BRUKER AXS SMART APEXII SINGLE CRYSTAL DIFFRACTOMETER

STANDARD OPERATION PROCEDURE

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1. SAFETY

Your safety is the first concern. Each individual who wishes to be authorized to use X-ray equipments at the Facility must first receive training concerning the X-ray radiation hazards associated with the use of the equipment, the function and importance of the equipment's safety devices, the proper operation procedures, and procedures to follow in the even of a suspected radiation exposure.

1.1 Links to On-line Safety Training:

http://www.safety.ncsu.edu/xray_training/
http://www.nyu.edu/fas/dept/chemistry/wardgroup/DoubleEdgedSword.mov

1.2 Radiation Dosimeter

Wear your radiation badge and finger dosimeter all the time when you work in the X-ray lab.

To obtain the whole body badge and finger dosimeter, please contact Charles Strom (charlie.strom@nyu.edu, 212-998-88480, 1001l Silver).

1.3 Geiger counter

A Geiger counter is available in the lab for detecting any X-ray leakage.

1.4 Standard Operation Protocol

Always follow this standard operation protocol to operate the machine. If you have any questions, do not hesitate to contact Dr. Chunhua (Tony) Hu (212-9988769, chunhua.hu@nyu.edu).

2. DATA COLLECTION

2.1. Log in the computer with the username of *APEXII User* and the password of *apexii*.

2.2. To start BIS, choose **Start > Programs > Bruker AXS > Administration > BIS** or double-click BIS icon on the Desktop. After initialization, BIS asks for the detector distance as shown in the Fig. 1. Click OK.

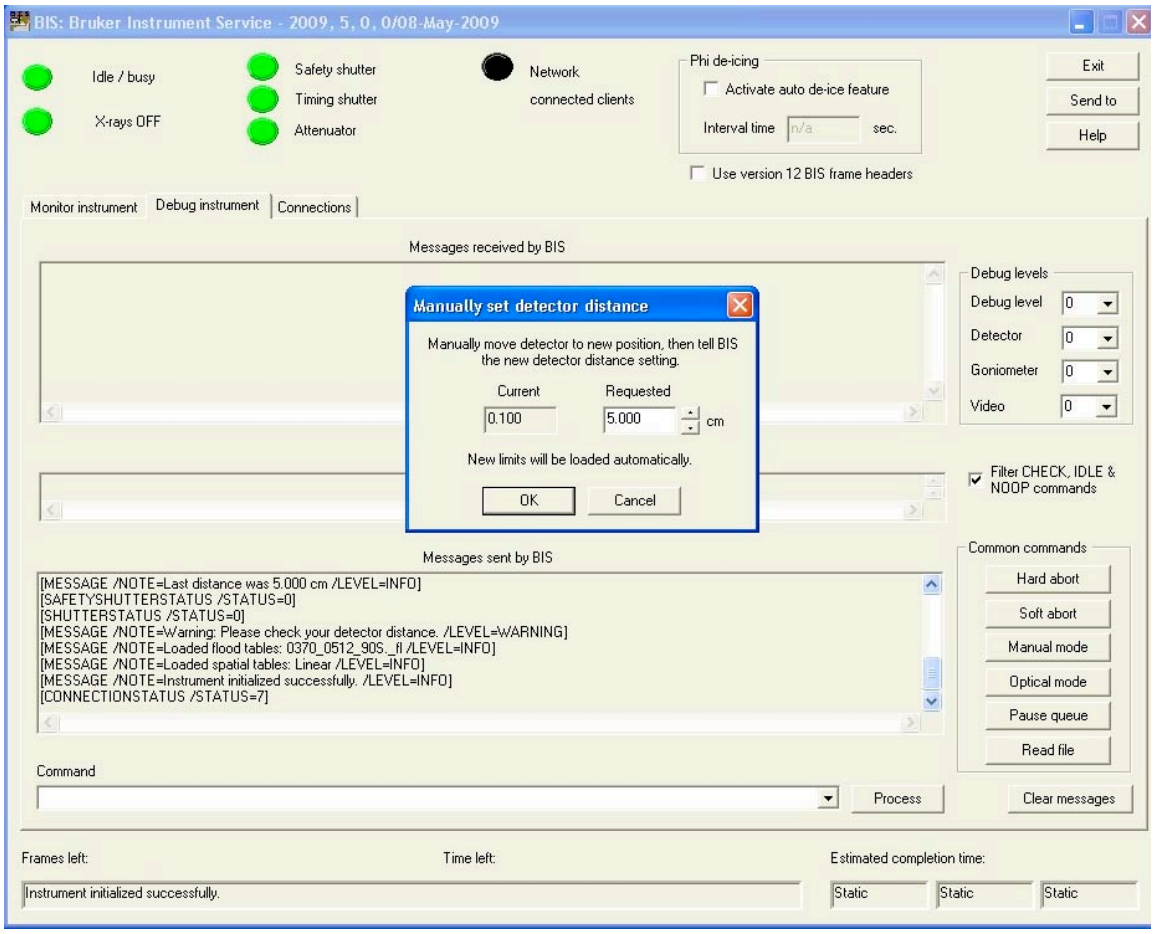

Fig. 1 BIS login

2.3. To start APEX2, choose **Start > Programs > Bruker AXS > APEX2** or double-click APEX2 icon on the Desktop. Choose **Instrument > Connect**, use the Host Name of apexii to connect the diffractometer.

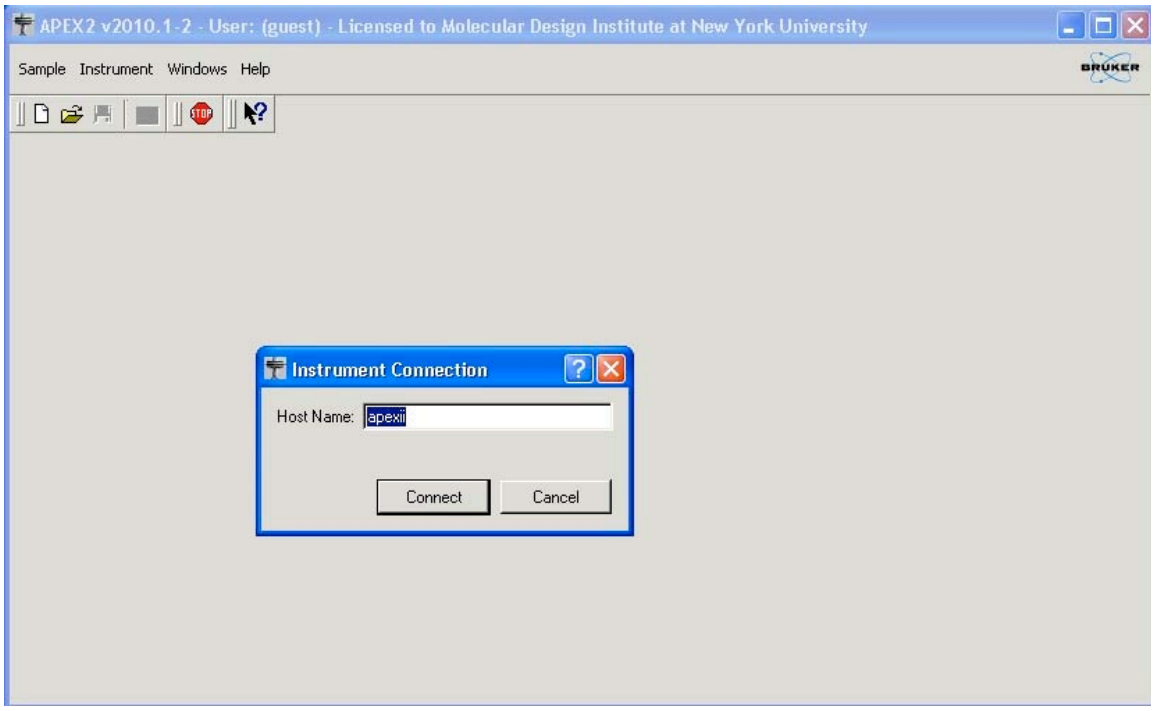

Fig. 2 Connect the host computer

2.4. To create a new sample, choose **Sample > New…** from the Menu Bar or click the New icon . Give a sample name under the pop-up window (Fig. 3). The sample name should be year (10 stands for 2010), initials of the group advisor (three letters, for example, mdw is Mike D. Ward), and the sequence number followed by an initial of the operator (for example, h stands for Chunhua Hu).

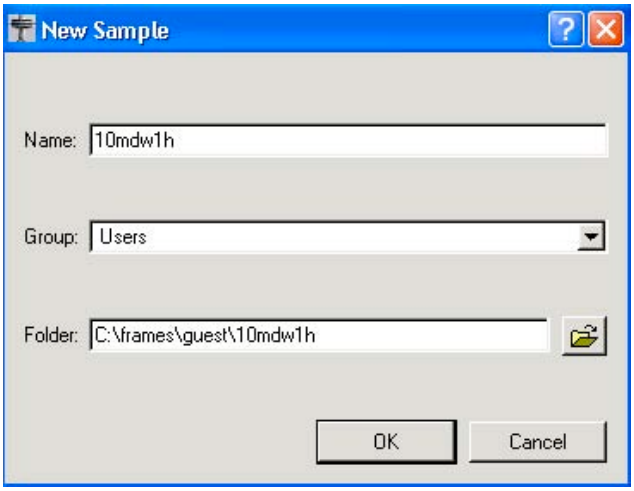

Fig. 3 New Sample

2.5. If VIDEO is not started, start it by double-clicking the VIDEO icon on the desktop.

2.6. In APEX2 program choose **Center Crystal**.

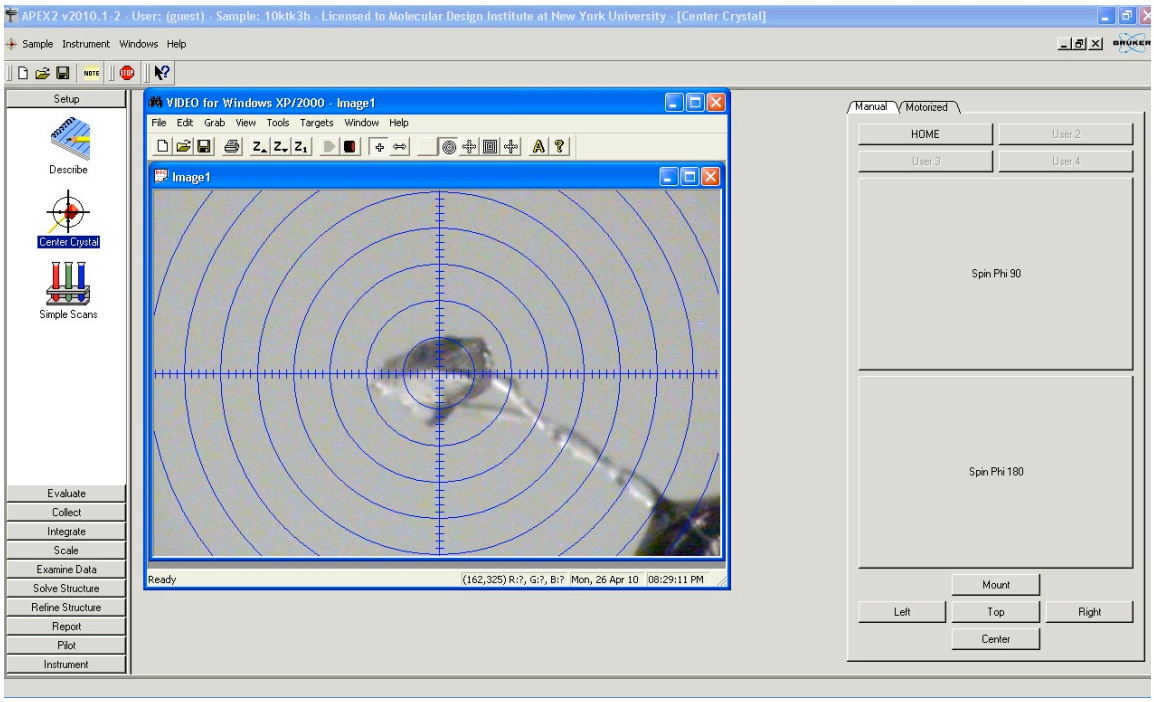

Fig. 4 Center Crystal

2.7. Click **HOME** to home the goniometer. Adjust the screw in the goniometer head and move the crystal to the center of the video crosshair. Click **Spin Phi 180** to check the position. Adjust it if necessary. Click **Spin Phi 90** to rotate the Phi in 90 degrees and move the crystal to the center. Click **Spin Phi 180** to check the position again. Adjust it if necessary. Click **Spin Phi 90** and **Spin Phi 180** a few times more to make sure that the crystal is centered.

2.8. Click  icon in the VIDEO program. Measure the crystal size by mouse clicking. Write down the crystal information in the **Describe** module (Fig. 5).

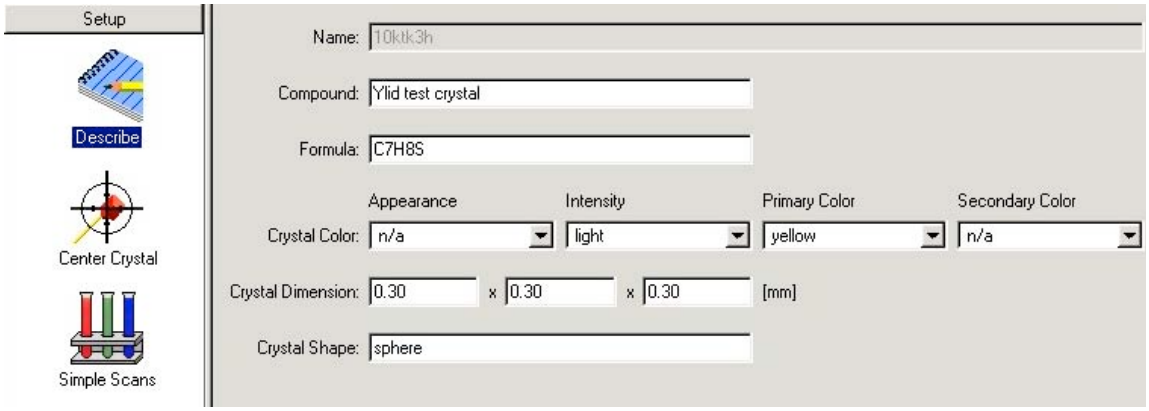

Fig. 5 Sample Description

2.9. Select **Simple Scans** module. Click **Still**, and **Drive + Scan**. After 10 seconds, an image collected is displayed on the screen. If you are not satisfied with the image (too weak intensity, obviously twinned crystal, or split crystal like

in Fig. 6), find another crystal under microscope and try again. If the crystal diffracts well and gives nice spot distribution, go ahead to collect matrix runs and determine the unit cell.

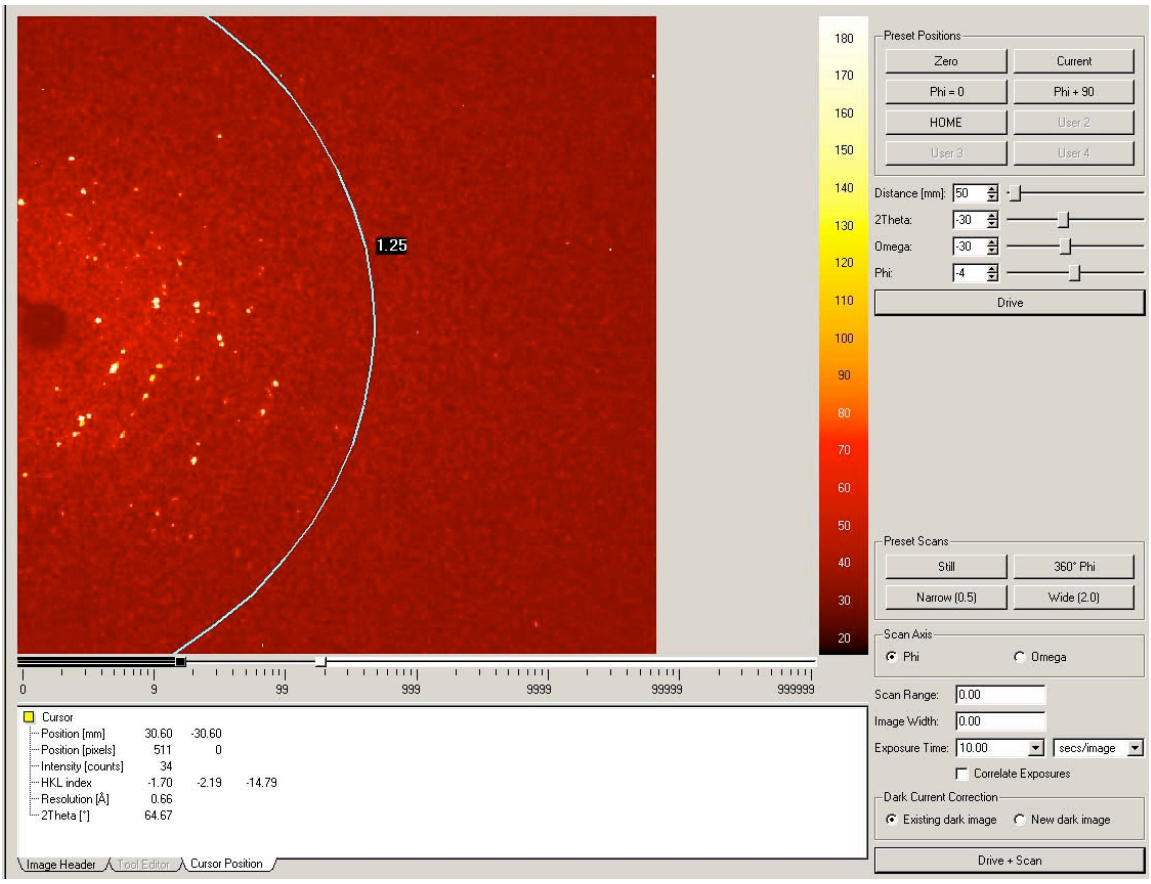

Fig. 6 Simple Scans

2.10. Select **Evaluate** module. Click **Determine Unit Cell**. Click **Run** to start the automatic unit cell determination. The unit cell parameters are listed in the table afterwards (Fig. 7).

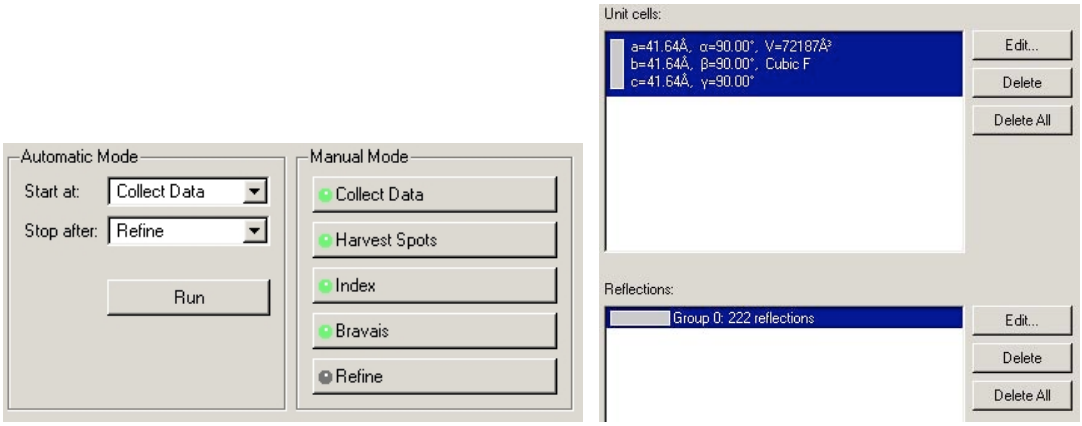

Fig. 7 Determine Unit Cell

2.11. Select **Collect** module. Click **Experiment**. Click **Load Table…** and load the file MIT_5cm_5runs.exp under c:\frames\. Change **Default time** (10 seconds) to *shorter* for the strongly diffracting crystal and *longer* for the weak ones. Click **Validate**. If passed, click **Execute** to collect data.

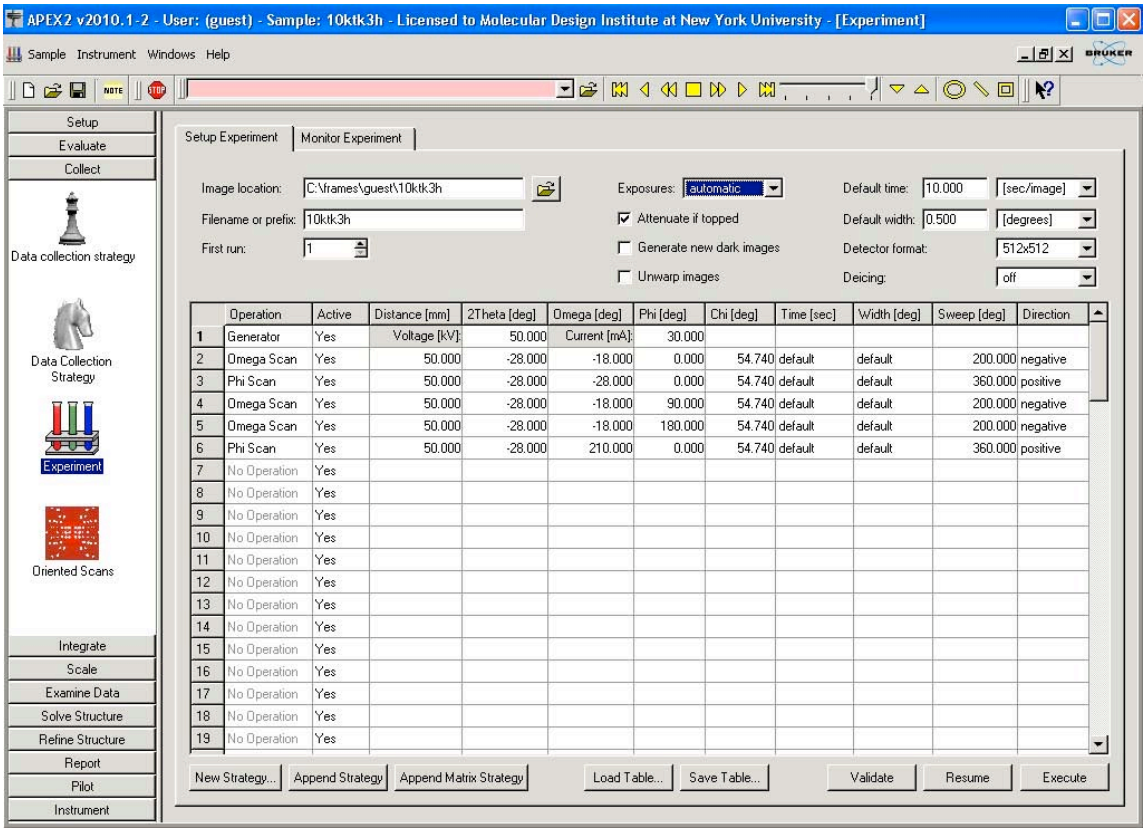

Fig. 8 Experiment

3. DATA PROCESSING

3.1. After data collection, select **Integrate/Integrate Images**.

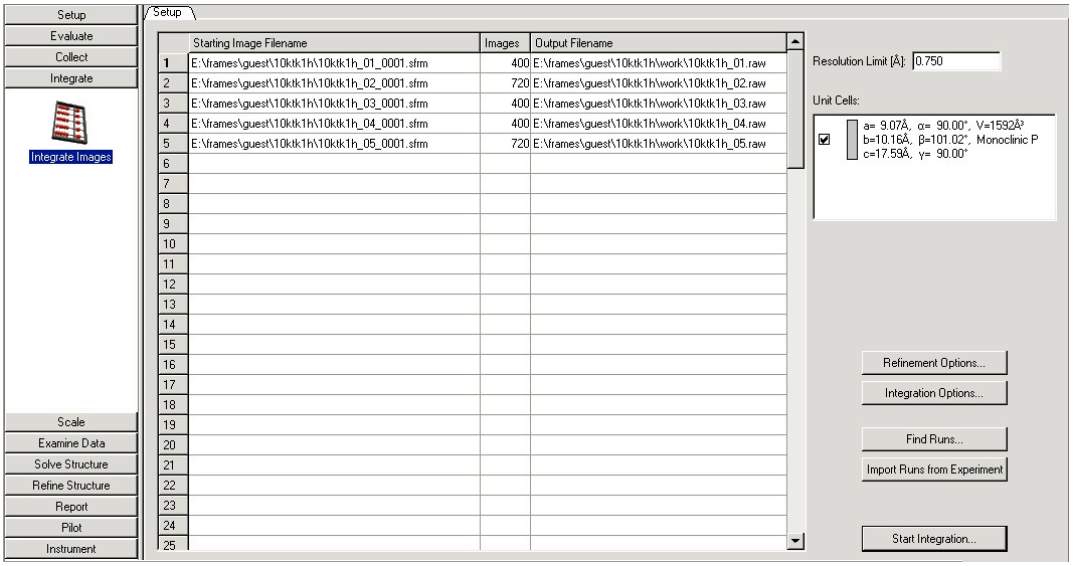

Fig. 9 Integrate Images

Click **Refinement Options** button. In the pop-up window, enable the options shown in the Fig. 10. Click **OK**.

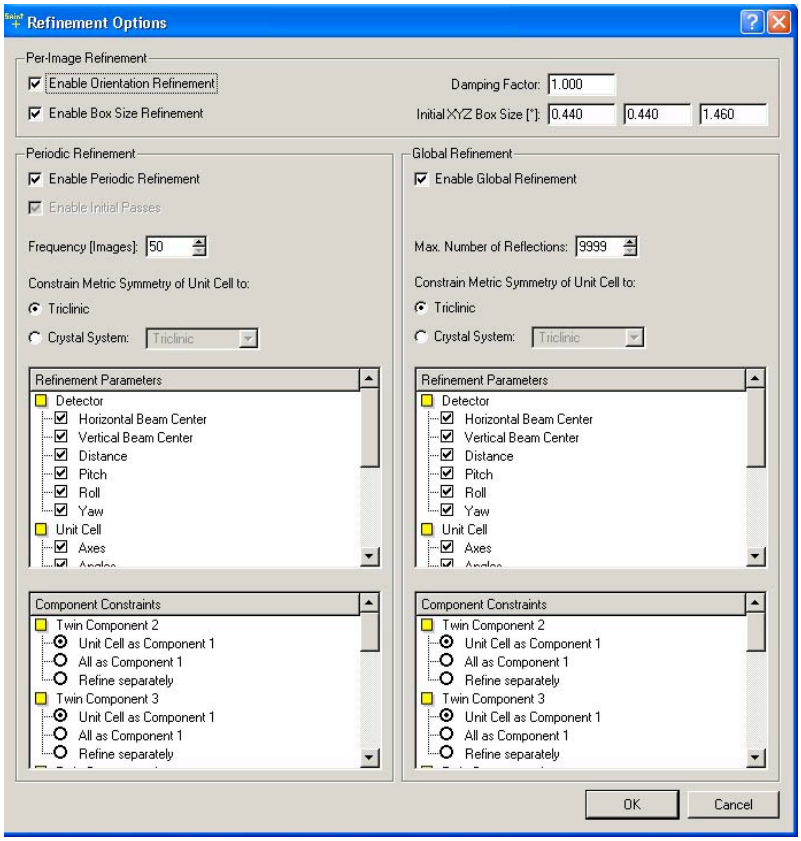

Fig. 10 Refinement Options

Click **Integrate Options** button. In the pop-up window, click **More Options** to extend the window. Enable the options shown in the Fig. 11 and use 0.500 for the **Generate Mask** option. Click **OK**.

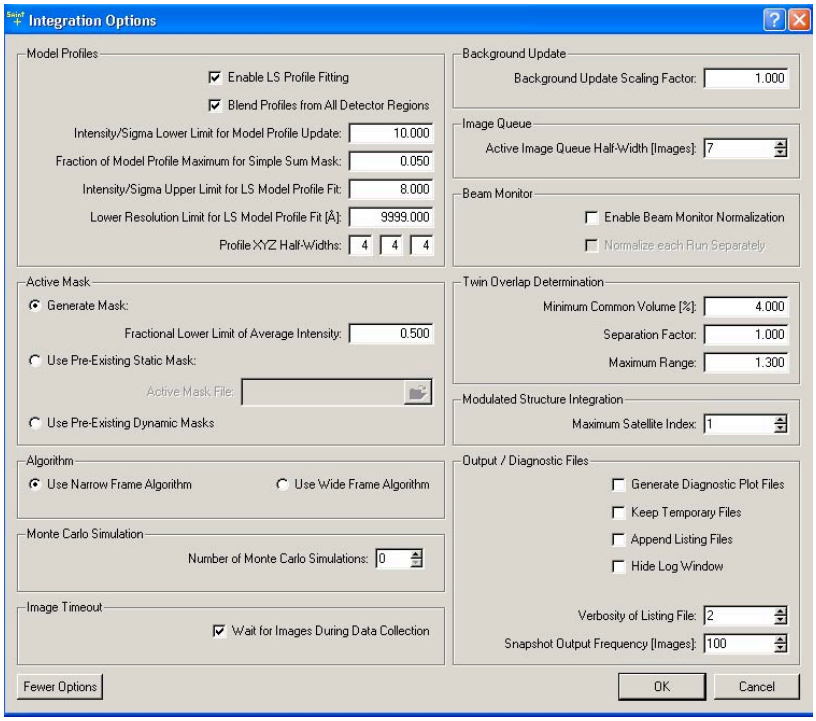

Fig. 11 Integrate Options

Click **Start Integration**. During the integration, the XYZ box size is re-calculated and updated. Write down these numbers, i.e. 0.73 for X, 0.62 for Y, and 1.38 for Z in this example (Fig. 12). When the integration finishes, select **Sample > import > P4P/SPIN file**, load the *m.p4p file and import all the information from this file (Fig. 13). Change the **Initial XYZ Box Size** with the updated ones under **Reflection Options**, re-run the integration. Doing so usually gives better results.

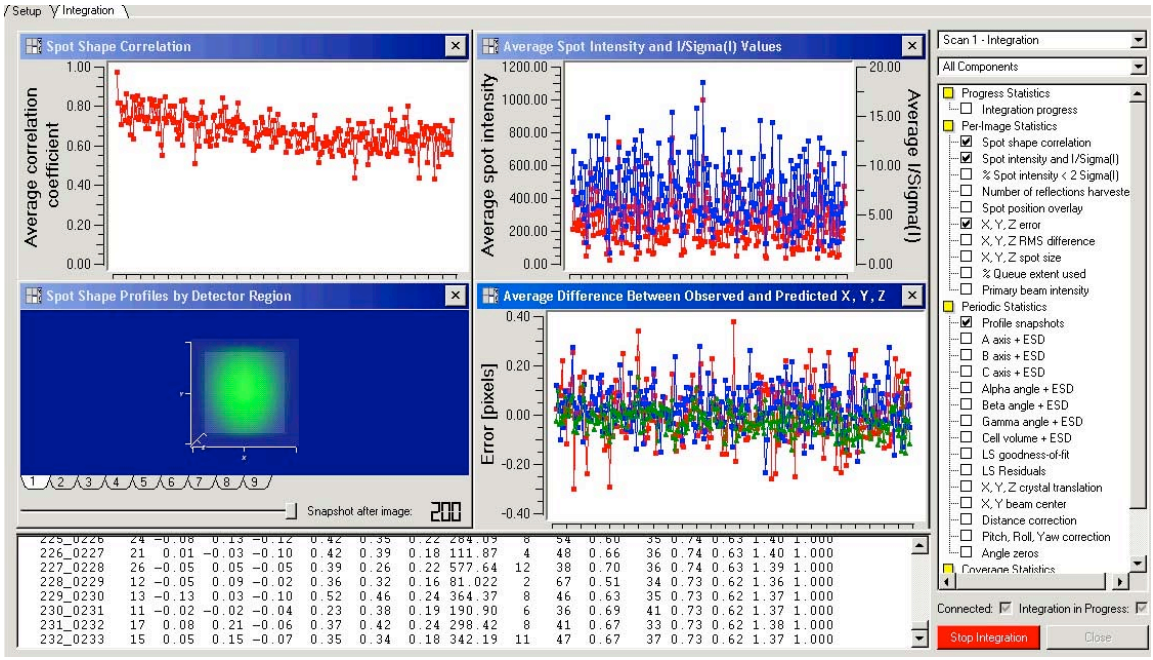

Fig. 12 Integration in Progress

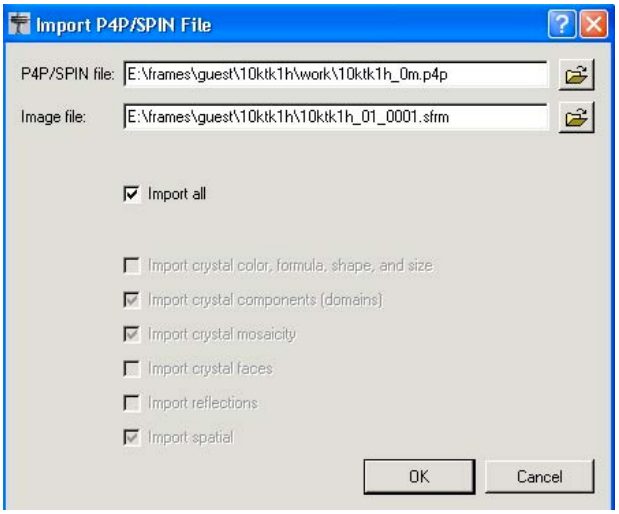

Fig. 13 Import P4P file

3.2. Select **Scale** module and click **scale** icon. In most cases the default settings are good enough (Fig. 14). If the space group of the structure is non-centrosymmetric, you need to select the corresponding non-centrosymmetric point group. Click **Next** to open the **Parameter Refinement** window (Fig. 15). Select the proper **Absorption Type** for your crystal. Click **Refine**. A graph (Fig. 16) appeared shows the intensity statistics. Click **Next** to continue. In the **Error Model** window (Fig. 17) click **Determine Error Model**. If you are happy with the results, click **Finish**. In the next window (Fig. 18) click on the tabs at the bottom of the screen to view diagnostic data. If the results are good, click **Exit AXScale** to exit the **Scale**.

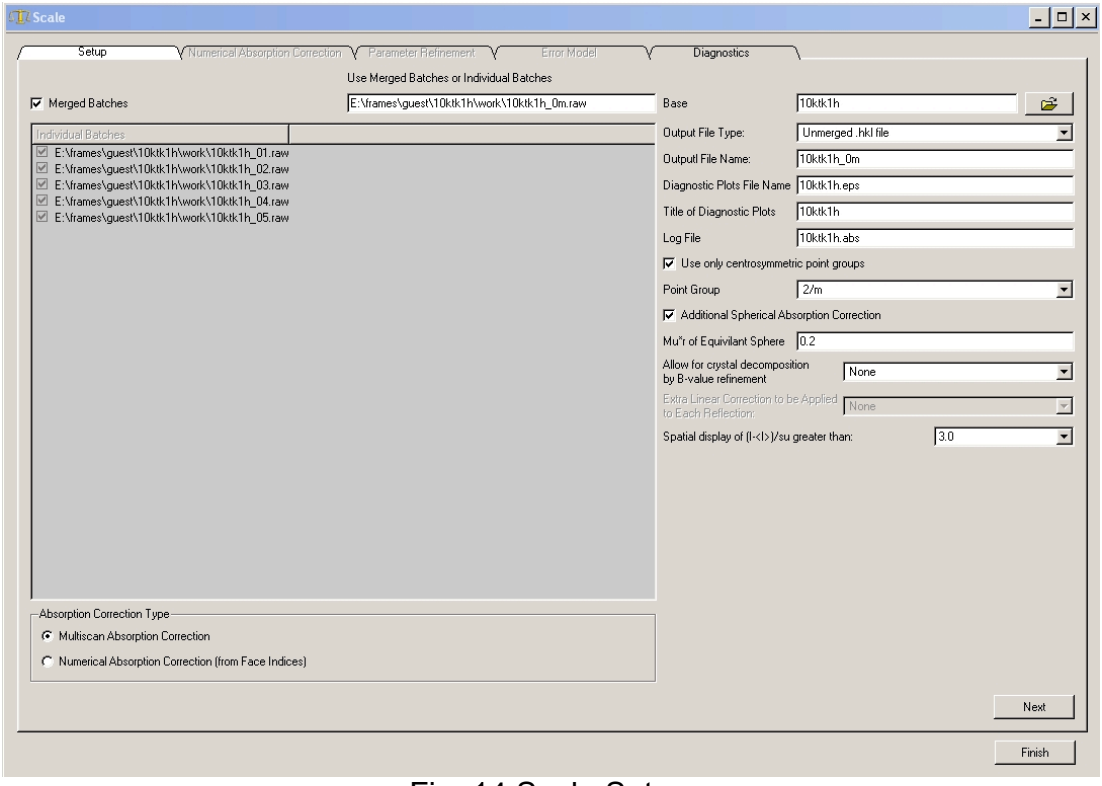

Fig. 14 Scale Setup

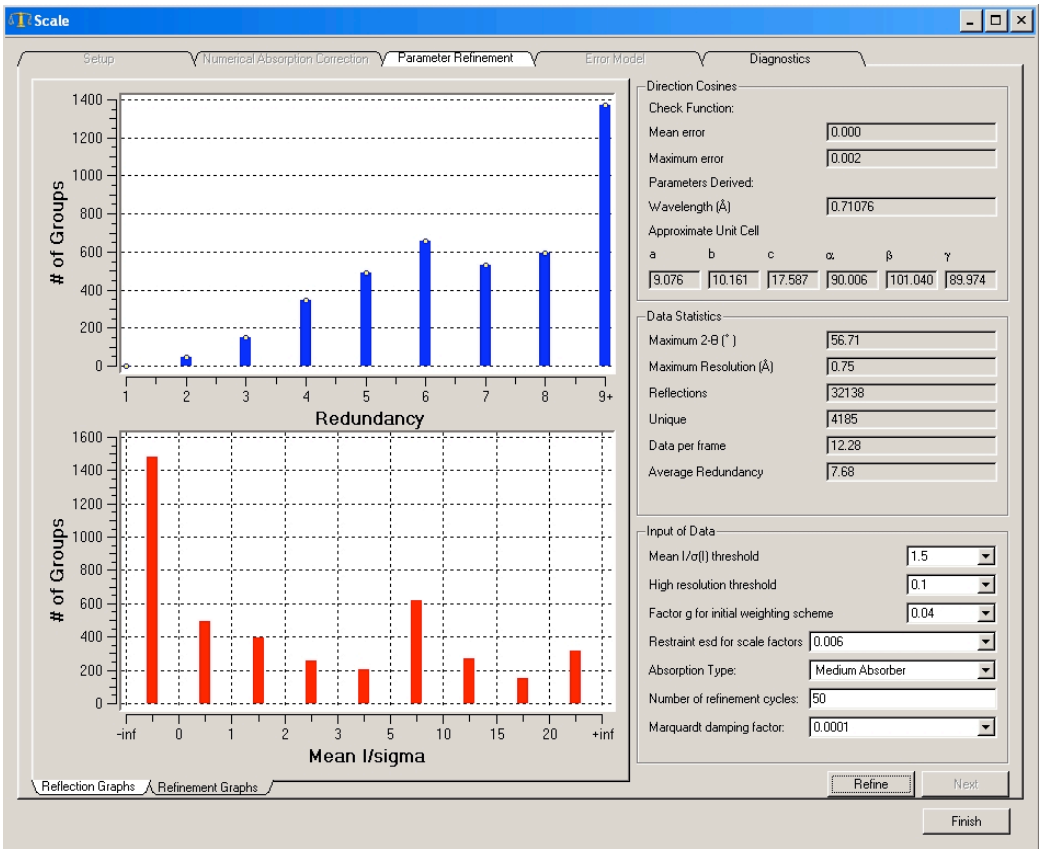

Fig. 15 Scale Parameter Refinement

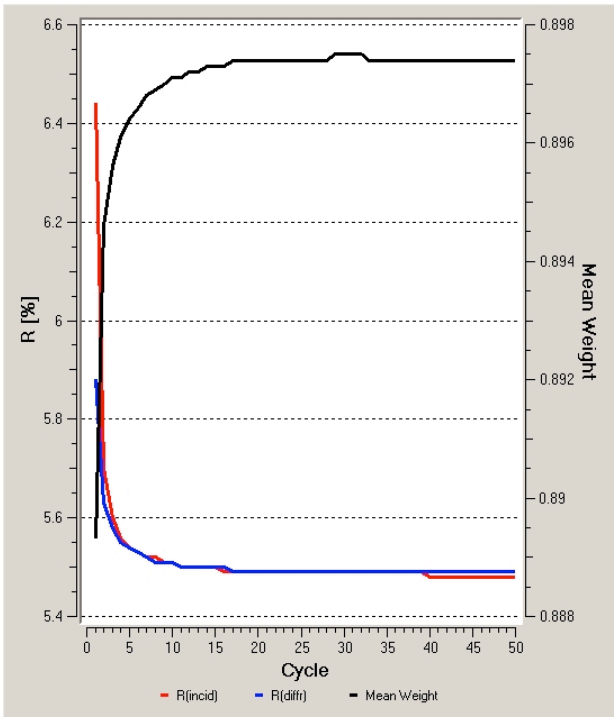

Fig. 16 Scale Graph

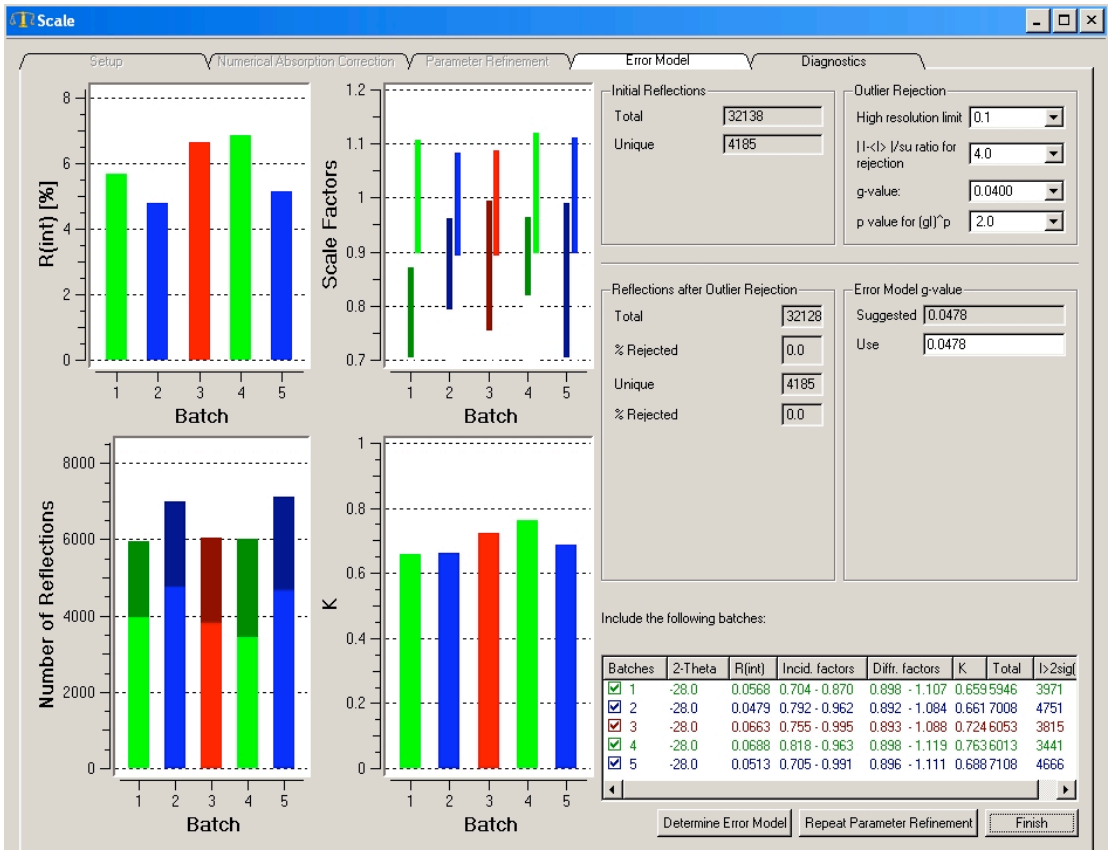

Fig. 17 Scale Error Model

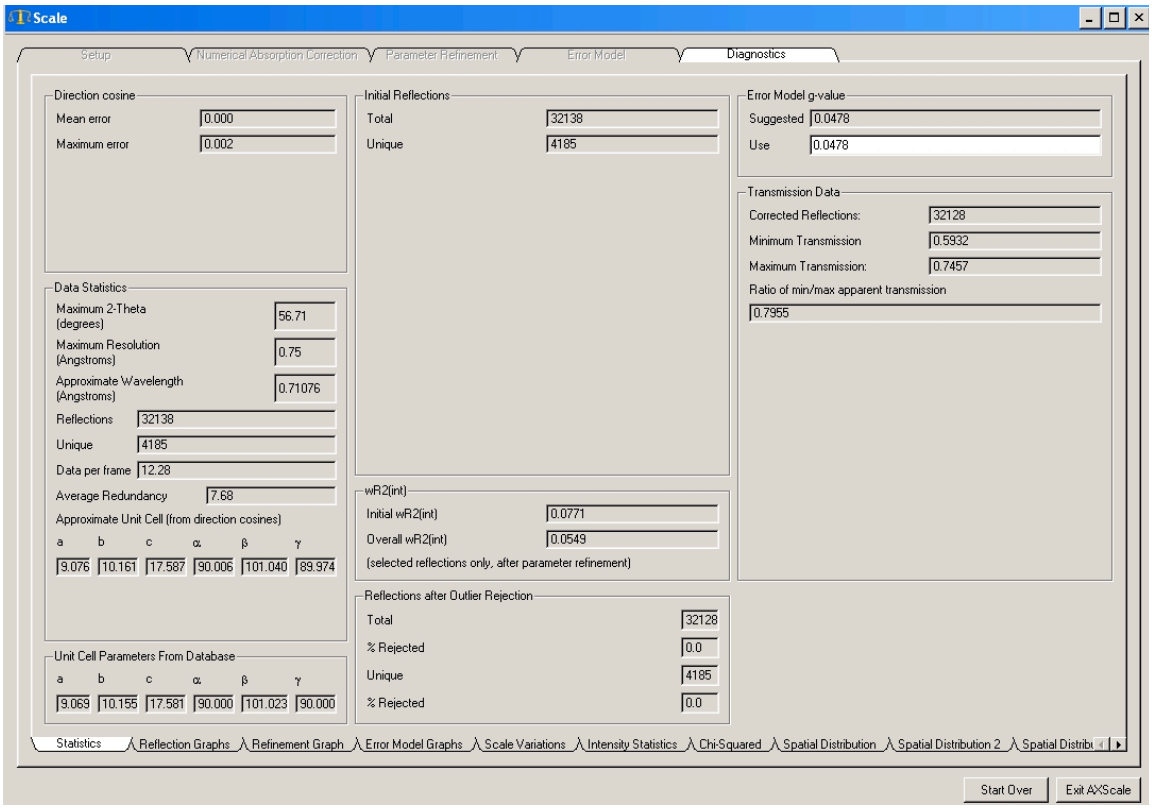

Fig. 18 Scale Diagnostics

3.3 Use the obtained *m.p4p and *m.hkl files as input files for space group determination (XPREP), structure solution (SHELTL xs), and structure refinement (SHELTL xl).

Please refer to the **APEX2 User Manual** for more details.

4. EXPERIMENTAL DESCRIPTION

A needle crystal with the size of 0.05 × 0.05 × 0.40 mm^3 was selected for geometry and intensity data collection with a Bruker SMART APEXII CCD area detector on a D8 goniometer at 100 K. The temperature during the data collection was controlled with an Oxford Cryosystems Series 700 plus instrument. Preliminary lattice parameters and orientation matrices were obtained from three sets of frames. Data were collected using graphite-monochromated and 0.5 mm-MonoCap-collimated Mo-K_α radiation (λ = 0.71073 Å) with the ω scan method [1]. Data were processed with the INTEGRATE program of the APEX2 software [1] for reduction and cell refinement. Multi-scan absorption corrections were applied by using the SCALE program for area detector. The crystal faces were determined by APEX2/Crystal Faces [1]. The structure was solved by the direct method and refined on F^2 (SHELXTL) [2]. Non-hydrogen atoms were refined with anisotropic displacement parameters, and hydrogen atoms on carbons were placed in idealized positions (C-H = 0.93 or 0.96 Å) and included as riding with U_{iso}(H) = 1.2 or 1.5 U_{eq}(non-H). Crystal data, data collection parameters and refinement results are listed in Table 1. Crystallographic data (excluding structure factors) for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publications no. ## (**compound 1**). Copies of available material can be obtained, free of charge, on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK, (fax: +44-(0)1223-336033 or e-mail: deposit@ccdc.cam.ac.uk).

[1] APEX2 (version 2009.11-0). *Program for Bruker CCD X-ray Diffractometer Control*, Bruker AXS Inc., Madison, WI, 2009.

[2] G. M. Sheldrick, SHELXTL, version 6.14. Program for solution and refinement of crystal structures, Universität Göttingen, Germany, 2000.

The underlined contents need be revised according to the experimental records and the journal requirements. The crystal size and color and the data collection temperature can be found in the CIF:

```
_exptl_crystal_description        block
_exptl_crystal_colour             yellow
_exptl_crystal_size_max           0.40
_exptl_crystal_size_mid           0.05
_exptl_crystal_size_min           0.05
_diffrn_ambient_temperature       100(2)
```

5. ACKNOWLEDGEMENT

The crystallographer should be considered for co-authorship when the structural information is an important part of the paper and structural information has been derived mainly from the diffraction data. The crystallographer will help completing the X-ray part of the experiments and reviewing the manuscript. If structure determination was used only to confirm information obtained by other means (NMR, MS, etc.) and no structural details will be given in the paper, only acknowledgment is more appropriate, i.e. we acknowledge the Department of Chemistry X-ray Diffraction Facility and Dr. Chunhua Hu for his help with structure determination.