panel

Of the other parameters in SEARCH parameters, only Minimum Intensity is important for a PHOTO search. The default of 10,000 counts is sufficient in most cases, but can be decreased for weakly diffracting crystals. When ENTER is pressed, XSCANS finds and determines the centers for the list of reflections you entered. The algorithms for searching and centering are described in **Section 13.14.1** of the **XSCANS Technical Reference Manual**. After each reflection is found and centered, an Omega profile is displayed onscreen (Fig. 8-8).
11. When all reflection centers have been determined, choose EDIT from the REFL_ARRAY menu and press ENTER. The reflection array looks like Fig. 8-9.

The flags changed from P (Photo coordinates) to ARL (Refined Angle coordinates, which can be used as input for indexing and least squares). The 2X and 2Y coordinates changed to 2θ, ω, φ and χ angles. Integrated intensities (normalized to 1°/minute) are displayed under Inorm.
8.2 INCREMENTAL Search Method

Instead of measuring 2X and 2Y coordinates from the full rotation photograph, you can use INCREMENTAL search.

1. Choose SEARCH from the REFL ARRAY menu (Fig. 8-3) and press ENTER.
2. Change Search Type to INCR on the SEARCH parameters input panel (Fig. 8-10).

![SEARCH parameters input panel (INCR selected)](image)

INCREMENTAL search automatically searches for and determines the centers of reflections with 2θ and χ values in the ranges bounded by the minimum and maximum values of these parameters (ω is always set to one half the value of 2θ, and ψ has the range 0.0° to 360.0°). If the full rotation photograph (Fig. 8-11) shows few reflections near the center of the film, you may have to increase Minimum 2Theta and Maximum 2Theta for the search range. The search continues until Number Of Reflections To Find are found or the search range is exhausted.

3. Press ENTER to begin searching and centering. The final result is a list of centered reflections (Fig. 8-9).

**Note:** The angular range for the reflections corresponds to those set in SEARCH parameters.
8.3 RANDOM Search Method

RANDOM search is the same as INCREMENTAL search, except the pattern of searching in 2θ and χ are random, not systematic. Set Search Type to RANDOM in SEARCH parameters (Fig. 8-11).

![Scan Statistics]

Fig. 8-11: SEARCH parameters input panel (RANDOM selected)

Press ENTER to begin searching and centering. The result is a centered reflections list (Fig. 8-9).

Note: The angular range for the reflections corresponds to those set in SEARCH parameters.
8.4 HEMISPHERE Search Method

To use HEMISPHERE search, follow these steps:

1. Choose SEARCH from the REFL_ARRAY menu (Fig. 8-3) and press ENTER.

2. Set Search Type to HEMIS on the SEARCH parameters input panel (Fig. 8-12).

![SEARCH parameters input panel (HEMIS selected)](image)

HEMISPHERE search picks one centered reflection from the reflection array or finds one reflection using INCREMENTAL search. It then searches for reflections that are close to that reflection (within a hemisphere in reciprocal space). If the full rotation photograph shows too few reflections near the center, you may need to increase Minimum 2 Theta and Maximum 2 Theta and decrease Minimum Axial Length.

The INCREMENTAL, RANDOM, and HEMISPHERE searches try to automatically index the reflections and determine the unit cell parameters as they find and center reflections, unlike PHOTO Search. See Sections 9-10 for INDEX and LEAST SQUARES procedures.

The search ends after 10 reflections have been found where no significant unit cell parameter changes are produced, or when the region to search has been completed.

3. Press ENTER to begin searching and centering. The final result is a list of indexed centered reflections (Fig. 8-9). These reflections have indices assigned to them and the H flag has been added to the Flag field.

**Note:** If you used HEMISPHERE search, skip to Section 11 Determining Unit Cell Size.
Determining Reflection Indices

If you used PHOTO, INCREMENTAL or RANDOM search, you need to assign indices to the centered reflections in the reflection array.

1. From the REFL_ARRAY menu, choose INDEX (Fig. 9-1).

The AUTOINDEXING input panel appears (Fig. 9-2).
2. Press ENTER (you do not need to change any of these parameters). After a few seconds, the autoindexing output appears onscreen (Fig. 9-3). For the YLID crystal, the reduced primitive cell parameters are close to those shown in Fig. 9-3.

3. Press PGUP/PGDN to move through the autoindexing output. The reflection array now contains a list of centered reflections with indices (Fig. 9-4).
### Fig. 9-4: Reflection array

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<tr>
<th>Flags</th>
<th>H</th>
<th>K</th>
<th>L</th>
<th>2Theta</th>
<th>Omega</th>
<th>Phi</th>
<th>Chi</th>
<th>Intens</th>
</tr>
</thead>
<tbody>
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<td>A1L</td>
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<td>0</td>
<td>-2</td>
<td>9.626</td>
<td>0.299</td>
<td>290.826</td>
<td>41.474</td>
<td>723437.08</td>
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<td>1948822.08</td>
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<td>0.294</td>
<td>185.739</td>
<td>31.849</td>
<td>427360.08</td>
</tr>
<tr>
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<td>1</td>
<td>-3</td>
<td>17.481</td>
<td>0.388</td>
<td>243.947</td>
<td>34.643</td>
<td>715757.60</td>
</tr>
<tr>
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<td>-1</td>
<td>1</td>
<td>18.462</td>
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<td>26.573</td>
<td>-0.343</td>
<td>179.980</td>
<td>44.749</td>
<td>1319781.80</td>
</tr>
</tbody>
</table>

Crystal: gilid, Extended w/K & w-20/K limits: pl, def

Edit text: Press left mouse button or ENTER for options.
10 LEAST SQUARES

10.1 Determining Least Squares Unit Cell Parameters

The reduced lattice cell parameters generated by autoindexing are based on angles for only three centered reflections. To obtain better lattice parameters and a good orientation matrix, which contains lattice parameter and crystal orientation information, use all the angle information for all the centered reflections. LEAST SQUARES is used to obtain these results. Choose LEAST SQUARES from the REFL ARRAY menu (Fig. 10-1) and press ENTER.

![Scan Statistics and Crystal Information](image)

---

Fig. 10-1: LEAST SQUARES

The LEAST SQUARES Options panel appears (Fig. 10-2).
Fig. 10-2: LEAST SQUARES input panel

Set Least Squares Constraint Type to A (triclinic). Because the centered reflections are all measured with the detector on the positive 2θ side, set Constraint Mask to T (hold the 2θ zero fixed). Press ENTER. The Least squares output looks like Fig. 10-3.

Press PGUP/PGDN to move through the least squares output. Notice the 2θ, ω and χ zeros and the crystal translations and their standard deviations. In Fig. 10-3, the indication is that the crystal
should be translated in the X-direction by 0.6 small microscope divisions. Because the next procedures use the crystal position as determined by LEAST SQUARES, you do not need to manually adjust the crystal. See Section 13.10.1 of the XSCANS Technical Reference Manual for a detailed description of the output. The lattice parameter angles are now much closer to 90.0° than they were after autoindexing (Fig. 9-3).

Press **PGUP**/**PGDN** to move through the least squares output to see how well the various centered angles of the reflections fit the least squares mathematical model.
11 Determining Unit Cell Size

You must determine if the unit cell size needs to be doubled, tripled, etc. The reduced cell parameters were determined from a small number of reflections (10 to 25). Because there are so few reflections, the reflections may be special. For example, all the reflections found may have h-indices equal to 2n (the h indices are all even). In this case, autoindexing gives a unit cell whose volume is only half of its true value. To test for this possibility, there are two procedures available. Normally, you only need to use one of these procedures.

11.1 Axial Photographs

Follow these steps to take an axial photograph:

1. From the COMMANDS menu, choose PHOTO and press ENTER. The PHOTOGRAPH menu appears (Fig. 11-1).

2. Choose AXIAL and press ENTER.

3. Set Rotation Axis to 1 (a axis) (Fig. 11-2) and press ENTER.
Fig. 11-2: AXIAL PHOTO input panel

Use the procedures described in steps 4 through 8 of Section 7.1 Rotation Photograph to take a Polaroid photograph. The resulting photograph looks like Fig. 11-3.

Fig. 11-3: Axial photograph
Repeat this procedure with Rotation Axes set to 2 (b axis) and 3 (c axis). The resulting photographs look like Figs. 11-4 and 11-5.

Fig. 11-4: Axial photograph example

Fig. 11-5: Axial photograph example

4. Using the Axial Photograph overlay provided with your system, measure the axial lengths on these three films (Fig. 11-6).
Use the appropriate scale (Cu or Mo) to make these measurements. The scales on the Axial Photograph overlay were prepared assuming a Crystal To Film distance of 19.50 cm (see Section 7.1 Rotation Photograph). If this is not the correct distance (for example, you are using a P4/RA system), you can measure the distance between the layers on the axial photographs with the centimeter scale.

4a. Select AXIAL LENGTHS from the PHOTOGRAPH menu (Fig. 11-7) and press ENTER.

Fig. 11-6: Mo measurement example

Fig. 11-7: PHOTOGRAPH menu
4b. Enter the cm reading (Fig. 11-8) and press ENTER. The corresponding axial length is displayed.

For the YLID crystal, the measured values for the three photographs should be approximately 6.0, 9.0 and 18.4 Å, respectively. These values should match the values from the LEAST SQUARES calculation. If any of these values are different by a factor of two, three, etc., the corresponding axial length and corresponding indices for the centered reflections must be doubled, tripled, etc. If such a situation occurs, proceed with FRACTIONAL search as described in Section 11.2 Search for Reflections with Fractional Indices.

In Figs. 11-3, 11-4, and 11-5, you can observe a horizontal line of symmetry (every reflection appearing above the horizontal line is at the same distance and has the same intensity as a reflection below the horizontal line).

**Note**: There may be a few exceptions (due to the beam stop position, etc.).

The line of symmetry indicates that each of the corresponding axes has a twofold symmetry axis coincident with that axis. Because the YLID crystal is orthorhombic, three twofold symmetry axes must be present. Other crystals may or may not show this symmetry. A better test for this kind of symmetry is discussed in Section 16 Determining the Laue Symmetry.

Occasionally (never for the YLID crystal) you may see a photograph where the spots do not lie on slightly curved layer lines (Figs. 11-3, 11-4, and 11-5). Instead, they seem to lie on diagonal lines. This indicates that the sample is a twinned crystal and it is probably best to reject the crystal and look for a better sample.

Sometimes some of the reflections are split. Again, this is an indication that the sample is twinned. Look for a better sample. In addition, there may be reflections that do not lie on the layer lines. If so, there is probably a small satellite crystal that is attached to the main crystal and it does not have the same orientation as the main part of the sample. If such reflections are moderately strong, reject the sample.
11.2 Searching for Reflections with Fractional Indices

Instead of using axial photographs, you can use FRACTIONAL search to determine whether the unit cell volume is to be increased.

1. Choose SEARCH from the REFL_ARRAY menu and press ENTER.

2. Set Search Type to FRACT (Fig. 11-9) and press ENTER.

![Figure 11-9: SEARCH parameters screen (FRACT selected)](image)

XSCANS begins searching for reflections with fractional parts of the indices equal to 0.3333, 0.5 or 0.6667 (Fig. 11-10). If a reflection is found, it is centered and indexed, and LEAST SQUARES is run to determine new lattice parameters. If the unit cell volume has changed significantly, FRACTIONAL search is run until no new reflections are found.
### XSCANS Users Guide

#### 11 Determine Unit Cell Size

---

**XSCANS: X-ray Single Crystal Analysis System**

**Copyright Siemens**

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<tr>
<th>2.0000</th>
<th>1.0000</th>
<th>2.0000</th>
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<td>3.0000</td>
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| A      | 5.961(8) |
| B      | 9.634(1) |
| C      | 18.301(1) |
| alpha  | 98.613(5) |
| beta   | 89.990(5) |
| gamma  | 98.986(5) |
| Volume | 989.0410 |

Crysta: ylid. Extended w/ X & w-29/X limits: pt_def

Searching...

**Fig. 11-10: Reflections search**
12 THIN SHELL Search

At this point, you can be reasonably sure that you have the correct reduced primitive cell. The lattice parameters, however, are probably not very accurate because the reflections chosen may have small 20 values and may all be from the same part of reciprocal space (especially if HEMISPHERE search was used).

Therefore, you need to find reflections that are well-distributed in reciprocal space at 20 values at about mid-range (25.0° for Mo radiation or 56° for Cu radiation). XSCANS searches for these reflections and centers on them if they are strong enough.
1. Choose SEARCH from the REFL_ARRAY menu and press ENTER.
2. Set Search Type to THIN (Fig. 12-1) and press ENTER.

![XSCANS: X-ray Single Crystal Analysis System](image)

**Table:**

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<th>Value</th>
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<td>Number of reflections to find</td>
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</tr>
<tr>
<td>Minimum 2-Theta</td>
<td>21.000</td>
</tr>
<tr>
<td>Minimum Chi</td>
<td>15.000</td>
</tr>
<tr>
<td>Maximum 2-Theta</td>
<td>56.010</td>
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<tr>
<td>Maximum Chi</td>
<td>30.000</td>
</tr>
<tr>
<td>Minimum Intensity</td>
<td>100000.000</td>
</tr>
<tr>
<td>Omega, 2-Theta speed</td>
<td>360.000</td>
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<tr>
<td>Phi, Chi speed</td>
<td>360.000</td>
</tr>
<tr>
<td>2-Theta Increment</td>
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</tr>
<tr>
<td>Phi Increment</td>
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<tr>
<td>Phi Increment</td>
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</tr>
<tr>
<td>Minimum axial length</td>
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</tr>
<tr>
<td>Maximum axial length</td>
<td>35.000</td>
</tr>
</tbody>
</table>

![Fig. 12-1: SEARCH parameters input panel (THIN selected)](image)

XSCANS finds and centers Number Of Reflections To Find with 20 values about equal to Maximum 2Theta.

3. Press ENTER. The reflections from the THIN SHELL search are added to the list of reflections already found (Fig. 12-2).
## Reflection Array (100 lines)

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<thead>
<tr>
<th>Flags</th>
<th>H</th>
<th>K</th>
<th>L</th>
<th>2Theta</th>
<th>Omega</th>
<th>Phi</th>
<th>Chi</th>
<th>Inten</th>
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<td>3</td>
<td>7</td>
<td>55.733</td>
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<tr>
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<td>343.123</td>
<td>315.670</td>
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<tr>
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<td>4</td>
<td>4</td>
<td>5</td>
<td>55.603</td>
<td>27.889</td>
<td>343.123</td>
<td>315.670</td>
<td>11887.27</td>
</tr>
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</table>

2-Theta: 55.65
Omega: 30.01
Phi: 343.12
Chi: 315.68
Shutter: C1002B

Fig. 12-2: Reflection array with THIN SHELL search
13 Precise Least Squares  
Unit Cell Determination

Now that you have obtained an increased number of centered reflections with THIN SHELL search, you can calculate precise unit cell parameters. Run LEAST SQUARES exactly as you did in Section 10 LEAST SQUARES:

1. Choose LEAST SQUARES from the REFL_ARRAY menu (Fig. 10-1) and press ENTER.

2. Set Least Squares Constraint Type to A (triclinic) (Fig. 10-2).

3. Set Constraint Mask to T (hold the 26 zero value fixed) and press ENTER. The result looks like Fig. 13-1, where the standard deviations of the unit cell parameters are lower than they were in Fig. 10-3. This added accuracy is due to the fact that there are now more reflections widely distributed in reciprocal space.

![Least Squares Output](image)

Fig. 13-1: LEAST SQUARES output
If your XSCANS system has a copy of the NIST Crystal Data Identification file on a CD-ROM, you can search this file to determine whether your crystal structure has been reported in the literature.

**Note:** You must create an index file for this version of the crystal data file. See the XSCANS Technical Reference Manual, Appendix E for information on how to create the index file.

The crystal data file consists of unit cell dimensions for all reported single crystal structures and all reported powder patterns that have been indexed.

1. Choose CDF-SEARCH from the REL_ARRAY menu (Fig. 14-1) and press ENTER. The unit cell dimensions of the reduced primitive cell appear in the CRYSTAL DATA FILE SEARCH options (Fig. 14-2).

   ![REFL_ARRAY menu](image)

   **Fig. 14-1: REFL_ARRAY menu**

2. Set Decimal Fraction Deviation to 0.02, although your measured unit cell parameters are much more accurate than 2%. You are trying to match your parameters with previous unit cell parameters that may have been measured much less accurately and possibly at a different temperature.

3. Search both the organic and inorganic parts of the crystal data file by setting Search Type to both. The output file contains all the structures whose unit cell dimensions are within Decimal Fraction Deviation (Fig. 14-2).
Fig. 14-2: CRYSTAL DATA FILE SEARCH options

4. Press ENTER. In a few seconds, the search is complete. Use PGUP/PGDN to move through the displayed results (Fig. 14-3).

Fig. 14-3: CDF-SEARCH output

The output file (HITS.OUT) contains additional information (literature references) and is sorted so that the best fits appear first. To print or view the output file using DOS commands, select
SYSTEM from the COMMANDS menu (see the XSCANS Technical Reference Manual). The contents of HITS.OUT are:

hit 1:  crystal data # = 518607 GOF = 0.0820 Record # = 2203422  
Reduced Unknown Cell: 5.959 9.034 18.381 90.01 90.01 90.00 989.58  
C11H1002S  
Reduced Crys Data cell: 5.949 9.012 18.331 90.00 90.00 90.00 982.77  
C11 H10 O2 S  
2-Dimethylsulfurylidene-1,3-dione  
Acta Crystallogr., Sect. B  
27 581 1971 Christensen, A.T., Thom, E.

hit 2:  crystal data # = G621171 GOF = 2.0785 Record # = 2213837  
Reduced Unknown Cell: 5.959 9.034 18.381 90.01 90.01 90.00 989.58  
C11H1002S  
Reduced Crys Data cell: 6.007 9.161 18.560 102.99 90.00 90.00 995.22  
C22 H22 O6  
(+)-7-(1-Benzoxethyl)-2-(p-methoxyphenoxy)-6,8-dioxabicyclo(3.2.1)oct-3-ene  
Chem. Lett.  

hit 3:  crystal data # = D591772 GOF = 2.1445 Record # = 2218489  
Reduced Unknown Cell: 5.959 9.034 18.381 90.01 90.01 90.00 989.58  
C11H1002S  
Reduced Crys Data cell: 6.035 9.073 18.486 90.00 90.00 102.83 986.94  
C11 H9 N3 O S  
2-(2-Benzothiazolyl)-1,2-dihydro-5-methyl-3H-pyrazol-3-one  
J. Heterocycl. Chem.  

hit 4:  crystal data # = 518376 GOF = 2.5525 Record # = 2188538  
Reduced Unknown Cell: 5.959 9.034 18.381 90.01 90.01 90.00 989.58  
C11H1002S  
Reduced Crys Data cell: 5.860 9.170 18.340 90.00 90.00 90.00 985.52  
C9 H9 N O2 S  
trans-3-(6-Methyl-2-pyridylthio)-propenic acid  
Acta Chem. Scand.  
26 1141 1972 Groth, P., Davidkov, K., Aasen, A.

The goodness-of-fit (GOF) is a measure of how well the experimental unit cell parameters fit specific entries in the Crystal Data file. The smaller the GOF, the better the unit cell parameters fit. As evident from these values, the first hit is clearly the correct one.
15 Bravais Lattice Determination

After the reduced primitive cell lattice parameters and the orientation of the crystal are known, you can collect all the data; however, if there is crystallographic symmetry in the unit cell, that may not be necessary. If crystallographic symmetry is present in the unit cell, there is symmetry in the intensities of the reflections. In this case, you only need to collect the unique portion of the data set.

To determine whether twofold, threefold, fourfold or sixfold axes are present, generate all possible Bravais lattices from the given reduced primitive cell parameters by following these steps:

1. Choose BRAVAIS from the REFL_ARRAY menu (Fig. 15-1) and press ENTER.

The unit cell dimensions and their standard deviations appear in the BRAVAIS LATTICE options input panel (Fig. 15-2). Values for two criteria are required.
2. Set Max Sigmas For Any Soln (the maximum root mean sum of squares of the weighted deviations for all displayed solutions) to 20.0.

3. Set Max Sigmas For Best Soln (the maximum root mean sum of squares of the weighted deviations for the best solution) to 6.0.

4. Press ENTER. The BRAVAIS LATTICE output looks like Fig. 15-3. All solutions that meet criterion (1) are displayed (press PGDN to see all the solutions) and the solution which meets criterion (2) and has the highest symmetry is chosen as the "best" solution by XSCANS. It is marked with >>>.
5. Press ESC. XSCANS displays the Solution # To Use screen (Fig. 15-4).

6. Select the Bravais lattice (set Solution Number To Use to 1 and press ENTER). If necessary, the indices in the reflection array are transformed to correspond to your selection (for the YLID crystal, no index transformation is required). The LEAST SQUARES calculations is automatically run again with Constraint Mask set to TOC.
16 Laue Symmetry Determination

BRAVAIS LATTICE indicates what symmetry might be present, but does not verify it. Check the intensities of many reflections before deciding what part of the data to collect. Choose LAUE from the REFL_ARRAY menu (Fig. 16-1) and press ENTER.

LAUE selects a few reflections from the reflection array and generates all the corresponding reflections which might be symmetry equivalent based on the selected Bravais lattice. Integrated intensities are measured for these reflections (Fig. 16-2).
Statistical tests are performed to determine whether the selected Bravais lattice or some lower symmetry type is the correct one (Fig. 16-3).

For YLID, Laue group number 3 (mmm symmetry) is selected. Tests for the 3 monoclinic Laue groups (Laue number 2) and the triclinic group (Laue number 1) are also made. Axial photographs can show whether mirror (m) symmetry is present. They cannot show whether threefold, fourfold, or sixfold symmetry is present. We recommend that you always perform the Laue symmetry tests.
17 Selecting Data Collection Parameters

You now have enough information to collect the intensities for all reflections. There are three methods of data collection: THETA/2THETA, OMEGA, and THETA/THETA. For each method, there are many parameters which control the course of the data collection. You can choose the data collection method and the parameter values to suit the crystal analysis requirements. The three methods are discussed in the XSCANS Technical Reference Manual.

The speed of data collection and the \( \theta \) range you select is dependent on the diffracting power of the crystal. The original rotation photograph gives a very crude estimate of this diffraction power. A better method is to use AUTOSPEED (see Section 18 Automatic Selection of Data Collection Parameters). Whether or not you use this procedure, examine and set parameters in all the data collection panels.

Parameters that affect data collection appear on the panels described in this section. For most of these parameters, choose the default. The values given in this section are appropriate for the YLID crystal; however, they may not be appropriate for other crystals. Before selecting and entering values for the data collection parameters, spend a few minutes reading the description of the data collection algorithms found in Section 5.13.1 of the XSCANS Technical Reference Manual, which provides you with a better understanding of the parameter functions.

To select the data collection parameters:
1. Choose COLLECT from the COMMANDS menu (Fig. 17-1) and press ENTER.

---

**Fig. 17-1: COLLECT option**
2. Choose MINMAX from the COLLECT menu (Fig. 17-2) and press ENTER.

![COLLECT menu](image)

Fig. 17-2: COLLECT menu (MINMAX option)

Point Group Name is mmm and Index Restriction is P (Fig. 17-3). These parameters were selected by BRAVAIS and LAUE (see Section 15 Bravais Lattice Determination and Section 16 Laue Symmetry Determination).

![MINMAX limits screen](image)

Fig. 17-3: MINMAX limits screen

3. Set Minimum 2Theta to 3.5°.
4. Set Maximum 2Theta to 50.0° for Mo radiation, or to 140° for Cu radiation.
5. Set HKL Sort Order to HKL.
6. Set Friedel Pairs flag to N.
7. Set Redundant Data flag to Y (Fig. 17-3).
8. Minimum H, K, And L and Maximum H, K And L are automatically changed to correspond to Point Group Name, the Redundant Data flag and 2Theta Range.
9. The number of reflections to be collected is 1607.
10. Press ENTER.
11. Select CHOOSECHECKS from the COLLECT menu (Fig. 17-4) and press ENTER.

![Fig. 17-4: COLLECT menu (CHOOSECHECKS option)]

XSCANS chooses three check reflections from the reflections in the reflection array.

12. If the message:

   **Check reflections are not well-dispersed**

   is displayed at the bottom of the screen, select GETCHECKS from the COLLECT menu and press ENTER. This choice causes up to 30 (usually far fewer) additional well-orthogonalized reflections to be centered. Repeat step 11, but do not repeat step 12 even if the message concerning satisfactory reflections is re-displayed.

13. Select EDITCHECKS from the COLLECT menu (Fig. 17-5) and press ENTER. XSCANS displays three check reflections generated from the reflections in the reflection array.
Check Reflections (20 lines)

<table>
<thead>
<tr>
<th>H</th>
<th>X</th>
<th>Imin</th>
<th>ScanSpeed</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>4</td>
<td>0.78</td>
<td>0.50</td>
</tr>
<tr>
<td>2</td>
<td>2.0.7</td>
<td>0.58</td>
<td>0.58</td>
</tr>
<tr>
<td>3</td>
<td>1.11</td>
<td>0.58</td>
<td>0.58</td>
</tr>
</tbody>
</table>

Information

2-Theta: 55.86
Omega: 27.36
Phi: 67.59
Chi: 346.91
Slit: 1.0059

You can edit or add to this list as needed. Following the H, K and L values for each check reflection are the minimum intensity and speed. By setting minimum intensity to 0.5, XSCANS measures the check reflection the first time and then resets minimum intensity to the observed intensity times 0.5. By setting the scan speed to 0.5° per minute, which is slower than the Slow Rate, the check reflection is first measured at the speed calculated in the data collection routine. Scan Speed for this check reflection is reset to the calculated value. All subsequent measurements of this check reflection are measured at the same speed as for the first measurement.

14. Press **ESC**.

15. Choose STARTS from the COLLECT menu (Fig. 17-6) and press **ENTER**. The starting H, K and L are chosen by the selections made in the MINMAX input panel.
16. Set the starting Sequence number to 1 and the starting Exposure hours to 0.0 (Fig. 17-7). The Sequence number, Starting H, Starting K and Starting L and Exposure time change automatically during data collection. You do not have to change these parameters to restart after an interruption.

17. Press ENTER.
18. Choose SCAN from the COLLECT menu (Fig. 17-8) and press ENTER.

![COLLECT menu](image)

**Fig. 17-8**: COLLECT menu (SCAN option)

Low Range is the number of degrees in $\omega$ that a reflection is scanned from the beginning of the scan to the $K\alpha_1$ position (Fig. 17-9). High Range is the number of degrees in $\omega$ that a reflection is scanned from the $K\alpha_2$ position to the end of the scan. These parameters were set based on the scan widths of the reflections measured during THIN SHELL (see *Section 12 THIN SHELL Search*). Ratio is the ratio of time spent on background to time spent scanning a reflection.
19. Set Ratio to 0.5.

20. Press Y to set Variable Speed Scan to “ON.”

21. Set Slow Rate to 3.0°/min for the \( \omega \) axis.

22. Set Fast Rate to 45.0°/min for the \( \omega \) axis.

23. Set \( I/\sigma \) ratio to 20.0. With these values, each reflection is measured at Fast Rate and (if necessary) at a speed between Fast Rate and Slow Rate to obtain an intensity to standard deviation ratio of about 20.0. Reflections are never scanned slower than Slow Rate or faster than Fast Rate.

24. Set Intercept and Slope to 6000.0 and 0.0, respectively. The values for these parameters disable the function of speed versus 26.

25. Set Shutter Open to Y. This causes the shutter to remain open between reflections.

26. Press ENTER.

27. Select OUTPUT from the COLLECT menu (Fig. 17-10) and press ENTER.
29. Set Index Generation to Internal (indices are generated by XSCANS).

30. Set Maximum Offset to 0.

31. Set Profile Steps to 96.

32. Set Recenter Mode to None. In this example, recentering is not used (see the XSCANS Technical Reference Manual, Section 8.6 OUTPUT for more information on recentering).

33. Set Recenter Intensity Percent and Recenter Frequency to 0.0 (Fig. 17-11). When you have finished, press **ENTER**.

34. Set Check Frequency to 97 (Fig. 17-11) This is the number of non-check reflections measured between measurement of check reflections.
35. Set Output File Status to New. A new file is opened for the intensity data. If data collection is interrupted and you want to add data to the end, set this parameter to Append.

36. Set Output File name to Ylid. The intensity data is saved in a file named YLID.P4Y. The Y in the file extension becomes the first character of the data collection method chosen when the file is saved. For example, Y becomes T for THETA/2THETA data collection, O for OMEGA data collection and X for THETA/XTHETA data collection.
Automatic Selection of Data Collection Parameters

Although you can start data collection at this point, there is probably not enough information to decide on a good value for Maximum 2Theta in MINMAX and the appropriate parameters for speed versus 20 function (Intercept and Slope in SCAN). If you choose AUTOSPEED, a fast $\omega$ data collection is performed using selected H, K, and L and 20 ranges. From the intensities and standard deviations measured during this fast data collection, Maximum 2Theta, Intercept and Slope are set (see the AUTOSPEED algorithm in Section 5.8 of the XSCANS Technical Reference Manual).

To begin automatic selection of data collection parameters:
1. Choose AUTOSPEED from the COLLECT menu (Fig. 18-1) and press ENTER. The fast data collection begins.

![COLLECT menu (AUTOSPEED option)](image)

The H, K, and L values and intensity, standard deviations of the intensities, and the speeds at which the intensity to standard deviation ratio ($I/s_I$) is approximately equal to the input parameter $I$/$\sigma$ (see item 23 in Section 17 Select Data Collection Parameters) are displayed (Fig. 18-2).
Fig. 18-2: Automatic Limits Determination

At the end of the fast data collection, the new values of Slope, Intercept and Maximum 2Theta are displayed (Fig. 18-3).

Fig. 18-3: Automatic Limits Determination showing new Intercept, Slope and Maximum 2Theta values

Look at the MINMAX and SCAN parameters. Maximum 2Theta has not changed, but Intercept and Slope have (Fig. 18-4).
Fig. 18-4: SCAN parameters screen

2. Choose REVIEW from the COLLECT menu (Fig. 18-5) and press ENTER.

Fig. 18-5: COLLECT menu (REVIEW option)

All the data collection parameters are displayed (Fig. 18-6).
### Data Collection Parameters

**Data collection parameters:**

- 2θ min., max: min hkl; max hkl: Lattice centering: point group
  - 3.5 137.9 -1 -1 7 10 22 P

  Scan ranges: Big ratio: Slow & Fast rates, Slope, Intercept, r/sig
  - 0.55 0.55 0.55 3.00 45.00 7.12 6063.9 28.8

- Seq. #: HRL: exposure order: variable speed: mode
  - 1 -1 -1 0.00 HRL V Bisect

- Psi start, stop, delta: Psi indices file
  - 0.00 0.00 0.00 gld.PER

**Check Reflections:**

<table>
<thead>
<tr>
<th>Frequency</th>
<th>Number</th>
<th>Profile steps</th>
<th>output File</th>
<th>Status</th>
</tr>
</thead>
<tbody>
<tr>
<td>97</td>
<td>3</td>
<td>96 gld.P40</td>
<td>APPEND</td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Sequence</th>
<th>HRL</th>
<th>min Int, speed</th>
</tr>
</thead>
<tbody>
<tr>
<td>Check 1</td>
<td>-1</td>
<td>2 0.5 0.50</td>
</tr>
<tr>
<td>Check 2</td>
<td>-2</td>
<td>2 0.5 0.50</td>
</tr>
<tr>
<td>Check 3</td>
<td>0</td>
<td>11 0.1 0.50</td>
</tr>
</tbody>
</table>

**Reflection Array:**

<table>
<thead>
<tr>
<th>2-Theta</th>
<th>182.86</th>
</tr>
</thead>
<tbody>
<tr>
<td>Omega</td>
<td>51.78</td>
</tr>
<tr>
<td>Phi</td>
<td>272.84</td>
</tr>
<tr>
<td>Chi</td>
<td>59.45</td>
</tr>
<tr>
<td>Shutter</td>
<td>CLOSED</td>
</tr>
</tbody>
</table>

**Crystal:**

- gld, Extended w/ X & w-20/ X limits: p4_4_d4

**Line:**

- 32: Press left mouse button or ENTER

---

*Fig. 18-6: DATA COLLECTION parameters screen*
You are now ready to start data collection. Several hours are required to complete this task. We recommend that you save the parameter file at this point:

1. Choose SAVE from the COMMANDS menu (see Fig. 2-8) and press ENTER twice.

   **Note:** If there is a power failure, re-start the data collection procedure as described in Section 5.15 of the XSCANS Technical Reference Manual.

2. Be sure the beam stop is behind the goniometer head, or high c data (c>2850) will be lost.
3. Be sure all enclosure doors are closed and the RESET button has been pressed.
4. Select one of the three data collection options from the COLLECT menu:

   OMEGA
   THETA:2THETA
   THETA:XTHETA

See Section 5.13.1 in the XSCANS Technical Reference Manual for more information. For YLID data collection, choose THETA/2THETA (Fig. 19-1) and press ENTER.

Data collection begins. As data is collected, a profile of each reflection is displayed, as well as a reciprocal space plot of the current layer. Intensities divided by standard deviations are approximated by colored diamonds (white is very strong, black is very weak, yellow is intermediate, red is weak, etc.).
The indices, integrated intensities and standard deviations of the intensities are displayed in the upper right-hand corner of the screen. Lines with a negative sequence number display the latest measurements of the check reflections. The values of the ratio of the latest intensities to the first intensities for the check reflections are displayed on the right-hand side of the screen. Information about the data collection progress is displayed at the bottom of the screen (Fig. 19-2).

**Note:** Black and white are reversed in Fig. 19-2.

Fig. 19-2: Data profile and reciprocal space plots

Fig. 19-3 shows the end of data collection. The last reflections collected are check reflections. It took 10.02 hours to collect all of the data in this example.
Fig. 19-3: Completed data collection screen
20 Absorption Correction Data

Data collected by the method selected in Section 19 Data Collection Methods must be corrected for X-ray absorption by the sample. It is assumed that the crystalline sample you have chosen is centered in the X-ray beam and is completely bathed in the uniform part of the X-ray beam for all angular settings of the goniometer. If these conditions are met, choose one of the two absorption methods described in this section.

**Note:** You can collect data when the sample is larger than the X-ray beam; it is important to use an appropriate absorption correction method (not supplied by Siemens) to correct the data.

There are two methods for gathering the information necessary to make these corrections:
- PSI scan (EMPIRICAL Absorption Correction)
- FACE INDEXING (ANALYTICAL Absorption Correction)

If the crystal has well-defined faces, is mounted on a thin fiber, and is not inside a capillary, use FACE INDEXING, which usually gives the most accurate absorption corrections for the reflection intensities. If these conditions are not met, use PSI scan. For the YLID crystal you used to collect data, you cannot use FACE INDEXING (the crystal has been ground into a sphere). For this crystal, use PSI Scan.

### 20.1 PSI Scan Data Collection

This method is also known as EMPIRICAL Absorption Correction and requires that several reflections are measured at different \( \psi \) values (rotation about the diffraction vector). See the algorithm for PSI Scan Data Collection in Section 5.14.1 of the XSCANS Technical Reference Manual.

1. Choose PSI from the COLLECT menu (Fig. 20-1) and press ENTER.
2. Set Starting Psi to 0 and Ending Psi to 340. Delta Psi changes automatically to 20, the extension on Input File changes to .PSR, the extension on Output File changes to .PST, and Index Generation changes to File. Set Minimum Psi Range to 180, # Of HKLs Required to 12, Minimum Intensity to 20,000, Autoselect to Y, and Collect Data to Y (Fig. 20-2).
3. Press **ENTER**, XSCANS chooses # Of HKLs Required from the YLID.P4T file generated by THETA/2THETA for the YLID sample (*Section 19 Data Collection Methods*). An ASCII file (YLID.PSR) containing the indices, intensities, 2θ angles, χ angles, accessible ψ angles, and selection (Y or N) for PSI scans for these reflections is generated. Then, using YLID.PSR as an input file, each reflection is measured over a ψ range between Starting Psi and Ending Psi (in this case, 0° and 340°). The total psi range for each reflection depends on goniometer collision limits. Data collection parameters set in *Section 18 Automatic Selection of Data Collection Parameters* are used by PSI scan, except 1/σ(I). The data collection method is the same as that used to collect the original data set (in this case, θ/2θ).

A plot of intensity versus ψ is generated for each of the reflections chosen (Fig. 20-3). The intensity data is written to Output File (in this case, YLID.PST).

![Intensity versus ψ plot](image)

**Fig. 20-3:** Intensity versus ψ plot

4. Press **ENTER** to return to the COLLECT menu. The intensity data from THETA/2THETA and PSI scan can now be used to obtain a reduced intensity data set (see *Section 21 Data Reduction*).
20.2 FACE INDEXING Data Collection

If you do not use EMPIRICAL Absorption Correction (see Section 20.1 PSI Scan Data Collection), you can use ANALYTICAL Absorption Correction (FACE INDEXING). For this method, you must identify crystal faces and measure their distances to the center of the crystal. YLID crystals supplied by Siemens have been ground into spheres. For these crystals, you cannot identify faces (only EMPIRICAL Absorption Correction is available).

For this section, a special YLID crystal (with faces) which has not been ground into a sphere is used. An orientation matrix was determined with procedures described in Sections 4–13.

1. Choose UTILITY from the COMMANDS menu (Fig. 20-4). Press ENTER.

2. Choose FACE INDEXING from the UTILITIES menu (Fig. 20-5) and press ENTER.
Fig. 20-5: UTILITY menu (FACE INDEXING option)

The Face Description input panel appears (Fig. 20-6).

Fig. 20-6: Face Description input panel

3. Press ESC. XSCANS is now in IMAGE mode. There is no image yet, because you have not entered face information in the Face Description array.
4. Using the manual control box (see Section 3.1.4 MANUAL Mode), move $\phi$ and $\chi$ until a crystal face is parallel to the horizontal axis of the microscope crosshairs and parallel to the direction of your line of sight (you are viewing the face edge-on). See Fig 20-7.

![Diagram of crosshairs as seen through the microscope's eyepiece](image)

*Fig. 20-7: View of crosshairs as seen through the microscope’s eyepiece*

5. Press the **AXIS PRINT** button. Indices appear at the bottom of the screen. These indices should be close to the ratio of whole numbers. One of the indices is always equal to 1.00 or -1.00. You may have to multiply the indices by a small positive integer to obtain indices that are all close to integer values. Round the result to the nearest integer value. The result is the indices of the face above and parallel to the horizontal crosshair.

6. Multiply the indices by -1 for a face below and parallel to the horizontal crosshair.

7. Measure the distance from the face to the center of the crystal in millimeters (the small divisions on the crosshairs are tenths of mm). You may have to rotate the eyepiece of the microscope to align it along the vertical axis. Write down the indices and distance for this face. Continue to rotate the $\phi$ and $\chi$ axes with the manual control box and use steps 5 through 7 to find the indices and distances of all the major faces.

8. Press **HOME**. The Face Description array reappears (Fig. 20-6).

9. Enter the first set of indices and distances separated by blank spaces. The result looks like Fig. 20-8.
10. Press the down arrow to highlight the next line. Continue to enter the indices and distances in this manner. To edit the input panel, use the arrows, BACKSPACE, HOME and END. To see a list of the EDIT options, press ENTER (Fig. 20-9).

When you have finished editing the list, it looks like Fig. 20-10.
Fig. 20-10: Complete Face Descriptions array input panel

11. Press **ESC**. If you have entered enough faces to produce a closed figure, a picture of the crystal appears onscreen (Fig. 20-11).

Fig. 20-11: Example of a single crystal image

XSCANS is now in IMAGE mode. To remove or add hidden lines, use **INS**. If the hidden lines are removed, the faces are labeled with their indices (Fig. 20-12).
The bright lines of the image are closest to you. The dim lines are furthest from you. You can simulate the movement of $\phi$ with left/right arrows and movement of $\gamma$ with up/down arrows. After using the arrow, press END to move the goniometer axes so the crystal (as you view it through the microscope) looks the same as the image on the screen.

**Note:** You can also use the manual control box to move the goniometer. After moving the axes, press the **AXIS PRINT** button. The image on the screen is updated to correspond to the view seen through the microscope. Press **CTRL/F** to see the **DRIVE TO FACE** input panel. You can then drive the goniometer to a selected face (see Section 25 Driving to a Reflection or Face).

Press **HOME** to finetune the distances and add additional faces to the Face Description array (Fig. 20-11) with these operations. Eventually, you have an accurate description of all the faces (Fig. 20-13).
Fig. 20-13: Face Description example

12. To exit FACE INDEXING, press ESC in IMAGE mode.

13. From the COMMANDS menu, choose SAVE (Fig. 2-8) and press ENTER twice. The Face Description array (Fig. 20-11) is saved to the parameter file.
21 Data Reduction

After the raw data has been collected, you must reduce the data to obtain structure factors in order to solve and refine the crystal structure. We suggest that you reduce the data immediately after data collection and before removing the crystal from the goniometer. The data reduction routine produces information about the quality of the data. If you determine that the data is not very good, you may want to collect more data.

1. Choose REDUCE from the COMMANDS menu (Fig. 21-1) and press ENTER.

![Image of X-ray Single Crystal Analysis System](image)

Fig. 21-1: REDUCE option

2. Choose FILES from the REDUCE menu (Fig. 21-2) and press ENTER.
Input File is the name of the raw data file collected (see Section 19 Data Collection Methods, Fig. 21-3). Output File is the name of the reduced data file. If there is a PSI scan raw data file (Section 20.1 PSI Scan Data Collection), it is reduced with Input File, giving a .PSI file.

3. Set List Standards to Y and List Data to N and press ENTER.
4. Choose GENERAL PARAMETERS from the REDUCE menu (Fig. 21-4) and press ENTER.

Fig. 21-4: GENERAL PARAMETERS option

5. Choose the default values for Asymmetry, Off-Center, Background, Int Weight and Spike (Fig. 21-5). These parameters are used as criteria for reflection rejection (see Section 12.2 of the XSCANS Technical Reference Manual).

Fig. 21-5: GENERAL PARAMETERS input panel
6. Set cosines to Y. This places direction cosines in the output file used for absorption corrections.

7. Set Volume, Batch, Starting Reflection and Ending Reflection to N, 1, 1 and 99,999, respectively, and press ENTER.

8. Choose PROFILE FITTING PARAMETERS from the REDUCE menu (Fig. 21-6) and press ENTER.

9. Set Fit Profile to Y and Diagnostic Output to N (Fig. 21-7). The PROFILE FITTING routine calculates integrated intensities by fitting the profile step data in the raw data file for each reflection to a learned profile shape and press ENTER (see Section 12.3 of the XSCANS Technical Reference Manual).
10. Choose OUTPUT OPTIONS from the REDUCE menu (Fig. 21-8) and press ENTER.

11. Set Rejections to N (Fig. 21-9). This produces a short output of the rejected reflection analysis.
12. Set Cosines to Y. This is the same parameter as in the GENERAL PARAMETERS input panel.
13. Set Angles to N.

**Note:** Do not keep the diffractometer angles in the reduced data file.

14. Set Standards to Elim. The check reflections are not written to the reduced data file.
15. Set List to N. This is the same parameter as in the FILES input panel. Press ENTER.
16. Choose PROCESS DATA from the REDUCE menu (Fig. 21-10).
The raw data is processed and Lorentz and Polarization corrections are made. The data is scaled using the check reflection information. No absorption corrections are made by REDUCE. See the XPREP Version 5 documentation for details about correcting for absorption.

Fig. 21-11 shows the data analysis. The average I/o ratio is 30.33. A variable scan option was chosen to try to achieve an I/o ratio equal to at least 20.0.

Fig. 21-11: Data analysis output example
You can now view the data as sections of a three dimensional reciprocal space plot. Choose RECIPROCAL SPACE SCANS from the REDUCE menu (Fig. 21-12) and press ENTER.

Fig. 21-12: RECIPROCAL SPACE PLOTS option

The H=0 layer appears. The intensities divided by the standard deviations of the reflections are shown as colored diamonds. White is the most intense and black is the least intense (Fig. 21-13). In Fig. 21-13, black and white are reversed.

Fig. 21-13: Reciprocal space plot showing the H=0 layer
Use up/down arrows to display other layers parallel to the currently displayed layer. To rotate to another axis, use left/right arrows (Fig. 21-14). Press **INS** to display the Laue equivalents.

![Reciprocal Space Plot](image)

**Fig. 21-14: Reciprocal space plot showing the K=0 layer**
Once the data has been reduced, you can determine the space group.

1. Choose SPACE GROUP PARAMETERS from the REDUCE menu (Fig. 22-1) and press ENTER.

The SPACE GROUP PARAMETERS menu contains the parameters necessary for SPACE GROUP to choose the best Bravais lattice and space group (Fig. 22-2). See Section 12.5 of the XSCANS Technical Reference Manual for descriptions of these parameters. For the YLID crystal, there is no need to change any of these parameters.
2. Press **ENTER**.

3. Choose **DETERMINE SPACE GROUP** from the REDUCE menu (Fig. 22-3) and press **ENTER**.

**Fig. 22-2**: SPACE GROUP DETERMINATION input panel

**Fig. 22-3**: DETERMINE SPACE GROUP option
Statistics for possible lattice centering are shown in Fig. 22-4. There is no lattice centering. The average intensities are large for each class of reflections. The only possible choice is LATTICE TYPE P.

![Fig. 22-4: Lattice statistics](chart)

4. Choose this option and press ENTER.

Fig. 22-5 shows the Niggli reduced cell and choices for the Bravais lattice. These should be the same as determined in Section 13 Precise Least Squares Unit Cell Determination and Section 15 Bravais Lattice Determination. Because some redundant data was collected, it is possible to calculate a reliability index for those reflections which should have equal intensities. There were 429 such reflections and the resulting merging reliability factor, R(int), is 0.021.
5. Choose option A ORTHORHOMBIC P and press ENTER. Fig. 22-6 shows the options concerning Chirality. You can enter your own choice for space group.

6. Select DETERMINE SPACE GROUP and press ENTER.
7. Choose ORTHORHOMBIC (O) (Fig. 22-7) and press ENTER.
Fig. 22-7: ORTHORHOMBIC (O) option

8. Choose LATTICE TYPE (P) (Fig. 22-8) and press ENTER.

Fig. 22-8: LATTICE TYPE (P) option

Fig. 22-9 shows the statistics for possible systematic absences due to screw axes or glide planes. From the average intensities, it is clear that there are three twofold screw axes present. The only choice is space group P2_12_12. In other cases (not the YLID), there can be several space groups.
from which to choose. See the SHELXTL Reference Manual for more examples of space group determination.

Fig. 22.9: Systematic absence statistics

9. Press ENTER.
You can now generate the input files to run SHELXTL.
1. Choose SHELXTL FILE PARAMETERS from the REDUCE menu (Fig. 23-1) and press ENTER.

![Fig. 23-1: SHELXTL File Parameters option]

You can use any of the TREF or PATT options, as well as any of the FMAP options (Fig. 23-2). See the SHELXTL Reference Manual for a description of these options. For the YLID crystal, use the default values and press ENTER.
2. Choose SHELXTL FILES from the REDUCE menu (Fig. 23-3) and press ENTER.

3. Enter the EMPIRICAL formula using the conventional atomic symbols. The first character is always upper case. If the symbol has two characters, the second is lower case (for example, Cu for copper). The atomic symbols are followed by the number of atoms of that type in the compound.
For the YLID, enter:

C11H1002S

with no spaces (Fig. 23-4).

Fig. 23-4: DATA REDUCTION input panel

4. Press ENTER. The value of Z (number of formula units per unit cell), the elemental composition, and the density of the crystal are shown (Fig. 23-5).
Fig. 23-5: Crystal elemental composition and density

5. Press **ENTER**.

6. Choose an output filename. Often, you should choose a new root name, because you may want to process the data differently later (for example, the first time you may want to use direct methods, and later you may want to use the Patterson method).

7. Enter **YLID** as the new root name (Fig. 23-6) and press **ENTER**.
The contents of the SHELXTL file with extension .INS are shown in Fig. 23-7. To include the direction cosines in the structure factor file, choose INCLUDE BATCH NUMBER AND COSINES. Direction cosines can be used to make absorption corrections later.

```plaintext
Fig. 23-7: SHELXTL file contents

8. Press ENTER.
```
Note: The data set collected by these procedures was processed with SHELXTL. The final conventional R-value was determined to be 0.026 and the goodness-of-fit (GOF) was determined to be 1.03. These values are typical for YLID test crystal data sets collected this way.
AUTOMATIC lets XSCANS make most decisions for you when determining the reduced unit cell, Bravais lattice, Laue symmetry and space group. It also establishes the data collection parameters. See Section 4 AUTOMATIC Mode Menu for more information.

1. Use the procedures described in Sections 2, 5, 6 and 7 to start XSCANS, optically align the crystal to take a rotation photograph, and analyze the photograph.

2. Set Configuration Filename to YLIDA (so it is different from that selected in Section 5.3 INTERACTIVE Method) and Title to AUTOMATIC MODE in the CRYSTAL input panel (Fig. 24-1).

<table>
<thead>
<tr>
<th>CRYSTAL Options</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>File Name</td>
<td>YLIDA</td>
</tr>
<tr>
<td>Title</td>
<td>Automatic mode</td>
</tr>
<tr>
<td>Chemical Formula</td>
<td>C11H10O2S</td>
</tr>
<tr>
<td>Crystal Color</td>
<td>yellow</td>
</tr>
<tr>
<td>Crystal Description</td>
<td>sphere</td>
</tr>
<tr>
<td>Crystal maximum size</td>
<td>.36 mm</td>
</tr>
<tr>
<td>Crystal intermediate size</td>
<td>.36 mm</td>
</tr>
<tr>
<td>Crystal minimum size</td>
<td>.36 mm</td>
</tr>
<tr>
<td>Density method</td>
<td>?</td>
</tr>
<tr>
<td>Measured Density</td>
<td>?</td>
</tr>
<tr>
<td>Temperature</td>
<td>25</td>
</tr>
</tbody>
</table>

Fig. 24-1: CRYSTAL options

3. Perform all the other operations in Sections 5, 6 and 7.

4. Choose COLLECT from the COMMANDS menu (Fig. 17-1) and press ENTER.

5. Select MINMAX from the COLLECT menu (Fig. 17-2) and press ENTER.

6. Set Minimum 2Theta to 3.5° and Maximum 2Theta to 140° (if you are using Mo radiation, set Maximum 2Theta to 50°).

7. Set Redundant Data to Y, Friedel Pairs to N and HKL Sort Order to HKL (Fig. 24-2). All the other parameters on the input panel are set during execution of AUTOMATIC mode.
8. Press **ENTER**.

9. Choose SCAN from the COLLECT menu (Fig. 17-8) and press **ENTER**.

10. Set all the parameters from Ratio to Shutter Open to the values shown in Fig. 24-3. Low Range and High Range are set during execution of AUTOMATIC mode.

---

**Fig. 24-2:** MINIMUM AND MAXIMUM LIMITS input panel

**Fig. 24-3:** SCAN PARAMETERS input panel
11. Press **ENTER**.
12. Choose **OUTPUT** from the **COLLECT** menu (Fig. 17-10) and press **ENTER**.
13. Set all the parameters to the values shown in Fig. 24-4 and press **ENTER**.

```
Output Parameters for data collection

Check Frequency: 57
Index Generation: INTERNAL
Maximum offset: 0
Profile step: 96
Recenter mode: NONE
Recenter I loss: 0.00
Recenter Frequency: 0.00
Input File: giida.PSR
Output File: giida.P40
Output file status: NEW
```

*Fig. 24-4: OUTPUT PARAMETERS screen*

You do not need to set parameters in the STARTS panel or choose CHECK REFLECTIONS. These tasks are performed by AUTOMATIC.

14. Choose **AUTOMATIC** from the **COMMANDS** menu (Fig. 24-5) and press **ENTER**.
15. Enter Chemical Formula as C11H10O2S.

   **Note:** Error Level is reserved for XSCANS future development.

16. Set Logging to Y.

17. Set Data Collection Type to O (OMEGA data collection). Set all the other path parameters to Y except Exit On Completion.

18. Set Exit On Completion to N. XSCANS exits to DOS to perform DIRECT METHODS (Fig. 24-6).
19. Press ENTER. XSCANS performs all the following tasks automatically (see the XSCANS Technical Reference Manual and Sections 4-23 of this manual):

- Using INCREMENTAL search, one reflection is found and centered (see Section 8.2 INCREMENTAL Search Method).
- Using HEMISPHERE search, about 14 reflections close to the initial reflection are found and centered. As they are found, they are assigned indices and the reduced unit cell parameters are determined (see Section 8.4 HEMISPHERE Search Method).
- Using FRACTIONAL search, additional reflections are searched for to determine whether the unit cell volume needs to be doubled or tripled (see Section 11.2 Searching for Reflections with Fractional Indices).
- More reflections are found and centered by THIN SHELL search to determine precise reduced unit cell parameters (see Sections 12 and 13).
- The best Bravais lattice is determined (see Section 15 Bravais Lattice Determination).
- Reflections which should have identical intensities are collected to determine the Laue symmetry (see Section 16 Laue Symmetry Determination).
- The scan widths for data collection are determined from the scan widths measured for the reflections already centered (see Section 17 Selecting Data Collection Parameters).
- Three check reflections are chosen from the list of centered reflections (see Section 17 Selecting Data Collection Parameters). Additional reflections are centered, if necessary, to obtain a better trio of check reflections.
- A fast data collection is performed using AUTOSPEED to determine Maximum 2Theta, Slope and Intercept for the speed versus 26 function (see Section 18 Automatic Selection of Data Collection Parameters).
- A complete data set is collected using OMEGA data collection (see Section 19 Data Collection Methods).
- PSI scan reflections are chosen from the raw reflection data set just collected. A PSI scan data collection using OMEGA data collection is performed (see Section 20.1 PSI Scan Data Collection).
- The data is corrected for the Lorentz and Polarization effects and is profile fitted using REDUCE (see Section 21 Data Reduction).
- The space group is determined (see Section 22 Space Group Determination).
- The SHELXTL file YLID0.INS is generated (see Section 23 Generating SHELXTL Files).
- The SHELXTL direct methods program, XS, is run to solve the crystal structure. See the SHELXTL Reference Manual.
- The SHELXTL plot program, XP, is initiated and the structure is displayed on the screen (Fig. 24-7). See the SHELXTL Reference Manual.
- You can use XP's features to assign atomic types and labels to the atoms, then to refine the structure using the LEAST SQUARES procedure, XLS.

In this example, all the procedures listed on the AUTOMATIC mode menu were used. You can do some of the procedures with INTERACTIVE mode, then select other procedures to be done automatically by setting the appropriate path variables to Y or N (Fig. 24-6).

Fig. 24-7: YLID structure after AUTOMATIC mode

The data set collected by XSCANS using AUTOMATIC was solved and refined using SHELXTL (see Fig. 24-8). The merging reliability factor R(int) was calculated to be equal to 0.012. The conventional R-value was found to be 0.026 and the goodness-of-fit (GOF) was found to be 1.03. These values are typical for test YLID crystal data sets collected in this manner.

Fig. 24-8 Example of a refined YLID structure
25 Driving to a Reflection or Face

If an orientation matrix has been determined, you can drive the goniometer to a given reflection whose indices or angles are known or to a crystal face whose indices are known.

25.1 Driving to a Reflection

To drive to a reflection:
1. Choose HKL from the Goniometer menu or DRIVE TO HKL from the UTILITIES menu.
2. Press ENTER.
3. Input the H, K, L and Psi values or the 2θ, ω, ϕ and χ values for the reflection (Fig. 25-1) and press ENTER. The goniometer drives to the appropriate angles so the reflection is in the diffracting condition.

Fig. 25-1: Goniometer Options input panel
25.2 Driving to a Face

To drive to a face:
1. Choose FACE from the GONIOMETER menu or DRIVE TO FACE from the UTILITIES menu.
2. Press ENTER.
3. Input the H, K and L values for the crystal face (Fig. 25-2) and press ENTER. The goniometer drives to angles so the face is above and parallel to the horizontal crosshair of the microscope, and so it is viewed edge-on.

Fig. 25-2: GONIOMETER Options input panel (HKL)
Follow these steps to replay a collected data set:

1. Choose REPLAY from the COLLECT menu (Fig. 26-1) and press ENTER. The PARAMETERS FOR DATA REPLAY input panel appears. You can select a reflection with given H, K and L indices or with a range of sequence numbers.

Fig. 26-1: COLLECT menu (REPLAY option)

2. Set Start to 1 and End to 25 (Fig. 26-2) and press ENTER.
For each reflection, the profile, indices, intensity and standard deviation are displayed in the same way as when data was collected. The reciprocal space plot is also generated. To stop the replay at any point, press CTRL/BREAK (Fig. 26-3).
27 Reflection Array Features

The reflection array contains information about a selected number (up to 100) of reflections, including photo coordinates, angles for the reflection centers, reflection indices, reflection intensities and X-ray wavelengths corresponding to the reflection centers.

This information is used to determine the lattice parameters of the reduced cell and the Bravais lattice, as well as the orientation of the crystal with respect to the goniometer axes.

In the example YLID data collection (Sections 4–23), some of the procedures associated with this array were used. There are many other useful features of the reflection array. We recommend that first time users of XSCANS load the YLID.P4P file (Section 2.6 Loading the Parameter File) which was used for the collection of the intensity data (Sections 4–23). See Section 13 of the XSCANS Technical Reference Manual.

27.1 Entering and Modifying Information

Detailed information about entering and editing data in the reflection array is in Section 2.8 Editing Parameter Input (in this manual) and Section 13.7 of the XSCANS Technical Reference Manual. The first field on any line indicates the type of information in the remainder of the line. Valid flags for the first field are:

H Reflection has valid indices
A Reflection has valid angle information
R Reflection angles have been centered (refined)
L Reflection is used in INDEX and LEAST SQUARES
1 Wavelength for the reflection is that for Ka1
2 Wavelength for the reflection is that for Ka2

Note: If neither a 1 nor a 2 flag is present, the wavelength is that for the intensity-weighted average of Ka1 and Ka2.

P Instead of angles, the information stored is 2X and 2Y from a rotation photograph
S Instead of angles, the information stored is X, Y and φ for a still photograph

You can make the same changes to many reflections in the array simultaneously. If, for example, you decide to translate the crystal according to the results of a LEAST SQUARES calculation (see Section 10 LEAST SQUARES), you must re-center all the reflections in the array.

To edit the reflections:

1. Select MODIFY from the REFL_ARRAY menu and press ENTER. Select the block of reflections to modify by setting First Reflection # to 1, Last Reflection # to 100, and Field To Modify to F (Fig. 27-1). To center on these reflections, remove the R flags.
Fig. 27-1: REFL_ARRAY menu

2. Set New Value to -R. The minus sign indicates that the corresponding flag(s) are to be removed. A plus sign indicates that the corresponding flag(s) are to be added. Flag characters without a plus or a minus sign indicate that the New Value flag(s) replace existing ones.

3. When you have entered all of the parameters, press ENTER (Fig. 27-2).

Fig. 27-2: REFL_ARRAY/MODIFY options input panel
All the R flags are removed (Fig. 27-3).

**Fig. 27-3: Reflection Array screen**

Another way to enter information into the reflection array is with CALCULATE. If an orientation matrix has been calculated, you can input H flags and H, K and L values into the array (Fig. 27-4).

**Fig. 27-4: New HKL entries**

1. Choose CALCULATE from the REFL_ARRAY menu (Fig. 27-5) and press ENTER.
Fig. 27-5: **CALCULATE** option

This procedure calculates the angles corresponding to the H, K and L values and enters them into the reflection array (Fig. 27-6).

**Fig. 27-6: Reflection array screen**

2. To sort the reflections in the array, choose **SORT** from the REFL_ARRAY menu and press **ENTER** (Fig. 27-7).
3. Input the range of reflections you want to sort.

4. Choose the field you want to sort on and whether you want the reflections sorted in ascending or descending order. To sort all the reflections into ascending 20 order, set First Reflection to 1, Last Reflection to 100, Field To Sort to T, and Descending to N (Fig. 27-8).

5. Press ENTER. The reflections are sorted into the desired order (Fig. 27-9).
### XSCANS: X-ray Single Crystal Analysis System

#### Reflection Array (100 lines)

<table>
<thead>
<tr>
<th>Flags</th>
<th>u</th>
<th>v</th>
<th>L</th>
<th>2Theta</th>
<th>Omega</th>
<th>Phi</th>
<th>Chi</th>
<th>Incorr</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>-2</td>
<td>-3</td>
<td>?</td>
<td>55.765</td>
<td>27.802</td>
<td>329.628</td>
<td>359.864</td>
<td>247962.28</td>
</tr>
<tr>
<td>2</td>
<td>-2</td>
<td>3</td>
<td>-7</td>
<td>55.728</td>
<td>27.871</td>
<td>194.070</td>
<td>385.458</td>
<td>251155.38</td>
</tr>
<tr>
<td>3</td>
<td>2</td>
<td>3</td>
<td>-7</td>
<td>55.733</td>
<td>27.882</td>
<td>148.628</td>
<td>0.934</td>
<td>254995.30</td>
</tr>
<tr>
<td>4</td>
<td>2</td>
<td>-3</td>
<td>?</td>
<td>55.784</td>
<td>27.926</td>
<td>14.870</td>
<td>54.561</td>
<td>243627.18</td>
</tr>
<tr>
<td>5</td>
<td>1</td>
<td>-4</td>
<td>?</td>
<td>55.891</td>
<td>27.981</td>
<td>4.290</td>
<td>36.939</td>
<td>9942.69</td>
</tr>
<tr>
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<td>2</td>
<td>3</td>
<td>7</td>
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<td>27.986</td>
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<td>57.688</td>
<td>266688.80</td>
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<td>-3</td>
<td>-7</td>
<td>55.750</td>
<td>27.805</td>
<td>82.264</td>
<td>359.201</td>
<td>231818.88</td>
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<td>3</td>
<td>?</td>
<td>55.747</td>
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<td>4</td>
<td>-7</td>
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<td>323.182</td>
<td>9775.32</td>
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<td>1</td>
<td>1</td>
<td>32.880</td>
<td>16.800</td>
<td>145.896</td>
<td>54.460</td>
<td>0.86</td>
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<td>11</td>
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<td>1</td>
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<td>44.381</td>
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<td>-1</td>
<td>-4</td>
<td>?</td>
<td>55.895</td>
<td>27.961</td>
<td>343.793</td>
<td>18.764</td>
<td>9183.30</td>
</tr>
<tr>
<td>13</td>
<td>3</td>
<td>3</td>
<td>-1</td>
<td>55.641</td>
<td>27.848</td>
<td>156.245</td>
<td>35.528</td>
<td>14742.45</td>
</tr>
<tr>
<td>14</td>
<td>-3</td>
<td>-3</td>
<td>-1</td>
<td>55.653</td>
<td>27.889</td>
<td>343.123</td>
<td>315.670</td>
<td>11887.27</td>
</tr>
<tr>
<td>15</td>
<td>3</td>
<td>-3</td>
<td>-1</td>
<td>55.652</td>
<td>27.833</td>
<td>72.957</td>
<td>33.353</td>
<td>11287.27</td>
</tr>
<tr>
<td>16</td>
<td>-2</td>
<td>-3</td>
<td>-7</td>
<td>55.778</td>
<td>27.932</td>
<td>36.938</td>
<td>382.340</td>
<td>221956.50</td>
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<td>17</td>
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<td>3</td>
<td>1</td>
<td>55.670</td>
<td>27.869</td>
<td>65.724</td>
<td>41.966</td>
<td>11828.80</td>
</tr>
<tr>
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<td>-3</td>
<td>3</td>
<td>-1</td>
<td>55.643</td>
<td>27.786</td>
<td>245.724</td>
<td>310.843</td>
<td>11347.48</td>
</tr>
<tr>
<td>19</td>
<td>-3</td>
<td>3</td>
<td>1</td>
<td>55.634</td>
<td>27.792</td>
<td>252.957</td>
<td>326.646</td>
<td>18896.52</td>
</tr>
<tr>
<td>20</td>
<td>2</td>
<td>3</td>
<td>3</td>
<td>18.980</td>
<td>0.347</td>
<td>228.583</td>
<td>10.556</td>
<td>725270.90</td>
</tr>
</tbody>
</table>

**Z-Theta** 0.88  
**Omega** 0.88  
**Phi** 329.54  
**Chi** 29.86  
**Shutter** CLOSED

Crystal: YLIB, Extended w/X & w-29/-X limits: pl_def

---

**Fig. 27-9:** Reflection array
27.2 Centering Reflections

During the example YLID data collection (Sections 4–23), reflections were found and centered with various search methods (see Section 8 Locating and Centering Reflections). You can also start centering of reflections which are in the reflection array. Only reflections with the A flag (angles present) but without the R flag (angles refined) are centered.

To center the reflections in the reflection array:
1. Choose CENTER from the REFL_ARRAY menu (Fig. 27-10) and press ENTER.

![Centering Reflections](image)

**Fig. 27-10: CENTER option**

The CENTERING PARAMETERS input panel appears (Fig. 27-11).

**Note:** See Section 7.2 of the XSCANS Technical Reference manual for a description of these parameters and of the CENTERING algorithm. Normally, you do not need to change any of these parameters.
Fig. 27-11. CENTERING PARAMETERS screen

2. Press ENTER. All appropriate reflections are centered and the refined angles and R flags are inserted into the reflection array.
27.3 Driving to a Reflection

You can drive the goniometer to any reflection in the array:
1. Choose DRIVE from the REFL_ARRAY menu (Fig. 27-12) and press ENTER.

Fig. 27-12: DRIVE option

The reflection array appears (Fig. 27-13).

Fig. 27-13: Reflection array screen
2. Move the cursor bar with up/down arrows until the desired reflection is highlighted. The reflection must have an A flag.

3. Press **ENTER**. The goniometer moves to the selected angles.
27.4 Entering and Transforming the Orientation Matrix

To view, modify or replace the current orientation matrix:

1. Choose MATRIX from the REFL_ARRAY menu (Fig. 27-14) and press ENTER.

2. The orientation matrix is displayed (Fig. 27-15).

   **Note:** If you make modifications, the indices of the reflections in the array are no longer correct with respect to the modified orientation matrix.
27 Reflection Array Features

**XSCANS: X-ray Single Crystal Analysis System Copyright Siemens**

**REFL/ARRAY/MATRIX Options**

<table>
<thead>
<tr>
<th>Matrix(1,1)</th>
<th>0.0161282</th>
</tr>
</thead>
<tbody>
<tr>
<td>Matrix(1,2)</td>
<td>0.0476869</td>
</tr>
<tr>
<td>Matrix(1,3)</td>
<td>0.8364552</td>
</tr>
<tr>
<td>Matrix(2,1)</td>
<td>-0.8445183</td>
</tr>
<tr>
<td>Matrix(2,2)</td>
<td>-0.8990848</td>
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<tr>
<td>Matrix(2,3)</td>
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<tr>
<td>Matrix(3,2)</td>
<td>0.8038681</td>
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</tbody>
</table>

<table>
<thead>
<tr>
<th>a</th>
<th>5.99548</th>
</tr>
</thead>
<tbody>
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<td>b</td>
<td>9.83441</td>
</tr>
<tr>
<td>c</td>
<td>18.38111</td>
</tr>
<tr>
<td>alpha</td>
<td>99.99244</td>
</tr>
<tr>
<td>beta</td>
<td>98.81446</td>
</tr>
<tr>
<td>gamma</td>
<td>98.88155</td>
</tr>
<tr>
<td>Volume</td>
<td>999.581</td>
</tr>
</tbody>
</table>

Crystal: VLID, Extended w/x & w-20/x limits: p4_4
Enter value for matrix row 1, column 1

---

**Fig. 27-15:** REFL_ARRAY/MATRIX options screen

To transform the indices of the reflections:

1. Choose TRANSFORM in the REFL_ARRAY menu (Fig. 27-16) and press ENTER.

---

**Fig. 27-16:** TRANSFORM option

2. Enter a 3x3 transformation matrix (Fig. 27-17). The determinant of the matrix entered must be greater than 0. The determinant of the matrix is shown on the HELP line.
**Note:** TRANSFORM must be followed by LEAST SQUARES to calculate a new orientation matrix.

![Table showing REFL_ARRAY/TRANSFORM Options input panel](image)

**Fig. 27-17:** REFL_ARRAY/TRANSFORM Options input panel
27.5 Clearing the Reflection Array

To clear the whole reflection array:

Choose CLEAR from the REFL_ARRAY menu (Fig. 27-18) and press ENTER. All the information in the reflection array is removed.

Fig. 27-18: CLEAR option
28  Plot Reflections

You can collect data for various types of scans, then display the data as plots on the screen. For example, to display a 0/20 plot of the reflection with indices equal to 2,1,1:

1. Load the YLID.P4P file (Section 2.6 Loading the Parameter File) if it is not already loaded.

2. Choose DRIVE TO HKL from the UTILITIES menu and press ENTER.

3. Set H to 0, K to 2, L to 2 and Psi to 0.0 (Section 25 Driving to a Reflection or Face) and press ENTER. The goniometer drives to the center of the reflection.

4. Choose PLOT REFLECTION from the UTILITIES menu (Fig. 28-1) and press ENTER.

5. Set Type Of Plot to t2t, Scan Range to 2.0° and Scan Speed to 10.0°/minute.

6. Set Number Of Window to 0 (full screen) (Fig. 28-2) and press ENTER.
The diffractometer makes the appropriate scan, then plots the data on the screen (Fig. 28-3).
It is sometimes convenient to simulate data collection before it is actually collected. Using the data collection parameters used during data collection, XSCANS generates all the indices and simulates a scan through the corresponding reflections. As it does so, XSCANS estimates the total time that it takes to collect the data. At the end of this simulation (which normally takes a few minutes), there is an estimate of the total time it will take to collect all of the data. If Variable Scan is N, this estimate is fairly accurate. If Variable Scan is Y, the estimate is not very accurate because it is impossible to estimate the intensities and the speeds at which the reflections are collected.

To simulate data collection:
1. Load the YLID.P4P file used to collect the example YLID data collection (Sections 4-23 and Section 2.6 Loading the Parameter File).
2. Choose STARTS from the COLLECT menu and set the parameters for data collection as described in Section 17 Select Data Collection Parameters.
3. Choose SIMULATE from the COLLECT menu (Fig. 29-1) and press ENTER.

Fig. 29-1: SIMULATE data collection example

The final result looks like Fig. 29-2.
Fig. 29-2: Final result of SIMULATE data collection
30 Displaying Graphs of Centered Reflections

If you suspect that the crystal is not a very good single crystal, it is useful to display the profiles of the centered reflections. To do this, plot the files that contain these profiles. The names of the files are REFL_NNN.DAT, where NNN is the number of the reflection in the reflection array.

1. Choose GRAPH from the COMMANDS menu (Fig. 30-1) and press ENTER.

2. Choose FILES from the GRAPH menu (Fig. 30-2) and press ENTER.
3. Set Plot Data Filename to the name of the file you want to plot. Set Quadrant to 0 (full screen).

4. Set Pre-Clear to Y (Fig. 30-3) and press ENTER.

The selected profile is displayed (Fig. 30-4).
Fig. 30-4: GRAPH/FILE plot example
Appendix A
YLID Test Crystal

A YLID test crystal is supplied with each new diffractometer. It is used to align the diffractometer and set the various detector adjustments.

**Note:** The diffractometer was aligned by Siemens before shipment and again during installation.

The full chemical name is 2-dimethylsulfuranylidene-1,3-dione. The YLID has several attributes which make it suitable for use as a test crystal:

- **Chemical and X-ray stability**: Mounted YLID test crystals can be stored at room temperature and give reproducible intensities for many years. They do not decompose or decay in either Mo or Cu X-rays.

**CAUTION**

EQUIPMENT DAMAGE! Do not use the test crystals with an LT-2 low temperature device. The test crystals are destroyed when warmed to room temperature.

- **Size and shape**: The test crystals have been ground into spheres, which makes them easy to align optically. Their size (0.4 mm) and shape simplifies correction for X-ray absorption.

- **Chemical composition**: The chemical formula is C_{11} H_{10} O_{2} S. It is representative of both organic and organometallic compounds. The structure can be solved using both direct and Patterson methods.

- **Crystallographic unit cell**: YLID crystals are orthorhombic. The cell dimensions are a=5.96, b=9.04 and c=18.39 Å. Because of the small unit cell, all of the data can be collected in a few hours.
Appendix B
Absorption Correction Programs

With XSCANS, you can collect the data necessary to make absorption corrections (see Section 20 Absorption Correction Data).

**Note:** XSCANS does not perform the absorption correction.

For ANALYTICAL Absorption Correction or EMPIRICAL Absorption Correction, the full data set and the PSI scan data must be reduced with REDUCE in XSCANS (see Section 21 Data Reduction). The output file must include the direction cosines for each reflection. The full reduced data file has the extension .RAW and the PSI scan reduced data file has the extension .PSI.

Use XPREP to correct the full data set for absorption. XPREP requires the cell constants, wavelength, Laue class, and for ANALYTICAL Absorption Correction, the face indices and distances it obtains from the .P4P file. XPREP reads the .RAW file, and for EMPIRICAL Absorption Correction, the .PSI file, and outputs the corrected full data set to a file with an .HKL extension. This file can be used as input to the XS or XL programs. See the XPREP documentation for more information.