

Version 5.0 User's Manual

USER'S MANUAL



SMART APEX User's Manual

This manual covers the SMART APEX software package. To order additional copies of this publication, request the part number shown at the bottom of this page.

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Address comments to:

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

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1 Introduction

1.1 SMART APEX Features

The Bruker AXS SMART APEX system is the newest member the SMART CCD product line of instrumentation for single crystal X-ray diffraction. This system is completely redesigned and features a new CCD detector, based upon a 4K CCD chip. It also incorporates a new goniometer with an enhanced interface, all enclosed in a fail-safe X-ray enclosure system.

From a software and operational viewpoint, the SMART APEX system shares many common features with its predecessors (SMART 1K, SMART 2K, SMART 1000, SMART 2000, SMART 1500, SMART 6000, and SMART 6500). These features are discussed in the SMART Reference Manual. Also, most of the information in the current SMART, SAINT, and ASTRO manuals applies to the SMART APEX system and the other SMART CCD systems.

From a hardware viewpoint, the SMART APEX also shares common hardware components. Other members of this new generation of instruments include the D8 ADVANCE and D8 DISCOVER, and the D8 GADDS systems for general diffraction. Documentation on some of these common hardware and software components is available in the user's manuals for the D8 family of instruments.

1.2 How to Use This Manual

This manual covers basic tutorial steps to get you started using the SMART APEX X-ray diffractometer system.

The manual leads you step-by-step through an actual data collection and structure determination experiment performed on a typical system. Additional reference is made (where necessary) to related program manuals (SMART, SAINT, ASTRO, and SHELXTL), appendices, other factory documentation, and standard crystallographic reference materials. And special notes are included where new users tend to have problems.

Information is organized in this manual as follows:

- Section 1, Introduction, presents system features and instructions on using this manual.
- Section 2, Software Overview, provides details on the software used with the system.

- Section 3, Hardware Overview, provides details on the system configuration for those not familiar with the equipment. Included is a brief description of each component as well as options available with the system.
- Section 4, Data Collection, describes basic operation of the hardware and software of the SMART APEX system to collect single crystal X-ray diffraction data. Procedural steps are presented in a typical fashion that you would use to analyze an unknown sample.
- Section 5, Data Integration, explains how to convert the raw frame data to a set of integrated intensities that can be used to solve and refine the crystal structure. This section will illustrate use of the SAINTPLUS interface program to carry out integration of a data set previously collected in Section 4.
- Section 6, Structure Determination & Refinement, demonstrates how to use the reduced intensity data to produce a crystal structure. The SHELXTL suite of crystallographic programs will be described.

Examples in this manual use the specimen sample 2-dimethylsulfuranylidene-indan-1, 3dione (YLID)¹, similar to the crystal provided with your system. By using a similar sample, you can duplicate the procedures described in this manual and obtain similar results to assure your understanding of the SMART programs. (That is, your results should match those outlined in this manual except for minor variations caused by slight differences in specimens or instrument parameters.)

Note: Before using this manual, ensure that the system is in proper working condition (e.g. the X-ray tube is aligned) and that all software has been properly installed.

2 Software Overview

The essential software components of the SMART APEX system (located in the Bruker AXS program folder on your Windows NT desktop) are:

SMART program



This on-line program controls the instrument to collect the experimental data used by the other programs in the system program suite. SAINTPLUS program



This program sets up and carries out the integration process.





This program suite produces a crystal structure from the integrated data.



SMART off-line This off-line version performs many data display and manipulation functions but cannot control the instrument.

VIDEO program



video

This program controls the real-time video images from the video camera.

2-2

3 Hardware Overview

3.1 SMART APEX System Components

The SMART APEX system (Figure 3-1) consists of the following basic components.

- 3-axis goniometer module with SMART APEX detector (see Section 3.2)
- Radiation safety enclosure with interlocks and warning lights
- D8 controller
- Refrigerated recirculator for SMART APEX detector
- Computer

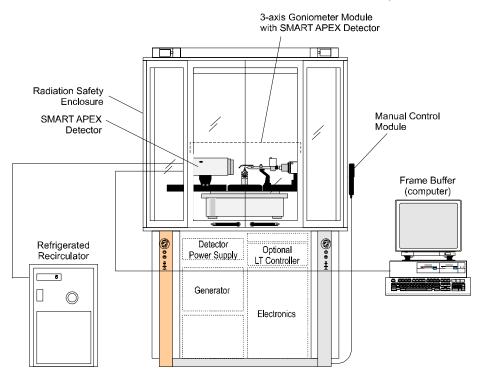


Figure 3-1. SMART APEX system components

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3-1

Radiation safety enclosure with interlocks and warning lights

A common component of all systems in the D8 family is the radiation safety enclosure. This new design is fully leaded (leaded metal sides and panels, leaded windows) to protect you from stray radiation. The enclosure also includes warning lamps (a government requirement) that alert you when X-rays are being generated. And, as a special feature, the enclosure incorporates interlocks (for both hardware and software)—an automatic system-interruption device that senses when the doors and panels are open and prevents use of the shutter and data collection until you close the doors.

D8 controller

The D8 controller is an electronic module enclosed in the rack behind the font panel of the instrument. It contains all of the electronics and firmware for driving goniometer angles, opening the X-ray shutters, and monitoring other instrument functions, such as safety interlocks, generator status, and detector statuses.

Refrigerated recirculator for SMART APEX detector

The refrigerated recirculator uses Peltier technology to cool the CCD chip to a required -40° C to minimize dark currents.

Computer

Included with the system is a high-speed computer, which is used for control of the experiment, storage of raw frame data, integration of data, and solution and refinement of the structure. The computer uses the Microsoft Windows NT[®] operating system and includes the software described in Section 2. Often the computer is attached to a network of similarly configured computers with access to local and/or network printers.

3.2 3-axis Goniometer Module with SMART APEX Detector

The 3-axis goniometer module and its associated SMART APEX detector comprise the unique hardware of the SMART APEX system. This is the part of the instrument that actually performs the experiment.

Several components comprise the 3-axis goniometer module with SMART APEX detector (Figure 3-2).

- Goniometer with fixed chi (χ)stage
- X-ray source (including shielded X-ray tube, X-ray safety shutter, and graphite crystal monochromator)

- SMART rotary shutter and incident beam collimator (with beam stop)
- SMART APEX detector
- K760 X-ray generator
- Manual control module
- Video camera

Goniometer with fixed chi stage

The standard SMART APEX system uses a horizontally oriented D8 PLATFORM goniometer base (with 2-theta [2 θ] and omega [ω] drives) with dovetail tracks for the X-ray source and the detector, and mounting posts for accessories such as the video camera and optional low-temperature attachment. The system also incorporates a fixed chi stage

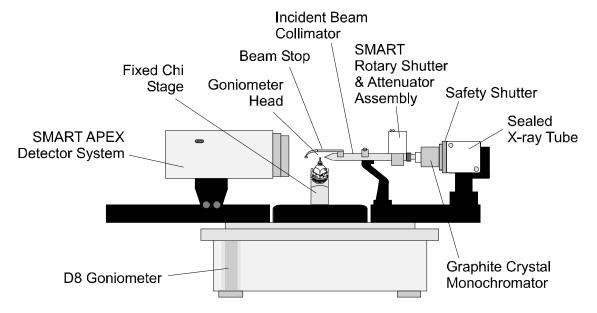


Figure 3-2. SMART APEX & goniometer module instrumentation

with chi angle of approximately 54.74° and a phi drive with 360° rotation.

All four axes (2 θ , ω , ϕ , and χ) intersect within a volume of approximately 10 microns. These axes are shown in Figure 3-3.

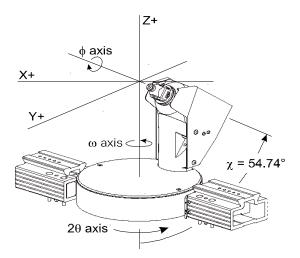


Figure 3-3. Fixed χ , 3-axis goniometer

X-ray source

Three components (Figure 3-2) comprise the X-ray source: a shielded X-ray tube, an X-ray safety shutter, and a graphite crystal monochromator.

The sealed tube X-ray source, with a molybdenum (Mo) target, produces the X-ray beam used by the SMART APEX system.

The X-ray safety shutter is built into the X-ray tube shield. The shutter opens upon initiation of a set of exposures and closes upon the end of collection. Status lamps on the shutter housing indicate when the shutter is open (green) and closed (red). The shutter is also interfaced to the controller and to the safety interlocks.

A tunable graphite crystal monochromator selects only the K_{α} line (λ =0.71073Å) emitted from the Mo X-ray source and passes it down the collimator system.

SMART Rotary Shutter and Collimator

The monochromatic X-ray beam then passes through the labyrinth, the SMART rotary shutter, and the incident beam collimator before striking the specimen.

- The labyrinth is a spring-loaded device, which ensures that the collimator and the SMART shutter are tightly connected to prevent X-ray leakage.
- The SMART shutter is a device which precisely controls the exposure time for each frame during data collection. Its

status lamps indicate when the shutter is open (ON) and closed (OFF). This assembly also houses an automatic attenuator.

- The incident collimator is equipped with pinholes at both front (near crystal) and rear (near source). These pinholes help to define the size and shape of the incident X-ray beam that strikes the specimen. You will normally use a collimator with 0.5 mm pinholes. (Collimators are available in a variety of sizes, depending on your application.)
- The beam stop (a hook-like assembly attached to the collimator) catches the remainder of the direct beam after it has passed the specimen. The beam stop has been aligned to minimize scattered X-rays and to prevent the direct beam from hitting the detector.

The entire collimator assembly is supported by a collimator support assembly, which has been precisely aligned to guarantee that the X-ray beam passes through the center of the goniometer.

SMART APEX detector

The SMART APEX detector is specific to this system. It is mounted on a 20 dovetail track. The track has a scale that is calibrated to indicate the distance from the crystal to the phosphor window (a typical distance is 6 cm). Status lamps on the detector housing indicate when the detector is on (green) and off (red).

K760 X-ray generator

The K760 X-ray generator is a highfrequency, solid-state X-ray generator, which provides a stable source of power for operations up to 60 kilovolts (kV) and 50 milliamps (mA). For the SMART APEX system, power settings should never exceed the maximum power rating of the X-ray tube. (Typical maximum power settings for the SMART APEX system with a normal focus tube are 50 kV, 40 mA. The kV setting should not exceed 50 kV.) This generator is interfaced to the controller, and the power settings may be adjusted either from front panel buttons or from within the SMART software.

Manual control module

The manual control module is a remote device that you will use in certain operations to manually drive angles (particularly in optical alignment of a specimen). The module is physically the same as in other D8 systems but has a different keypad and functions. In this application, you will use only the first three rows of buttons and the AXIS PRINT button.

Video camera

The video camera, an essential part of the system, allows you to visualize the crystal to optically align it in the X-ray beam and to measure the crystal dimensions and index crystal faces. The camera is interfaced to the computer and is operated through the VIDEO program. The camera is mounted in the accessories track of the goniometer base.

3.3 Accessories

Various devices can be mounted in an accessories track on the goniometer base. These include an optional low-temperature attachment.

4 Data Collection

We are now ready to begin actual operation of the instrument, using the YLID test crystal (mentioned in section 1). We assume that your system manager has set up the system properly and that all system default parameters have been set appropriately.

At this time, double click the BrukerAXS Programs icon on the Windows NT desktop (Figure 4-1).

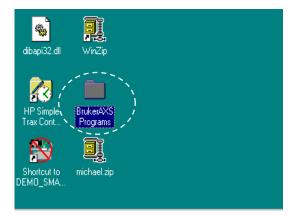


Figure 4-1. Bruker AXS programs icon on desktop

The BrukerAXS Programs window appears (Figure 4-2), containing icons for the Bruker AXS Program modules described in Section 2.

🖻 C:\\/INNT\Profiles\All Users\Start Menu\... 📃 🗖 🗙 <u>File E</u>dit <u>V</u>iew <u>H</u>elp ß M 斛 SMART Shortcut to SMART video D8tools.exe off-line 12 ß 100 (ASTRO) RLatt COSMO GEMINI SHELXTL SAINTPLUS 4.51KB 10 object(s)



4.1 Activate the Video Program

Before starting the SMART program, you must first activate the video camera as follows:

1. Double-click the Video (binoculars) icon (recall Figure 4-2) to start the Video program and display the main window (Figure 4-3).

NUDEO for Windows NT	_ 🗆 🗵
<u>File View Help</u>	
	+ II + A 8
Ready	[???,?] R:?, G:?, B:? Thu, 08 Jun 00 09:40:54 AM //

Figure 4-3. Video program's main window

 Click File > New Image to open a new file (Figure 4-4).

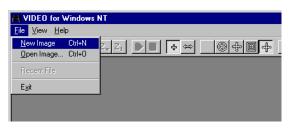


Figure 4-4. Create new video image

4-2

3. Then press the green arrow button in the toolbox to start the video frame grabber (Figure 4-5).

ie Edit Grab View Iools Window Help □ 22 A Z, Z, Z, I ■ II I II	
	_
image1	
eady (470,91) R:0, G:0, B:0 Fri, 09 Jun 00 (07:46:45.4	M

Figure 4-5. Start video frame grabber

The video camera is now ready for later use. Continue now to the SMART Program section of this manual.

4.2 Activate the SMART Program

Activate the SMART program as follows:

 Double-click the SMART (goniometer) icon to start the SMART program. SMART establishes communication with the D8 controller, the SMART APEX detector, and the X-ray generator. Then it displays its main screen without unit cell information (Figure 4-6).

ie -	<u>E</u> dit	<u>C</u> rystal	Acquire	Analyze	Goniom	Detector	Level	∐ser	Help			
										WorkDev	wtrlooß d: wtrlooß d: wtrlooß\	488 447 406 366 325 284 244
										2-Theta Omega Phi	0.00 0.00 0.00	203 163 122
										Chi Shutter Distance FloodFld	54.80 CLOSED 6.000 4004L170	81
										Spatial Dark Size,Spd	LINEAR 4004L030 512 400	

Figure 4-6. Main screen without unit cell information

Then SMART reminds you where you last worked and displays the next message (Figure 4-7).

SMART	V5.52 🛛 🕅
ৃ	Last project was in d:\frames\mylid1 Return to this project?
C	Yes <u>N</u> o

Figure 4-7. Return to project

2. Press the Yes button (normal response). The program then informs you that it has changed directories and asks for confirmation to continue (Figure 4-8).

SMART V5.52 🔀					
•	Current, working directory is now: d:\frames\mylid1				

Figure 4-8. Confirmation of current directory

3. Press OK (normal response). The program then communicates with the instrument, then loads the calibration files and parameters from the previous project (Figure 4-9).

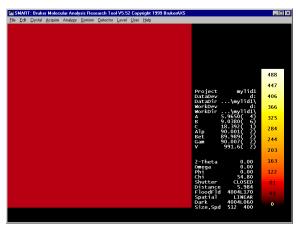


Figure 4-9. Screen showing data from previous project

Note: While presenting data (handshaking), the menu headings appear grayed (inactive). Once all data has been displayed (handshaking finished), the menu headings return to black (active).

 Click Crystal > New Project to start a new project. A new project options window appears (Figure 4-10).

Options for Crystal > New Project		×
Crystal Name (32-X chars)	NewProject	
Crystal Number (up to 4 digits)	1	
Title	?	
Chemical Formula	?	
Crystal Morphology	?	
Crystal Color	?	
Maximum Dimension	?	
Intermediate Dimension	?	
Minimum Dimension	?	
Collection Temperature	?	
Measured Density	?	
Density Method	?	
Working Directory		
Data Directory		
Backup Work Directory?	Check for yes	1
OK Cancel		

Figure 4-10. New project options window after entering information

5. Fill in all available information as shown (Figure 4-11). Note that you must specify a Project Name (lines 1 and 2) and a Working Directory and a Data Directory (which may be the same). You should also enter additional information specific to your specimen so that it will appear in the final structure report. When you are finished, click OK.

Options for Crystal > New Project	Options for Crystal > New Project					
Crystal Name (32-X chars)	YLID					
Crystal Number (up to 4 digits)	1					
Title	YLID Test Crystal					
Chemical Formula	cC11 H10 O2 S					
Crystal Morphology	Sphere					
Crystal Color	Pale Yellow					
Maximum Dimension	0.36					
Intermediate Dimension	?					
Minimum Dimension	?					
Collection Temperature	23					
Measured Density	?					
Density Method	?					
Working Directory	d:\frames\ylid1\					
Data Directory	d:\frames\ylid1\					
Backup Work Directory?	Check for yes					
OK Cancel						

Figure 4-11. Options window

The following save message appears (Figure 4-12).

SMART V5.52						
?	Save the current configuration?					
	Yes No					

Figure 4-12. Save the current configuration

 Press Yes. Another message prompts you to create the new directory (Figure 4-13). The message will appear twice if the working directory and the data directory are not the same.

SMART V5.52					
?	d:\frames\ylid1 does not exist. Create it ?				
	<u>Yes</u> <u>N</u> o				

Figure 4-13. Create the new directory

7. Click Yes (normal response). SMART then asks you to load the system default settings (Figure 4-14).

PROJECT DEFAULTS	×
Select defaults for new project	
Use current settings	
Small Molecule Protein	
OK Cancel	

Figure 4-14. Project defaults

 Select Small Molecule, then press OK. SMART loads the system defaults and displays a SMART screen. This screen will contain the sample-to-detector plane distance, the current goniometer angle settings, current dark and flood files, and information concerning your project and its working and data directories. No unit cell information displays. (Figure 4-15.)

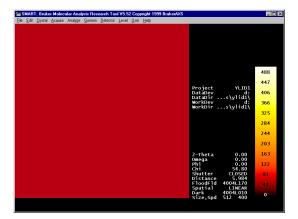


Figure 4-15. SMART screen with loaded data

 Click Level > Level1 (Figure 4-16). This level has a minimum number of options for routine problems, such as YLID, and will be used for this tutorial experiment. Level2 has more crystallography options required for special problems. Level3 is designed for use by a system manager only.

न S	MAR	F: Bruke	er Molec	ular Anal	ysis Res	earch Too	l V5.52 Cop	right 1999 Br	ukerAXS
<u>F</u> ile	<u>E</u> dit	<u>C</u> rystal	<u>A</u> cquire	Analyze	<u>G</u> oniom	Detector	Level User	<u>H</u> elp	
							✓ Level <u>1</u> Level <u>2</u> Level <u>3</u>	Ctrl+1 Ctrl+2 Ctrl+3	
							<u>C</u> ommand L	ine Ctrl+K	

Figure 4-16. Level menu

 Click Goniom > Zero to drive all angles to zero, and check the goniometer scales to ensure that all angles read zero. The following drive message appears (Figure 4-17).

SMART V5.52		
?	Drive all angles to zero: are you sure?	
	<u>Yes</u> <u>N</u> o	

Figure 4-17. Drive all angles to zero

The system is now ready for you to begin the experiment.

CAUTION: Use extreme care when handling the goniometer head to prevent damage to your expensive sample on the end of the small glass fiber.

- 11. Carefully remove from its case the goniometer head containing the YLID test crystal.
- Place the goniometer head onto its base on the phi (φ) drive, aligning the head's key slot with the key (pin) in the base. Snugly screw the head's collar to the base such that the head does not move, but do not overtighten it.

Note: At $\phi=0^\circ$, the key on the mounting base of the goniometer head will be at the 12:00 position.

 Click Crystal > Generator to check the Xray power. The Goniometer /Generator Options panel appears (Figure 4-18).

GONIOMETER /GENERATOR options		×
kV	50	Т
mA	30	
Wait (Y/N)	Check for yes	
OK Cancel		

Figure 4-18. Goniometer /Generator Options panel

14. Set appropriate values for kilovolts (kV) and milliamps (mA). For this experiment, we want 50 kV and 30 mA. Click OK to program these new settings.

4.3 Optically Align the Sample

To obtain accurate unit cell dimensions and to collect good quality data, you must align the center of the sample with the center of the Xray beam and maintain the alignment for the entire experiment. We assume that your video camera has been aligned so that the crosshairs of the video camera coincide with the center of the goniometer and the center of the X-ray beam.

Align the sample as follows:

 Click Crystal > Evaluate to begin the alignment of the sample in the center of the X-ray beam.

The alignment process combines optical alignment steps with rotational photo steps. The menu bar at the top of the SMART screen remains gray (Figure 4-19) until you complete the optical alignment step and exit by pressing the ESC key.

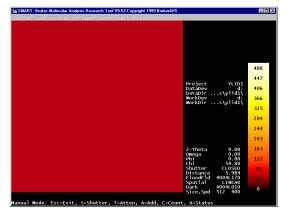


Figure 4-19. Grayed SMART screen menu bar during optical alignment

During the first stage (optical alignment), you will control the instrument from the manual control module (Figure 4-20) using buttons A and B.



Figure 4-20. Manual control module

Also during optical alignment, you will adjust the goniometer head at screw locations shown in Figure 4-21. Use the goniometer wrench to unlock the axis adjustment locks and later lock them. Use the other end of the wrench to turn the adjustment screws.

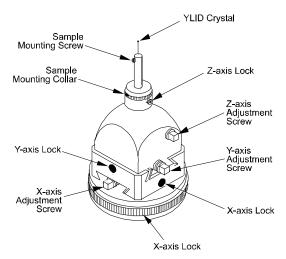


Figure 4-21. Goniometer head adjustment locations

 Click on the Video window to display the real-time image of the crystal (Figure 4-22).

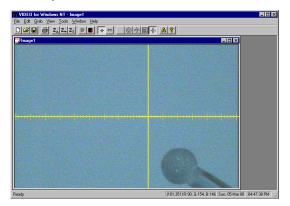


Figure 4-22. Real-time image of the crystal

3. Press the A button on the manual control module to drive the goniometer angles to the base position $(2\theta = -30^\circ, \omega = -30^\circ)$ and $\phi = 0^\circ$ for optical alignment.

Note: If you have difficulty seeing the image of the crystal, you may want to better illuminate the sample with a high-intensity lamp and/or temporarily place a light-colored piece of paper on the front of the detector.

4. Adjust the goniometer head's Z-axis adjustment screw (recall Figure 4-21) until the crystal is near the crosshairs intersection on the video screen (Figure 4-23).

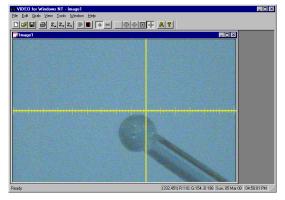


Figure 4-23. Crystal near the crosshairs intersection

5. Adjust the goniometer head's X-axis adjustment screw to center the crystal on the crosshairs intersection (Figure 4-24).

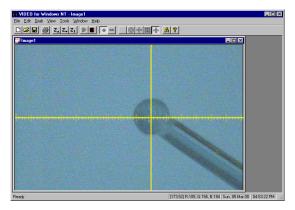


Figure 4-24. Crystal centered on crosshairs ($\phi = 0^{\circ}$)

6. Press the A button again to rotate the phi by 180° and to verify that you have correctly centered the sample at both φ =0° and φ =180°. (Do not be concerned if the crystal moves away from its center as it rotates; you have not yet adjusted the Y axis.)

Note: Repeat this process as many times as necessary. Each time you press the A button, the phi angle will rotate between 0° and 180°.

 Press the B button to rotate the phi angle to 90° for adjustment of the Y axis (Fig 4-25).

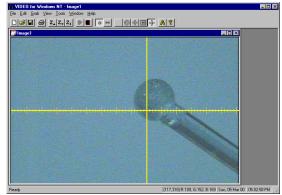


Figure 4-25. Crystal oriented for Y-axis adjustment

8. Adjust the goniometer head's Y-axis adjustment screw (Figure 4-19) to center the crystal on the crosshairs intersection (Figure 4-26).

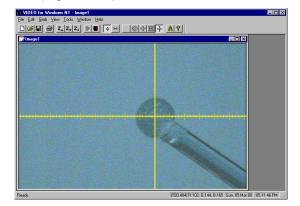


Figure 4-26. Crystal centered on crosshairs ($\phi = 90^{\circ}$)

9. Press the B button again to rotate the phi angle to 270° and to verify that you have correctly centered the sample at both ϕ =90° and ϕ =270°.

Note: Repeat this process as many times as necessary. Each time you press the B button, the phi angle will rotate between 90° and 270°.

The center-of-mass of a properly aligned specimen should stay in the same place with respect to the center of the crosshairs in all angle settings.

Note: If the video camera has become misaligned, the center of the crosshairs may need to be adjusted by your system manager.

- 10. Cycle between the two positions of both A and B one last time to ensure that the crystal remains centered in all positions.
- 11. After the alignment has been completed using A and B positions, you should verify the alignment by using the C and D buttons in a similar manner.
- 12. When you have completed the optical alignment steps, remove any paper you might have placed on the detector face and close the doors of the enclosure.
- 13. Click on the SMART program window, then press the ESC key to exit the optical alignment stage. SMART then prompts for a rotation photograph (Figure 4-27).

Figure 4-27. Prompt for a rotation photograph

14. Press Y (normal response). SMART will then perform a 60-second rotation photo. During this time, phi data on the screen will be highlighted and the shutter data will read: OPEN. Upon completion of the photo, the image frame will display on the screen (Figure 4-28).

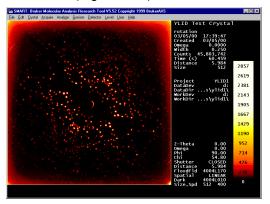


Figure 4-28. Image frame following 60-second rotation

At this time, proceed to Unit Cell Determination.

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4-11

4.4 Unit Cell Determination

Perform a unit cell determination as follows:

 From the SMART program, click Crystal

 Unit Cell. The following message appears, asking you to verify that the distance and beam center are correct (Figure 4-29).

SMART V5.52				
?	CONFIGURE/EDIT Dist,Xc,Yc = 6.0, 261.1, 250.8. Is this correct?			
	<u>Yes</u> <u>N</u> o			

Figure 4-29. Distance and beam center verification

2. Ensure that the distance shown on the detector scale agrees with the value shown on the screen. Then press Yes.

SMART then displays a set of default values for unit-cell determination and asks you to confirm them (Figure 4-30).

Options for Crystal > Unit Cell	×
# Frames	20
Frame width	-0.3
Seconds/frame	10
Title	Unit Cell Determination
Job name	matrix
Max display counts	-1
Lower axial limit	3.0
Upper axial limit	200.0
Indexing HKL tolerance	0.2
LS RLV tolerance	0.01
OK Cancel	

Figure 4-30. Set of default values for unit-cell determination

 Press OK (as these values are appropriate for this crystal). The program then collects three sets of 20 frames called MATRIX0, MATRIX1, and MATRIX2 (Figure 4-31).

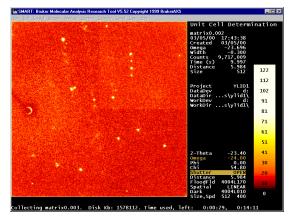


Figure 4-31. Frame collection (MATRIX0, MATRIX1, and MATRIX2)

When all frames have been collected, SMART performs the steps of thresholding, indexing, Bravais lattice determination, and least-squares refinement of cell constants. The YLID test crystal has an orthorhombic primitive unit cell, as shown in Figure 4-32.

Least Squares Output	×
Orientation Matrix: -0.00199829 0.10665119 0.01465839 0.11306161 -0.02113262 0.03875523 0.12383696 0.02125401 -0.03511810	
Lattice parameters & Standard deviations: 5.9631 9.0267 18.4111 89.980 90.022 90.091 991.02 0.0007 0.0031 0.0042 0.020 0.017 0.020 0.48 Standard deviations corrected for GOF:	
0.0014 0.0058 0.0077 0.037 0.032 0.036 0.89 Histograms:	
.00 .05 .10 .15 .20 .25 .30 .35 .40 .45 +Inf H 85 0 0 0 0 0 0 0 K 85 0 0 0 0 0 0 0 L 83 2 0 0 0 0 0 0 Omega 76 6 1 0 2 0 0 0	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	
H K L Omega X Y	•
OK Print Write	

Figure 4-32. Least-squares output

The YLID test crystal should have an orthorhombic Primitive cell with approximate cell dimensions of <u>a</u>=5.95Å, <u>b</u>=9.03Å, <u>c</u>=18.38Å, and α = β = γ =90°.

You are now ready to collect data.

4.5 Data Collection

Perform data collection as follows:

From the SMART program, click Acquire
 > Hemisphere. SMART displays the data
 collection options (Figure 4-33).

SCAN /HEMISPHERE Options	×
Job name	YLID1
Title	YLID Test Crystal
Max display counts	4
Suppress correlation (Y/N)	Check for yes
Suppress bias detn. (Y/N)	Check for yes
XENGEN output format (Y/N)	Check for yes
Sequence # of starting run	1
Sequence # of ending run	9999
Oscillate (Y/N)	Check for yes
OK Cancel	

Figure 4-33. Data collection options

2. Enter the job name, then press OK to begin data collection (Figure 4-34).

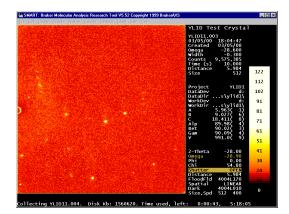


Figure 4-34. Data collection

The hemisphere data set specified in this example will require about four hours to collect. Upon completion of data collection, the following message displays (Figure 4-35).

SMART V5.52 X		
⚠	No more runs in EditRuns array	
	OK	

Figure 4-35. No more runs in Edit/Runs array

SMART V5.52		
?	Drive all angles to zero: are you sure?	

Figure 4-36. Drive all angles to zero

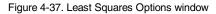
4. Press Yes.

4.6 Least Squares Refinement

Before leaving the SMART program, you might want to improve the orientation matrix that will be used for integration. Do so as follows:

 Click Crystal > LS to further perform the least squares refinement. The Least Squares Options window appears. (Figure 4-37).

east Squares Op	otions (center at	261.5 254.2 of a 512x5	i12 frame)		
Output P <u>4</u> P file	matrix0.p4p				
Co <u>n</u> straint	5 Ortho	▼ Max RL <u>V</u> error	0.01	Constraint	<u>m</u> ask: 512
Unit Cell	-2 Mono C -3 Mono B	-			
<u>A</u> axis	-4 Mono A	<u>B</u> axis	9.005	<u>C</u> axis	18.404
Alpha	-5 Ortho -6 Tetrag	Beta	90.000	<u>G</u> amma	90.000
	-7 Hexag	•			·
Detector Corr	ections				
⊻ beam cente	er -0.093	Y beam center	-0.006	Distance cor.	-0.005
Detector pitch	0.359	Detector <u>r</u> oll	-0.388	Detector yaw	-0.240
Eulerian angle	1 68.886	Eulerian angle ;	2 50.016	Eulerian angle ;	3 -104.522
Crystal X-trans	0.0000	Crystal Y-trans	0.0000	Crystal Z-trans	0.0000
<u>O</u> mega zero	0.0590	Ch <u>i</u> zero	0.1365	Frame half <u>w</u> idth	0.15
ОК	Cancel				



The YLID crystal is orthorhombic and, therefore, the alpha, beta, and gamma angles should be 90°. You may constrain them to be so with the Constraint field of the Least Squares Options window.

2. Change the Constraint field to Ortho and press OK. The program may remove a few of the poorest fitting reflections. The following removal message appears (Figure 4-38).

SMART V	SMART V5.52			
?	3 reflection(s) removed from LS, because HKL's were not integers within tolerance			
	OK Cancel			

Figure 4-38. 3 reflections removed from LS

 Press OK. SMART displays a Least Squares Output window showing, among other data, a histogram for the reflections and the unit cell parameters with errors (Figure 4-39).

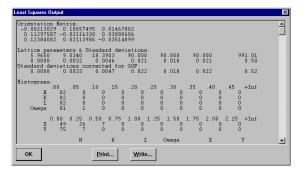


Figure 4-39. Histogram for reflections and unit cell parameters with errors

If the refinement process was done correctly, the histogram will show zeros in all columns except the first.

4. Press OK. A message asking you to overwrite the default file for refined unit cell parameters (matrix0.p4p) appears (Figure 4-40).

SMART V5.52		
?	Overwrite d:\frames\ylid1\matrix0.p4p?	
	Yes No	

Figure 4-40. Overwrite default matrix0.p4p file

5. Press Yes. You have now completed the least-squares refinement process.

 Click File > Exit to leave the SMART program. The exit message appears (Figure 4-41).

SMART	V5.52 🛛 🕅
?	EXIT: are you sure?
	Yes <u>N</u> o

Figure 4-41

7. Press Yes. The save message appears (Figure 4-42).



Figure 4-42

8. Press Yes.

This completes the data collection process.

M86-E02015 -0800

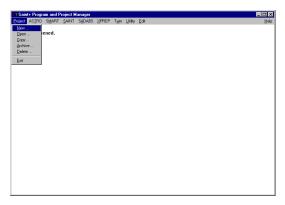
4-16

5 Data Integration

Before the data can be used to solve and refine the crystal structure of the YLID, you must convert the frame data produced by SMART to a set of integrated intensities. To do so, you must run the SAINT program. SAINTPLUS helps you set up the SAINT input parameters.

Integrate the data set as follows:

- Click the SAINTPLUS icon in the Bruker AXS programs folder to start that program menu (recall Figure 4-1).
- 2. Click Project > New to create a new project (Figure 5-1).



3. Assign a name to the project. Then use the built-in explorer to locate the data directory used by the SMART program (Figure 5-2).

Dpen a New Project	? ×
Project name YLID1	
Crystal Type C Protein C Small Molecule	
C Eloten C Sitial Molecule	
Use the file locator to help identify frames for SAINT to process. Click on a .p4p or	
frame filename with the base name of the series of frames you wish to process.	
Look jn: 🔄 frames 💽 🗈 📰	
i poly4 i semo5 i yiid1 Projects i stu3 i yiid6	
in recon in skb50 in ylid8 in sample1 in skb60	
sample2 itest semo4 invitioo8	
۲	
<u>O</u> pen	
Files of type: *,p4p files Cancel	
Erames path	
Dutput directory D:\work\	

Figure 5-2. Assign a name and locate the directory

Figure 5-1. Create a new project

4. Select one of the .P4P files (i.e., not a MATRIX file) from the full data set. Then click open (Figure 5-3).

Open a New Project	? ×
Project name YLID1	
Ciystal Type C Protein C Small Molecule	
Use the file locator to help identity frames for SAINT to process. Click on a ,p4p or frame filename with the base name of the series of frames you wish to process.	
Look jn: 🔄 yiid1 💽 💽 🕅	
mahrixipAp MyLD14,p4p mahrixipAp mahrixipAp mahrix2p4p	
@ YUD11.p4p @ YUD12.p4p @ YUD13.p4p	
Open Files of type: * p4p files Cancel	
Erames path D:\frames\yiid1\YLID1.*	
Qutput directory D:\frames\ylid1\work\	

Figure 5-3. Select a .P4P file

A summary window appears, showing the name and directories used by the project (Figure 5-4).

		am and P							
roject	ASIRO	SMART	SAINT	SADABS	WPREP	T <u>w</u> in	Utility	Edk	He
ame:	YLID1								
rystal	I Type:	Small N	Aolecul	e					
rames	s Direc	tory Pat	h: D:\fr	ames\yli	d1\YLID	1.*			
utout	Directo	orv: D:\fr	ramest	ylid1\wor	к				
		,,.		, ,					

Figure 5-4. SAINTPLUS project summary window

- 5. Click SAINT > Initialize to initialize the project.
- 6. Click SAINT > Execute.

An input box appears, summarizing the information that SAINTPLUS has determined (Figure 5-5).

Basic SAINT menu for analyzing :	small molec	ule area	detector frar	nes					×
Title YLID Test Crys	tal								More options
Laue class mmm orthorhon	mmm orthorhombic: a,b,c; 90,90,90								Integrate
Lattice centering P (primitive)									<u>S</u> ort
Resolution limit for output				⊢ Cell pa	arameters —				Global
C 2*theta (degrees)				A	5.9647	Alpha		90.000	
Sin(theta)/lambda (1/angstroms)) [.75	_	В	9.0049	Beta		90.000	<u></u> ilter
 d-spacing (angstroms) 				С	18.4035	Gamm	na	90.000	I <u>n</u> strument
Integration files									
Maximum wait for frame file (second	s) 0.000000								
Starting Frame Filename	:	# of Frame	s Matrix	(.p4p) File	ename				Output Filename
D:\frames\ylid1\YLID11.001	Browse	600	D:\frames\yli	d1\matrix0	l.p4p	Browse	Cell	D:\frames'	\ylid1\work\YLID11.raw
D:\frames\ylid1\YLID12.001	Browse	435				Browse	Cell	D:\frames'	\ylid1\work\YLID12.raw
D:\frames\ylid1\YLID13.001	Browse	230				Browse	Cell	D:\frames	\ylid1\work\YLID13.raw
	Browse	0				Browse	Cell		
	Browse	0				Browse	Cell		
	Browse	0				Browse	Cell		
More integration files	Incremen	t last run	j						
Integrate + Sort + Global	<u>V</u> alidate		<u>O</u> pen listing	ı file				<u>H</u> e	elp <u>C</u> lose

Figure 5-5. Information that SAINTPLUS has determined

7. Check to see that the values for your data set match those shown. Change any parameters that do not match. In particular, change the input file to matrix0.p4p for the first run, and delete the .p4p filename for subsequent runs. A resolution of 0.75 (d-spacing) should be used for a sample-to-detector plane distance of 6.00 cm.

8. Click the Integrate button to display the parameters for integration (Figure 5-6). Check to see that the values for your data set match those shown. Change any parameters that do not match.

Integrate		×
Reflection size X size (degrees) 0.60000 Y size (degrees) 0.60000 Z size (degrees) 0.30000 Image: size (degrees) Image: size (degrees) Image:	Periodic orientation matrix updating ✓ Enable periodic updating Periodic updating frequency 100 Constraints ✓ Constrain integration by Laue class Crystal system Orthorhombic (a,b,c; 90,90,90) ✓ Detector center X Detector center X Detector pitch Detector distance Utit cell available	Post-integration global (all data) refinement Enable global least squares refinement Limit on number of reflections to refine 9999 Constraints Constraints Constrain refinement by Laue class Crystal system Orthorhombic (a,b,c; 90,90,90) Detector center X Detector center Y Detector pitch Detector roll Detector yaw Detector distance
More integration options Integration <u>Files</u> <u>A</u> dvanced Integrate Integrate + Sort + Global	□ Unit cell axes □ Unit cell angles □ Goniometer zeros □ Crystal translations □ Continuous crystal and detector orientation updating □ Enable continuous updating □ Damping factor X: P' = P_+ P - P_s 1.000000	Unit cell axes Unit cell angles Goniometer zeros Crystal translations Post-integration sorting and filtering ✓ Sort by Laue class ✓ Point group mmm_orthorhombic Minimum I/sigma(I) to output -3.000000 ✓ Enable correlation filter Help _lose

Figure 5-6. Integrate input window

 Click the Advanced Integrate button to display the Advanced Integrate input box (Figure 5-7). Compare the values for your data set with those shown. Make required changes.

Advanced Integrate	×
Model profiles	Beam monitor (synchrotron systems)
I/sigma lower limit for reflections used to update model profiles	Enable beam monitor normalization
Fraction of model profile maximum used to generate simple sum limits 0.050000	Normalize all runs to the first
I/sigma threshold for least squares fit (below) vs. simple sum (above) 4.000000	Multiwire detectors
Resolution lower limit (A) above which simple sum is always used 9999.0000	
🔽 Blend 9 profiles	Output listing and diagnostic files
	Generate diagnostic plot files
Background and Active Mask	Keep temporary files
Base 2 log of background update scaling factor X: b=b,+(b-b,)/(XN) 0	Append listing file
Use pre-existing active pixel mask	Verbosity of listing file (1-24)
Fractional lower limit to generate active mask (fraction of average intensity) 0.000000	Background file output frequency 100
Active mask (am) file Browse	Corrections to intensity esd's
	Instrument error (fraction of intensity) 0.000000
Spatial correction	Factor multiplying intensity esd's 1.000000
Apply spatial calibration correction from separate indexed fiducial (ix) file	Frames stored to monitor reflection overlap
Spatial (_ix) file Browse	Active frame queue half-width 7
Override selected input frame header information	Crystal
Overide frame header Scan axis Frame width	Starting exposure time (hours) 0.000000
2-theta Omega Phi Chi	Batch # 1 Crystal # 1
Integrate + Sort + Global ⊻alidate Dpen listing file	<u>H</u> elp <u>C</u> lose

Figure 5-7. Advanced Integrate input window

 Click the Integrate + Sort + Global button to execute the SAINT program. The integration process takes about 15 minutes, displaying data as shown (Figure 5-8).

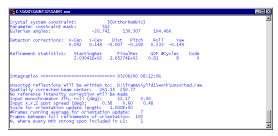


Figure 5-8. Integration data

 When the integration process has finished, the results of the global refinement of unit cell parameters display (Figure 5-9).

C:\SAXI\SAINT32\SAINT.exe	
Performing final unit cell least squares on file D:\frames\ylid1\work\YLID11ma Input file contains 5346 reflections Maximum allowed reflections = 9399 Dne reflection will be stored in memory per 1 reflections read	-
Performing orientation least squares (5346 reflections) Drientation ("UB") matrix: -0.113024 -0.0217837 -0.0389220 -0.0017302 -0.1667011 0.0149414 -0.124528 0.021237 0.0351184	
A 8 C Alpha Beta Gamma Vol 5.9460 9.0125 18.3448 90.000 90.000 90.000 98.007 0.0003 0.0004 0.0009 0.001 0.001 0.001 0.13 Corrected for goodness of fit:	
0.0003 0.0005 0.0010 0.001 0.001 0.001 0.16	
Range of reflections used: Worst res Best res Min ZTheta Max ZTheta 9.1724 0.7496 4.441 56.600 Crystal system constraint mask: 512 Parameter constraint mask: 512 Eulerian angles: 68.999 49.891 -104.504 Goniometer zeros (deg): 6.0000 0.1185 0.0000 -0.0013 Crystal translations (pixels): 0.0000 0.0000 0.00000	
Detector corrections: X-Cen Y-Cen Dist Pitch Roll Yaw 0.721 -0.097 -0.013 0.332 -0.305 -0.210	
Refinement statistics: StartingRes FinalRes GOF #Cycles Code 2.04334E+05 2.34486E+04 1.46 3 0	
New orientation is in D:\frames\ylid1\work\YLID1m.p4p End final global unit cell least squares refinement ======= 08/08/00 13:48:31	
Press ENTER to continue	-

Figure 5-9. Integration summary

12. Before pressing the Enter key, scroll up the screen to display the summary table from the integration process (Figure 5-10).

🏀 C:\SAXI\S	AINT32\	SAINT.e	xe														_	
24.000 25.000	0	0	0	0	0.0	0.000	0.00	0.00	0.000	0.000	0.0	0.000	0.000	0.00	00	00	00	00
0verall 0.000 1.000	# 7061	Pairs 1279	Uniq 1454	Merg 7027		<i> 1704.109</i>	<#Sig> 35.81			dI/I 0.000								
Centric 0.000 1.000	# 1268	Pairs 280	Uniq 452			<i> 2508.639</i>				dI/I 0.000								
overage St. Angstrms to 1.616 to 1.283 to 1.120 to 1.018 to 0.845 to 0.845 to 0.845 to 0.845 to 0.777 to 0.750 weraged or -0.113100 -0.001607 -0.001607	#Obs 176 325 471 612 762 901 1035 1185 1318 1454 ocal " c ocal re ientati 2 -0.0 1 -0.1 8 0.0	Theory 178 328 476 618 765 906 1040 1189 1323 1454 ell lea finemer on ("UE 218930 067129 212608	ts aver -0.038 0.014 0.035	5.65 5.88 5.79 5.67 5.49 5.25 5.22 5.09 4.98 4.86 res ret aged: ix: 9057 9832 1417	0.02 0.02 0.02 0.02 0.02 0.02 0.02 0.02	2 282 3 412 4 538 5 668 5 792 6 913 6 1045 6 1164 7 1279	Pairs Rs 33.71 0 35.98 0 36.55 0 37.06 0 37.32 0 37.42 0 37.42 0 37.79 0 37.98 0 37.98 0	shell #S 0.020 9 0.027 3 0.037 2 0.037 1 0.040 1 0.047 1 0.047 1 0.055 0.059 0.070	2.04 3 3.46 1 5.57 3 2.22 6 3.52 7 3.52 7 3.52 11 2.72 11 5.89 16 5.70 23 4.75 19	2s .5 .9 .7 .6 .0 .8 .2 .3 .4	,							
eighted ave A 5.9440 0.0004 td deviatio	9.01 0.00	B 00 18 06 0	.3419 .0015 1 input	Alph 90.00 0.00)O)3	Beta 90.000 0.003 sults:	Gamma 90.000 0.002		Vol 982.31 0.20									
0.0059 ange of ret	0.00 flectio		.0184 I:	0.00	0	0.000	0.000		1.58					_				

Figure 5-10. Viewing log files

Your results should be similar to those displayed above.

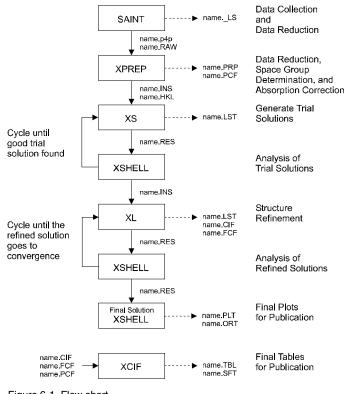
13. Press Enter, and close all SAINT windows and exit from the SAINTPLUS menu.

You are now ready to begin the structure determination and refinement process.

5-8

6 Structure Determination & Refinement

You are now ready to solve and refine the crystal structure for the YLID test crystal. The integration process (SAINT) has produced two important files—YLID1M.p4p, containing the final unit cell parameters and other important information on how the experiment was carried out, and YLID1M.raw, containing the actual intensity data. These files are all that is



required to begin the structure solution and refinement process. The various steps in solving and refining the structure are carried out using the programs of the SHELXTL package.

A simplified flow chart is shown in Figure 6-1.

Figure 6-1. Flow chart

Solve and refine the crystal structure as follows:

- Click the SHELXTL icon in the Bruker AXS folder to start SHELXTL (recall Figure 4-2).
- 2. Click Project > New to create a new project for the YLID (Figure 6-2).

🖾 Shelxtl Program											- D ×
Project XPREP XS	⊠SHELL	ХĿ	Х <u>Р</u>	X <u>w</u> AT	XP <u>R</u> O	XCIF	XEOG	XPOW	XPS	Edit	Help
<u>N</u> ew											
Open pene	d.										
Eopy Archive											
Delete											
_											
<u>E</u> xit											

Figure 6-2. Create a new project

3. Use the built-in Explorer to locate the YLID1m.raw file. In this example, the file is in D:\frames\ylid1\work (Figure 6-3).

Open a New Pro	ject									? ×
Project <u>n</u> ar	ne:	YLID1m					_			
Locate a .p	o4p or	data file to	associate wi	ith the new p	roject:					
Look jn:	6	ylid1			•	È	Ċ		J	
AYUD1 YUD1 YUD1 YUD1 YUD1	2.raw 3.raw m.p4p									
Files of typ	e: [*.p4p, *.hkl	, and *.raw f	iles		•		<u>O</u> pen Cancel		
Project pat	h:	D:\frames	vlid1\YLID1	m.*	_	_				

Figure 6-3. Locate the YLID1m.p4p file

The SHELXTL Program and Project Manager window appears (Figure 6-4).

Shelxtl Program and Project Manager
Project XPREP XS XSHELL XL XP XWAT XPB0 XCIF XEOG XPOW XPS Edit Help
Project name: YLID1m

Project path: D:\frames\ylid1\YLID1m.*

Figure 6-4. SHELXTL Program and Project Manager window

4. Click XPREP > XPREP (or XPREP > BigXPREP). The first XPREP window appears (Figure 6-5).

No. 1997 BIGXPREP - [V5.1 Copyright (c) 1997 Bruker AXS			
+ XPREP - DATA PREPARATION & RECIPRO	CAL SPACE EXPLORATIO	N Ver. 5.1/NT +	
+ COPYRIGHT(c) 1997 Bruker Analytics	+++++++++++++++++++++++++++++++++++++++	+++++++++++++++++++++++++++++++++++++++	
** Data multiplied by 0.1000 to brir 6498 Reflections read from file yl		ale **	
Mean (I/sigma) = 19.52	Iuim.Law		
Lattice exceptions: P & B		Obv Rev All	
N (total) = 0 3233 3249 N (int>3sigma) = 0 2840 2794	3254 3260 4868 2902 2852 4268	3768 3767 5669	
Mean intensity = 0.0 168.1 161.2 Mean int/sigma = 0.0 20.0 18.9	154.4 171.9 161.2 19.2 19.8 19.4	165.6 166.2 164.9 19.6 19.5 19.5	
Lattice type [P, A, B, C, I, F, O(obv	.), R(rev. rhomb. on	hex. axes)]	
Select option [P]:			

Figure 6-5. First XPREP window

5. The lattice is Primitive [P]. Press RETURN. The second XPREP window appears (Figure 6-6).

No. 1 Co	opyright (c) 19	197 Bruker A	XS]								_ 8
Current dataset:	ylid1m.ra	w		Wavele	ength: O.	71073 Ch	iral: ?				
Original cell: Esds:		.034 18 .001 0			90.00 0.00	Vol Lat	989.8 tice: P				
Current cell:	5.960 9	.034 18	.384 90.0	90.00	90.00	Vol	989.8				
Matrix: 1.0000 0	0.0000 0.0	0000 0.	0000 1.000	0.0000	0.0000	0.0000	1.0000	0			
[D] Read, modify [P] Contour PATTE [H] Search for HI [S] Determine or [A] Apply ABSORFT [L] Reset LATTICE Select option [H]	ERSON sect: [GHER metr: input SPA FION correct type of (ions ic symme CE GROUP ctions	[try [[(C) Define (F) Set up (R) RECIPRO (U) UNIT-CF (T) Change (Q) QUIT pr (Q) QUIT pr	shelxtl CAL spac CLL trans TOLERANC	FILES e displa formatio:	ys				

Figure 6-6. Second XPREP window

6-4

6. Press RETURN to execute the search for higher metric symmetry [H]. The program has determined that the YLID crystal has an orthorhombic primitive lattice (Figure 6-7).

Note: The second	1997 Bruker AXS]				
Current dataset: ylid1m.r	aw	Wavel	ength: O.	71073 Ch:	iral: ?
Original cell: 5.960 Esds: 0.000		90.00 90.00			989.8 Sice: P
Current cell: 5.960	9.034 18.384 9	90.00 90.00	90.00	Vol	989.8
Matrix: 1.0000 0.0000 0	.0000 0.0000 1.	0000 0.0000	0.0000	0.0000	1.0000
<pre>[D] Read, modify or merge [P] Contour PATTERSON sec [H] Search for HIGHER met [S] Determine or input SP [A] Apply ABSORPTION corr [L] Reset LATTICE type of Select option [H]: Determination of reduced Transformation from origi 1.0000 0.0000 0.0000 Unitcell: 5.960</pre>	tions ric symmetry ACE GROUP ections coriginal cell (Niggli) cell nal cell (HKLF-me	[Q] QUIT p: atrix): 00 0.0000	shelxtl OCAL spac ELL trans TOLERANC rogram 0.0000	FILES e display formation ES 0.0000 :	ys ns
Niggli form: a.a = b.c =	35.52 b.b = 0.00 a.c =	81.61 0.00			
Search for higher METRIC	symmetry				
Option A: FOM = 0.000 deg Cell: 5.960 9.034 1 Matrix: 1.0000 0.0000 0	8.384 90.00 9	90.00 90.00	Volum	e:	989.82
Option B retains original	cell				
Select option [A]:					

Figure 6-7. YLID crystal, orthorhombic primitive lattice

Press RETURN to select cell choice A. The next XPREP window appears (Figure 6-8).

👆 BIGXPREP - [¥5.1	Copyright (c) 1997 Bru	iker AXS]									_ 8
Current dataset	: ylid1m	n.raw			Wavele	ngth: O.	71073 Ch	niral: ?				
Original cell: Esds:	5.960 0.000		18.384 0.001	90.00 0.00	90.00 0.00	90.00 0.00	Vol Lat	989.8 tice: P				
Current cell:	5.960	9.034	18.384	90.00	90.00	90.00	Vol	989.8				
Matrix: 1.0000	0.0000	0.0000	0.0000									
Crystal system:	Orthorh	ombic		Lattice								
[D] Read, modifi [P] Contour PAT [H] Search for [S] Determine on [A] Apply ABSOR [L] Reset LATTI Select option [TERSON s HIGHER m r input PTION co CE type	ections etric sy SPACE GP prrectior	mmetry COUP 18	[F] [R] [U] [T]	Set up RECIPRO UNIT-CE	unit-cel shelxtl (CAL space LL trans TOLERANC ogram	FILES e displa formatic	iys				

Figure 6-8. Next XPREP window

6-6

 Press RETURN to choose the SPACE GROUP option [S]. SMART will then prompt you for four selections: determine space group [S], the crystal system [O], the lattice centering [P], and the space group [A] = P2₁2₁2₁. All prompts and data are shown in Figure 6-9.

Image: Space State Stat
[C] Must be CHIRAL (sample is optically active) [N] NOT NECESSARILY chiral (eg. may be racemate)
[N] NOT NECESSARILY chiral (eg. may be racemate)
[E] EXIT to main menu or [Q] QUIT program
[1] Inte of Math Metha of [4] gott program
Select option [S]:
[A] Triclinic, [M] Monoclinic, [O] Orthorhombic, [T] Tetragonal, [H] Trigonal/Hexagonal, [C] Cubic or [E] EXIT
Select option [0]:
Lattice exceptions: P & B C I F Obv Rev All
N (total) = 0 3233 3249 3254 3260 4868 4329 4326 6498
N (int>3sigma) = 0 2840 2794 2902 2852 4268 3768 3767 5669
Mean intensity = 0.0 168.1 161.2 154.4 171.9 161.2 165.6 166.2 164.9
Mean int/sigma = 0.0 20.0 18.9 19.2 19.8 19.4 19.6 19.5 19.5
Lattice type [P, A, B, C, I, F, O(obv.), R(rev. rhomb. on hex. axes)]
Select option [P]:
Mean $ E^*E-1 = 0.686$ [expected .968 centrosym and .736 non-centrosym]
Systematic absence exceptions:
b c n 21can21abn21
N 334 330 326 7 212 222 212 11 127 121 122 15
M = 334 = 330 = 326 = 7 = 212 = 212 = 11 = 127 = 121 = 122 = 15 M I>38 = 283 = 274 = 265 = 0 = 158 = 162 = 134 = 0 = 0100 = 100 = 94 = 1
M 1>38 263 274 265 0 136 122 134 0 100 94 1
$\langle I \rangle = 105 \cdot 0 \cdot 20 \cdot 0 \cdot 20 \cdot 0 \cdot 0 \cdot 0 \cdot 0 \cdot 1 \cdot 1 \cdot 27 \cdot 0 \cdot 15 \cdot 0 \cdot 0 \cdot 0 \cdot 0 \cdot 17 \cdot 1 \cdot 17 \cdot 15 \cdot 0 \cdot $
Option Space Group No. Type Axes CSD R(int) N(eq) Syst. Abs. CFOM
[A] P2(1)2(1)2(1) # 19 chiral 1 5917 0.032 5070 1.0 / 15.0 0.96
Select option [A]:

Figure 6-9. Next XPREP window

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6-7

 Press RETURN to choose option [A]. The next XPREP window appears (Figure 6-10).

Matrix: 1.0000 0.0000 0.0000 0.0000 1.0000 0.0000 0.0000 1.000	NAME AND A STATE A	in Cop	yright Bruk	er-AXS 200	0				_ 🗆 ×
Esds: 0.000 0.001 0.001 0.00 0.00 Lattice: Current cell: 5.951 9.021 18.361 90.00 90.00 90.00 Vol 985. Matrix: 1.0000 0.0000 0.0000 1.0000 0.0000 0.0000 1.0000 Crystal system: Orthorhombic Space group: P2(1)2(1)2(1) # 19 [chi] Laue:	Current dataset	: ylid1m.	hkl			Wavele	ngth: O.	71073 CH	niral: ?
Matrix: 1.0000 0.0000 0.0000 0.0000 1.0000 0.0000 0.0000 0.0000 1.000 Crystal system: Orthorhombic Space group: P2(1)2(1)2(1) # 19 [chi] Laue:	-								
Crystal system: Orthorhombic Space group: P2(1)2(1)2(1) # 19 [chi] Laue:	Current cell:	5.951	9.021	18.361	90.00	90.00	90.00	Vol	985.7
	Matrix: 1.0000	0.0000	0.0000	0.0000	1.0000	0.0000	0.0000	0.0000	1.0000
[D] Beed wodify or warge DiTASTTS [C] Define unit_cell CONTENTS	Crystal system:	Orthorho	ombic S	pace gro	up: P2(1)2(1)2(1) # 19	[chi]	Laue: 3
[P] Contour PATTERSON sections [F] Set up shelxtl FILS [H] Search for HIGHER metric symmetry [R] RECIPROCAL space displays [S] Determine or input SPACE GROUP [U] UNIT-CELL transformations [A] Apply ABSORPTION corrections [T] Change TOLERANCES [L] Reset LATTICE type of original cell [Q] QUIT program	 [P] Contour PAT [H] Search for [S] Determine o [A] Apply ABSOR 	TERSON se HIGHER me r input S PTION cor	ections etric sy SPACE GF crection	mmetry COUP IS	[F] [R] [U] [T]	Set up RECIPRC UNIT-CE Change	shelxtl CAL spac LL trans TOLERANC	FILES e displa formatio	ιys

Figure 6-10. Next XPREP window

10. Press RETURN to choose the read, modify, or merge datasets option [D]. The next XPREP window appears (Figure 6-11).

: XPREP	° ver 6.07/₩i	in Copyright E	ruker-AXS 2000		- 🗆
Index	# Data	Filename or	Source of D	ata	
	6860	ylid1m.hkl	<- current	dataset	
[M] Sor	rt-MERGE d	urrent data	(no scaling)	[C]	Change CURRENT dataset
[L] LIM	NEAR scale	e and merge d	atasets	[ឃ]	WRITE dataset to file
[I] IS(OTROPIC so	cale and merg	e datasets	[R]	READ in another dataset
[S] Dis	splay inte	ensity STATIS	TICS	[D]	DELETE stored dataset
[F] FAC	CE-indexed	absorption (corrections	[P]	PSI-scan absorption corr.
[T] Cor	py file, 7	FRANSFORM hkl	and cosines	[A]	MAD, SAS, SIR or SIRAS
[H] App	ply HIGH/1	low resolutio:	n cutoffs	[N]	NORMALIZE/scale sigmas
[E] EXI	IT to mair	n menu		[Q]	QUIT program
	option [2	_		[Q]	2011 program

Figure 6-11. Next XPREP window

11. Press RETURN to choose the display intensity statistics option [S]. The next XPREP window appears (Figure 6-12).



Figure 6-12. Next XPREP window

12. Press RETURN to choose the merge all equivalents including Friedel mates option [A]. The next XPREP window appears (Figure 6-13).

Compare data in the circled areas and note that the data set is virtually complete to 0.75 Å with an average redundancy of >4. The overall R_{merge} is 2.72%.

Resolution	#Data #	Theory	%Complete	Redundancy	Mean I	Mean I/	s Rint	Rsigma
Inf - 2.20	76	77	98.7	4.75	1104.3	138.74	0.0206	0.0054
.20 - 1.70			100.0	5.82	437.2	95.96	0.0206	0.007
.70 - 1.45	80		98.8	5.81	318.5		0.0247	0.010
			98.8			59.77	0.0296	0.014
			99.2	5.46	155.8	48.25	0.0368	0.017
		133	100.0	5.03	85.8		0.0348	0.025
.05 - 0.95	176	176	100.0	4.85		24.58	0.0390	0.034
.95 - 0.90			100.0	4.42		17.91	0.0450	0.047
.90 - 0.85	147	147	100.0	4.19	28.6	12.94	0.0537	0.064
.85 - 0.80		197	100.0	4.11	24.6	11.41	0.0606	0.074
.80 - 0.75	238	238	100.0	3.68	17.5	8.15	0.0763	0.105
			100.0	3.00	27.6	11.15	0.0196	0.089
.85 - 0.75	 436	436	100.0	3.87	20.7	9.63	0.0673	0.089
Inf - 0.75	1457	1461	99.7	4.70	150.5	35.99	0.0272	0.016
erged [A],	lowest	resolut	ion = 18.3	36 Angstroms	3. 35	7 outlie	rs downw	eighte

Figure 6-13. Next XPREP window

13. Press RETURN to continue displaying the window. The next XPREP window appears (Figure 6-14).

: dataset
g) [C] Change CURRENT dataset
[W] WRITE dataset to file
[R] READ in another dataset
[D] DELETE stored dataset
[P] PSI-scan absorption corr.
es [A] MAD, SAS, SIR or SIRAS
[N] NORMALIZE/scale sigmas
[Q] QUIT program
19 3

Figure 6-14. Next XPREP window

14. Press option [E] to return to the main window. The following window appears (Figure 6-15).

SREP ver 6.07/₩	/in Cop	yright Bruk	er-AXS 200	0				_ 🗆 ×
Current dataset	: ylid1m	.hkl			Wavele	ngth: O.	71073 Ch	niral: ?
Original cell: Esds:	5.951 0.000						Vol Lat	
Current cell:	5.951	9.021	18.361	90.00	90.00	90.00	Vol	985.7
Matrix: 1.0000	0.0000	0.0000	0.0000	1.0000	0.0000	0.0000	0.0000	1.0000
Crystal system:	Orthorh	ombic S	pace gro	up: P2(1)2(1)2(1) # 19	[chi]	Laue: 3
[D] Read, modify or merge DATASETS [C] Define unit-cell CONTENTS [P] Contour PATTERSON sections [F] Set up shelxtl FILES [H] Search for HIGHER metric symmetry [R] RECIPROCAL space displays [S] Determine or input SPACE GROUP [U] UNIT-CELL transformations [A] Apply ABSORPTION corrections [T] Change TOLERANCES [L] Reset LATTICE type of original cell [Q] QUIT program				iys				
Select option [C]:							

Figure 6-15. Next XPREP window

15. Press RETURN to choose the Define Unit Cell option [C]. The unit cell contents will be summarized on the next menu (Figure 6-16).

Figure 6-16. Summarized unit cell contents

6-12

- 16. If you did not enter the chemical formula when you created the new project for the YLID crystal in the SMART program, you must do so now. The correct chemical formula for the YLID crystal is C11 H10 O2 S. If the information is correct, press RETURN to display the next window (Figure 6-17).
- 17. Press RETURN to keep the current name, then type Y to write the YLID1m.HKL file.
- 18. You may now press RETURN, for option [E], to return to the main SHELXTL menu.

rrent dataset: ylidim.raw
iginal cell: 5.960 9.034 18.384 90.00 Esds: 0.000 0.001 0.001 0.00
rrent cell: 5.960 9.034 18.384 90.00
trix: 1.0000 0.0000 0.0000 0.0000 1.0000
ystal system: Orthorhombic Space group: P2(1
rmula: C11_H10_02_S 4.00 Density: 1.384 At.vol: 17.7 F(000
P1 Contour PATTERSON sections [F] [1] Search for HIGHER metric symmetry [R] [1] Determine or input SPACE GROUP [U] [1] Determine or input SPACE GROUP [U] [1] Apply ABSORPTION corrections [T] [1] Reset LATTICE type of original cell [Q] [2] clect option [F]: [Status of the second se

Figure 6-17. Next window

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6-13

The XPREP program has created an YLID1m.ins file and an YLID1m.hkl file (recall Figure 6-1). You are now ready to solve the structure of the YLID sample.

19. Click on the XS command at the top of the menu to launch the XS (structure solution) program (Figure 6-18).

MS C:\₩/INNT\System32\CMD.exe
104 Reflections and 1066. unique TPR for phase annealing 149 Phases refined using 2642. unique TPR 169 Reflections and 3344. unique TPR for R(alpha) 594 Unique negative quartets found, 594 used for phase refinement 141 Unique NQR employed in phase annealing 128 Parallel refinements, highest memory = 4261 / 44577
Try Ralpha Ngual Sigma-1 M(abs) CFOM Seminvariants 181393. 0.053 -0.775 0.967 1.122 0.053* ***********************************
Fourier and peaksearch RE = 0.130 for 14 atoms and 452 E-values Fourier and peaksearch RE = 0.118 for 14 atoms and 452 E-values Fourier and peaksearch
++++++++++++++++++++++++++++++++++++++

Figure 6-18. Launch the XS program

20. When the program has finished running, press RETURN. The SHELXTL summary window redisplays (recall Figure 6-3).

21. Click on the XSHELL button to start the XSHELL program. The Fourier peaks will be displayed (Figure 6-19).

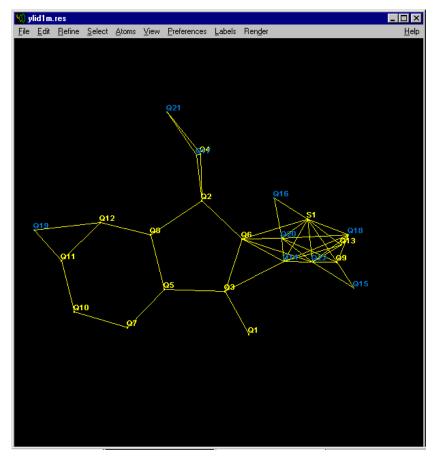


Figure 6-19. Fourier peaks

22. You may rotate the structure by dragging the mouse with the left button depressed. Select all false peaks (peaks Q14 to Q22 in the example shown) by moving the mouse to each peak and clicking the right mouse button.

Note: For more detailed instructions on use of the XSHELL program, see the XSHELL User's Manual.

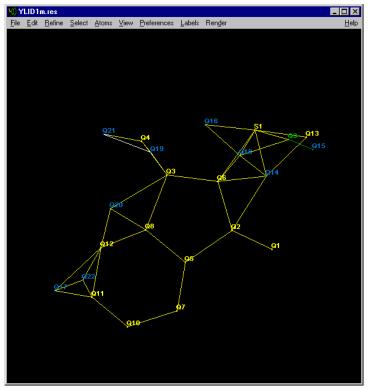


Figure 6-20. Molecule with peaks Q14 to Q22 selected

6-16

Note: You can also use the S key to select a peak directly.

The peak should turn blue when selected. The molecule may be rotated during this process (Figure 6-20).

- 23. When all false peaks have been selected, click the Kill Selected button in the Select menu. The peaks will disappear (Figure 6-21).
- 24. Select the 11 peaks that correspond to carbon atoms (9 atoms in fused 5- & 6-membered ring, 2 atoms attached to S atom) in the order in which they are to be numbered.

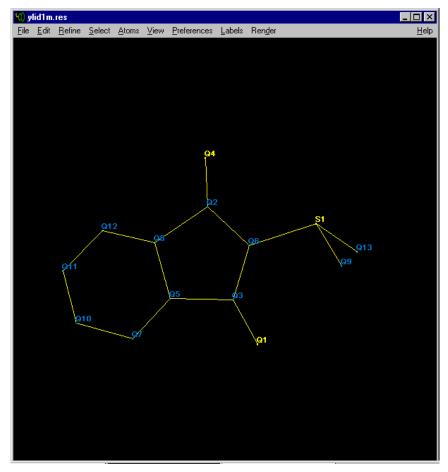


Figure 6-21. Peaks disappear

25. Click Labels > Group Label (Figure 6-22).

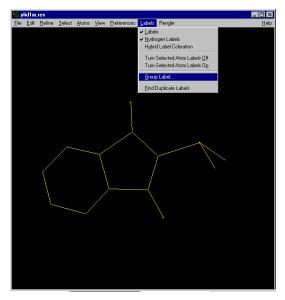


Figure 6-22. Select Group Labels

A panel will appear to label these peaks (Figure 6-23).

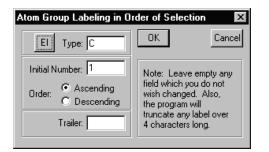


Figure 6-23. Input panel

26. Click OK to label these peaks as C with a starting number of 1 (Figure 6-24).

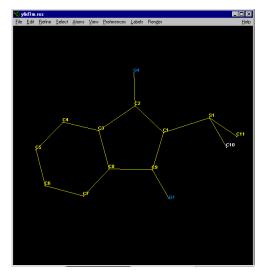


Figure 6-24. Label the peaks

27. Select the two remaining atoms. Then click Labels > Group Label again, and click the El button. A periodic table will appear (Figure 6-25).

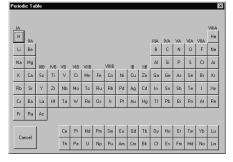


Figure 6-25. Periodic table

28. Click the O button for Oxygen. A panel appears for you to label these peaks (Figure 6-26).

Y)) ylid1m.res	_ 🗆 ×
<u>Eile Edit Refine Select Atoms View Preferences Labels Render</u>	<u>H</u> elp
Atom Group Labeling in Order of Selection X EE Type: O OK Cancel Initial Number: T OK Cancel Initial	×c11

Figure 6-26. Input panel appears

- 29. Click OK to label the peaks as O with a starting number of 1.
- 30. Click the Refine button at the top of the XSHELL Menu. A panel will appear (Figure 6-27).

Refinement Control
Cycles Extinction Plan 20
Aniso Invert Fourier Difference Map Comit
Bond 🗸 Acta List None 💌
Weights:
Previous
Suggested
Program Priority C Idle © Normal C High C Realtime
OK Cancel

Figure 6-27. Refinement control

31. Click OK to launch the XL (least-squares refinement) program (Figure 6-28).

Refine	×
GooF = S = 2.372; Restrained GooF = 2.372 for 0 restraints	
Mean shift/esd = 2.439 Maximum = -11.246 for U11 S1 at 16:47:09	
Max. shift = 0.040 A for C4 Max. dU =-0.006 for C1	
wR2 = 0.2582 before cycle 3 for 2435 data and 57 / 57 parameters	
GooF = S = 2.065; Restrained GooF = 2.065 for 0 restraints	
Mean shift/esd = 0.330 Maximum = 2.089 for x C4 at 16:47:09	
Max. shift = 0.012 A for C4 Max. dU = 0.000 for C5	
wR2 = 0.2574 before cycle 4 for 2435 data and 57 / 57 parameters	
GooF = S = 2.052; Restrained GooF = 2.052 for 0 restraints	
Mean shift/esd = 0.128 Maximum = -0.501 for x O2 at 16:47:09	
Max. shift = 0.003 A for C6 Max. dU = 0.000 for C6	
wR2 = 0.2575 before cycle 5 for 2435 data and 2 / 57 parameters	
GooF = S = 2.052; Restrained GooF = 2.052 for 0 restraints	
R1 = 0.0883 for 2297 Fo > 4sig(Fo) and 0.0917 for all 2435 data	
wR2 = 0.2575, GooF = S = 2.052, Restrained GooF = 2.052 for all data	
** Absolute structure probably wrong - invert and repeat refinement **	
Absolute structure probably wrong - invert and repeat reminiment	
R1 = 0.0933 for 1436 unique reflections after merging for Fourier	
Highest peak 1.03 at 0.2526 0.3357 0.2429 [0.50 A from S1]	
Deepest hole -0.64 at 0.1642 0.2805 0.2391 [0.53 A from S1]	

+ YLID1m finished at 16:47:10 Total elapsed time: 1.9 secs +	

	-
	_
OK <u>P</u> rint	

Figure 6-28. Least-squares refinement

In the output above, note that the R1 value is 9.0%. This is typical for a preliminary isotropic refinement with no H atoms included. Also note that, for our sample, the program informs us that we must invert the molecule to obtain the correct absolute structure.

6-20

32. Click OK to return to XSHELL.

The Q peaks on the diagram represent difference peaks.

33. You may delete them with the Edit > Kill all <u>Q</u> peaks command (Figure 6-29).

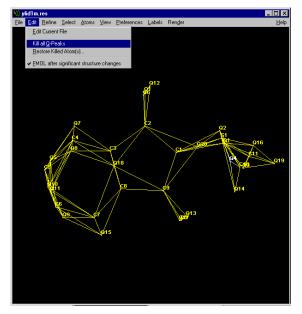


Figure 6-29. Kill all Q-Peaks

34. Click the Refine button at the top of the XSHELL Menu. A panel will appear (Figure 6-30).

Refinement Control
Cycles 4 Extinction 001 Plan 5
Aniso Image Invest Image Difference Map Image Omit InSignas 2theta
Bond 🔽 Acta List None 💌
Weights:
Previous
Suggested 0.151300
Program Priority C Idle © Normal C High C Realtime
OK Cancel

Figure 6-30. Refinement control

35. Change the input values to include Extinction and Anisotropic refinement. Also, reduce the number of difference peaks to 5. If the refinement indicates an inverted structure (as in our example), also check the Invert box.

36. Click OK to launch the XL (least-squares refinement) program (Figure 6-31).

Refine
Mean shift/esd = 1.557 Maximum = 11.250 for U13 S1 at 08:09:29
Max. shift = 0.020 A for C5 Max. dU =-0.002 for C2
wR2 = 0.1614 before cycle 2 for 2332 data and 128 / 128 parameters
GooF = S = 0.697; Restrained GooF = 0.697 for 0 restraints
Mean shift/esd = 1.439 Maximum = 11.279 for U13 S1 at 08:09:30
Max. shift = 0.008 A for C5 Max. dU = 0.003 for C5
wR2 = 0.1393 before cycle 3 for 2332 data and 128 / 128 parameters
GooF = S = 0.596; Restrained GooF = 0.596 for 0 restraints
Mean shift/esd = 0.331 Maximum = -2.923 for EXTI at 08:09:30
Max. shift = 0.002 A for C10 Max. dU = 0.001 for C5
wR2 = 0.1383 before cycle 4 for 2332 data and 128 / 128 parameters
GooF = S = 0.591; Restrained GooF = 0.591 for 0 restraints
Mean shift/esd = 0.081 Maximum = -2.917 for EXTI at 08:09:31
Max. shift = 0.001 A for C5 Max. dU = 0.000 for C5
wR2 = 0.1382 before cycle 5 for 2332 data and 2 / 128 parameters
GooF = S = 0.590; Restrained GooF = 0.590 for 0 restraints
R1 = 0.0505 for 2231 Fo > 4sig(Fo) and 0.0518 for all 2332 data
wR2 = 0.1382, GooF = S = 0.590, Restrained GooF = 0.590 for all data
R1 = 0.0535 for 1407 unique reflections after merging for Fourier
Highest peak 0.51 at 0.3138 0.5799 0.2321 [0.96 A from C10]
Deepest hole -0.24 at 0.1814 0.8834 0.1210 [1.30 A from C2]
++++++++++++++++++++++++++++++++++++++
OK <u>P</u> rint

Figure 6-31. Least-squares refinement

In the output above, note that the R1 value is 5.0%. This is typical for a preliminary anisotropic refinement with no H atoms included.

6-22

- 37. Click OK to return to XSHELL.
- Kill the Q-peaks as before. Then click Atoms > Hybridize All (Figure 6-32). The atoms will appear in different colors.

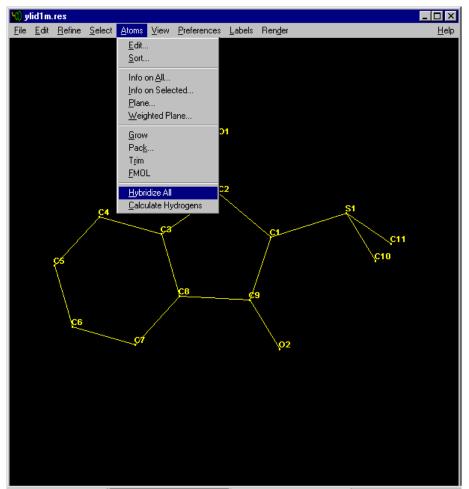


Figure 6-32. Hybridize all

39. Click Atoms > Calculate Hydrogens (Figure 6-33).

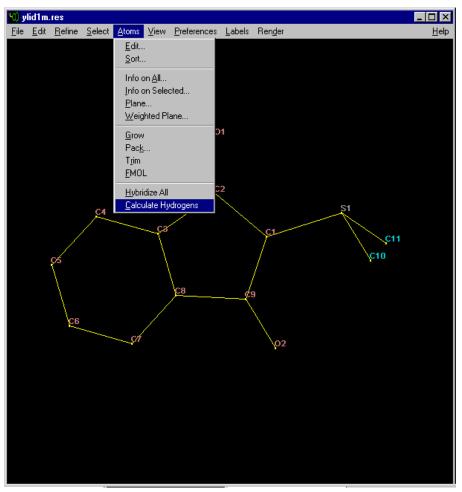


Figure 6-33. Calculate hydrogens

6-24

40. The 10 H atoms will be added (Figure 6-34).

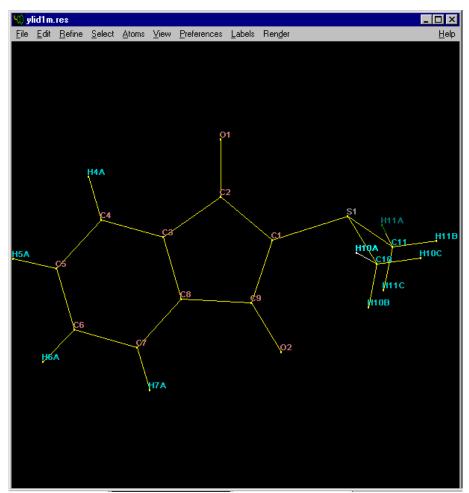


Figure 6-34. Ten H atoms added

41. Click the Refine button at the top of the XSHELL Menu. A panel will appear (Figure 6-35).

Refinement Control	×
Cycles Extinction 0.000000 Plan 5	
Aniso	
Bond 🗹 Acta List None 🔽	
Weights:	
Previous 0.151300	
Suggested 0.102700 🔽 0.000000 🔽	
Program Priority ◯ Idle ⊙ Normal ◯ High ◯ Realtime	
OK Cancel	

Figure 6-35. Refinement control

42. Click OK to launch the XL (least-squares refinement) program (Figure 6-36).

Refine	×
Mean shift/esd = 1.161 Maximum = 5.496 for y C4 at 17:01:06 Max. shift = 0.072 A for H10B Max. dU =-0.001 for C4	_
wR2 = 0.0886 before cycle 2 for 2435 data and 130 / 130 parameters	
GooF = S = 0.686; Restrained GooF = 0.686 for 0 restraints	
Mean shift/esd = 0.525 Maximum = -4.775 for EXTI at 17:01:07	
Max. shift = 0.008 A for H10B Max. dU =-0.001 for C10	
wR2 = 0.0873 before cycle 3 for 2435 data and 130 / 130 parameters	
GooF = S = 0.675; Restrained GooF = 0.675 for 0 restraints	
Mean shift/esd = 0.073 Maximum = -4.817 for EXTI at 17:01:07	
Max. shift = 0.001 A for H10B Max. dU = 0.000 for C10	
wR2 = 0.0873 before cycle 4 for 2435 data and 130 / 130 parameters	
GooF = S = 0.676; Restrained GooF = 0.676 for 0 restraints	
Mean shift/esd = 0.045 Maximum = -4.814 for EXTI at 17:01:08	
Max. shift = 0.001 A for H10B Max. dU = 0.000 for O1	
wR2 = 0.0873 before cycle 5 for 2435 data and 2 / 130 parameters	
GooF = S = 0.675; Restrained GooF = 0.675 for 0 restraints	
R1 = 0.0318 for 2297 Fo > 4sig(Fo) and 0.0334 for all 2435 data	
wR2 = 0.0873, GooF = S = 0.675, Restrained GooF = 0.675 for all data	
R1 = 0.0326 for 1436 unique reflections after merging for Fourier	
Highest peak 0.29 at 0.8214 0.4402 0.1532 [0.72 A from C1]	
Deepest hole -0.17 at 0.1898 0.4771 0.0215 [1.28 A from C6]	

+ YLID1m finished at 17:01:09 Total elapsed time: 3.6 secs +	
	_
OK Print	

Figure 6-36. Least-squares refinement

In the output above, note that the R1 value is 3.2%. This is typical for an anisotropic refinement with H atoms included.

- 43. Click OK to return to XSHELL and delete Q peaks.
- 44. Click the Refine button at the top of the XSHELL Menu. A panel will appear (Figure 6-37).

Refine	K
Mean shift/esd = 1.161 Maximum = 5.496 for y C4 at 17:01:06	J
Max. shift = 0.072 A for H10B Max. dU =-0.001 for C4	
wR2 = 0.0886 before cycle 2 for 2435 data and 130 / 130 parameters	
GooF = S = 0.686; Restrained GooF = 0.686 for 0 restraints	
Mean shift/esd = 0.525 Maximum = -4.775 for EXTI at 17:01:07	
Max. shift = 0.008 A for H10B Max. dU =-0.001 for C10	
wR2 = 0.0873 before cycle 3 for 2435 data and 130 / 130 parameters	
GooF = S = 0.675; Restrained GooF = 0.675 for 0 restraints	
Mean shift/esd = 0.073 Maximum = -4.817 for EXTI at 17:01:07	
Max. shift = 0.001 A for H10B Max. dU = 0.000 for C10	
wR2 = 0.0873 before cycle 4 for 2435 data and 130 / 130 parameters	Ľ
GooF = S = 0.676; Restrained GooF = 0.676 for 0 restraints	H
Mean shift/esd = 0.045 Maximum = -4.814 for EXTI at 17:01:08	H
Max. shift = 0.001 A for H10B Max. dU = 0.000 for O1	H
wR2 = 0.0873 before cycle 5 for 2435 data and 2 / 130 parameters	H
GooF = S = 0.675; Restrained GooF = 0.675 for 0 restraints	H
R1 = 0.0318 for 2297 Fo > 4sig(Fo) and 0.0334 for all 2435 data	H
wR2 = 0.0873, GooF = S = 0.675, Restrained GooF = 0.675 for all data	H
R1 = 0.0326 for 1436 unique reflections after merging for Fourier	H
Highest peak 0.29 at 0.8214 0.4402 0.1532 [0.72 A from C1]	H
Deepest hole -0.17 at 0.1898 0.4771 0.0215 [1.28 A from C6]	H
	H
+ YLID1m finished at 17:01:09 Total elapsed time: 3.6 secs +	H
+++++++++++++++++++++++++++++++++++++	-1
OK Print	

Figure 6-37. Refinement control

Note that the suggested weighting scheme has values of 0.0485 and 0.000. You are now ready for the final leastsquares refinement run.

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45. Check the Acta box to create .cif files for publication. Click OK to launch the XL (least-squares refinement) program (Figure 6-38).

Refine	×
Mean shift/esd = 0.566 Maximum = 4.142 for OSF at 17:09:07	
Max. shift = 0.007 A for H10C Max. dU = 0.001 for C10	
wR2 = 0.0778 before cycle 2 for 2435 data and 130 / 130 parameters	
GooF = S = 1.029; Restrained GooF = 1.029 for 0 restraints	
Mean shift/esd = 0.226 Maximum = -3.951 for EXTI at 17:09:08	
Max. shift = 0.002 A for H10C Max. dU = 0.000 for C10	
wR2 = 0.0776 before cycle 3 for 2435 data and 130 / 130 parameters	
GooF = S = 1.025; Restrained GooF = 1.025 for 0 restraints	
Mean shift/esd = 0.043 Maximum = -3.930 for EXTI at 17:09:08	
Max. shift = 0.000 A for H10A Max. dU = 0.000 for C6	
wR2 = 0.0776 before cycle 4 for 2435 data and 130 / 130 parameters	
GooF = S = 1.026; Restrained GooF = 1.026 for 0 restraints	
Mean shift/esd = 0.033 Maximum = -3.929 for EXTI at 17:09:09	
Max. shift = 0.000 A for H10C Max. dU = 0.000 for C7	
wR2 = 0.0776 before cycle 5 for 2435 data and 2 / 130 parameters	
GooF = S = 1.025; Restrained GooF = 1.025 for 0 restraints	
R1 = 0.0309 for 2297 Fo > 4sig(Fo) and 0.0326 for all 2435 data	
wR2 = 0.0776, GooF = S = 1.025, Restrained GooF = 1.025 for all data	
R1 = 0.0317 for 1436 unique reflections after merging for Fourier	
Highest peak 0.26 at 0.7701 0.3559 0.2036 [0.82 A from C1]	
Deepest hole -0.18 at 0.1900 0.4769 0.0242 [1.30 A from C6]	

+ ylid1m finished at 17:09:09 Total elapsed time: 3.3 secs +	
***************************************	_
	-
OK Print	

Figure 6-38. Least-squares refinement

In the output above, note that the R1 value is 3.1% and that the goodness-of-fit (GooF) value is now 1.026. We have carried out a complete refinement of suitable quality for publication. This is typical for a final anisotropic refinement with H atoms included.

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- 46. Click OK to return to XSHELL.
- 47. To display the thermal ellipsoids for the final structure, click on the background with the right mouse button and select Thermal Ellipsoids (Figure 6-39).

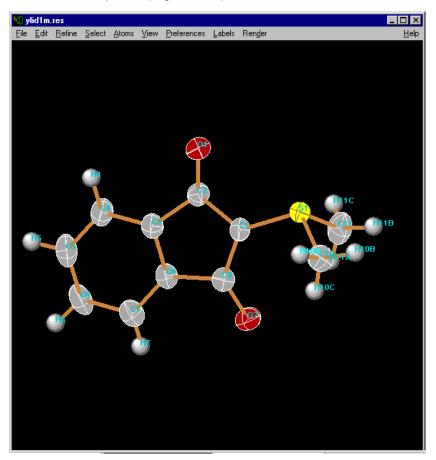


Figure 6-39. Thermal Ellipsoids

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