

SMART X2S Remote Access: Initial Setup

The SMART X2S at Otterbein can be accessed remotely using a software package called CyberLab. The CyberLab software is a “Java Web Start” program, meaning that it can be started just like a locally installed program, but is actually running via the web. Detailed instructions for downloading and installing the CyberLab software are given below.

Note: This software only runs under **Windows** (XP or Win7). *It is not compatible with OS X.*

Downloading the CyberLab software (once per computer)

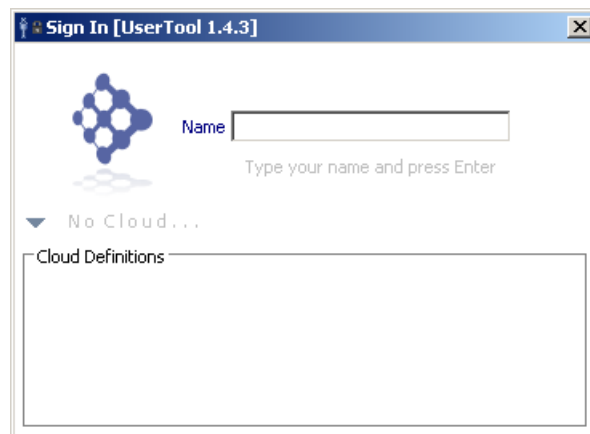
Point your browser to: <http://www.zethussoftware.com/tools-1.4.x/>

When asked to log in, Username is **otterbein**, password is (*contact Dean Johnston*).

Then select **CyberLab UserTool**. This will download a “Java Web Start” (.jnlp) file. The program will be downloaded and run from the Internet. Whether it runs automatically will depend upon your browser. You may have to go to your downloaded items folder and double-click on it.

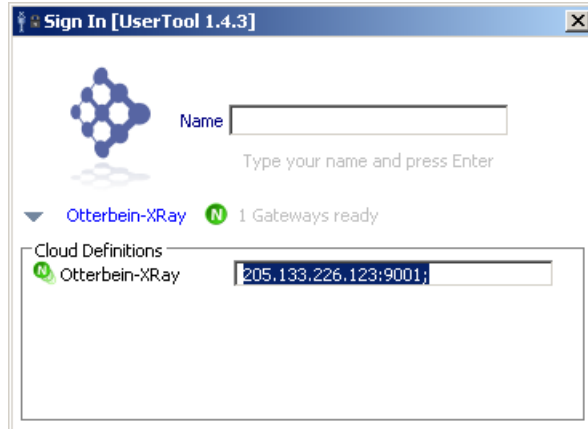
Defining the Otterbein SMART X2S Node (once per computer):

After starting the CyberLab software for the first time, you will need to tell the software how to find the Otterbein X2S Node.



- Click the small grey triangle to show the Cloud Definitions box
- Right-click in the Cloud Definitions box and select **Add Cloud**
- Enter the following information:
 - **Otterbein-XRay** in left box,
 - and **205.133.226.123:9001**; in the right box (see figure below)

- The completed definition should look like the figure below:



Once the initial set up is complete, refer to the **SMART X2S Remote Access: Logging In** instruction sheet.

SMART X2S Remote Access: Logging In

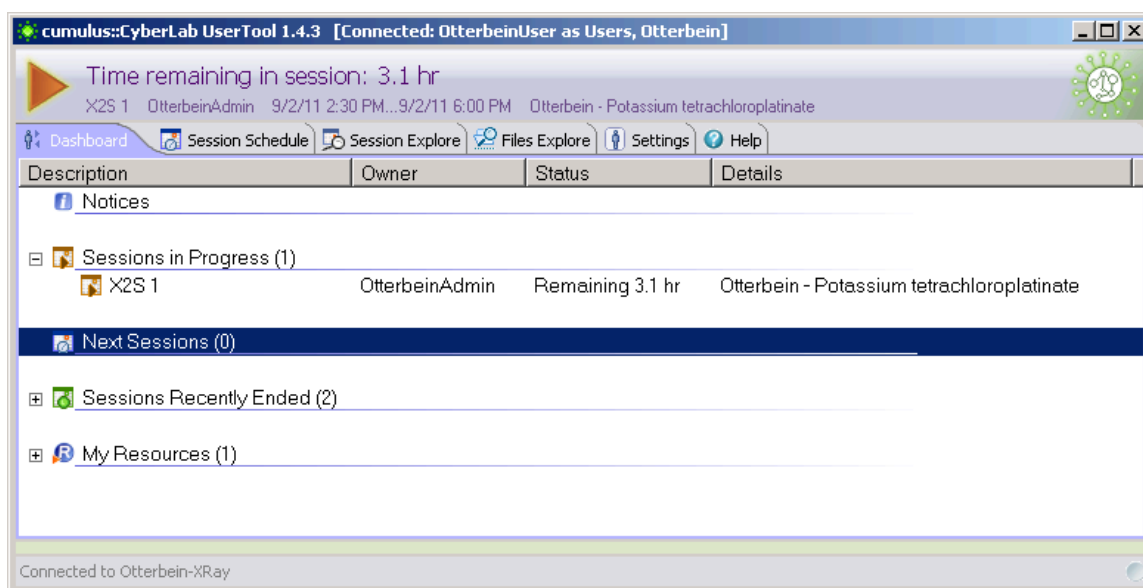
Logging into the SMART X2S *via* CyberLab will allow you to remotely access data files and reports, view scheduled sessions, and view/control the SMART X2S instrument in real time. Note that you will only have permission to control the instrument during scheduled instrument sessions (viewable under the Session Schedule tab). You can access files at any time.

Each institution will be assigned an individual user identity (otterbeinUser, wittenbergUser, etc.) and password. These will be given out individually.

Name: **otterbeinUser** (for example)

Key: ●●●●●● (contact Dean Johnston for your Name and Key)

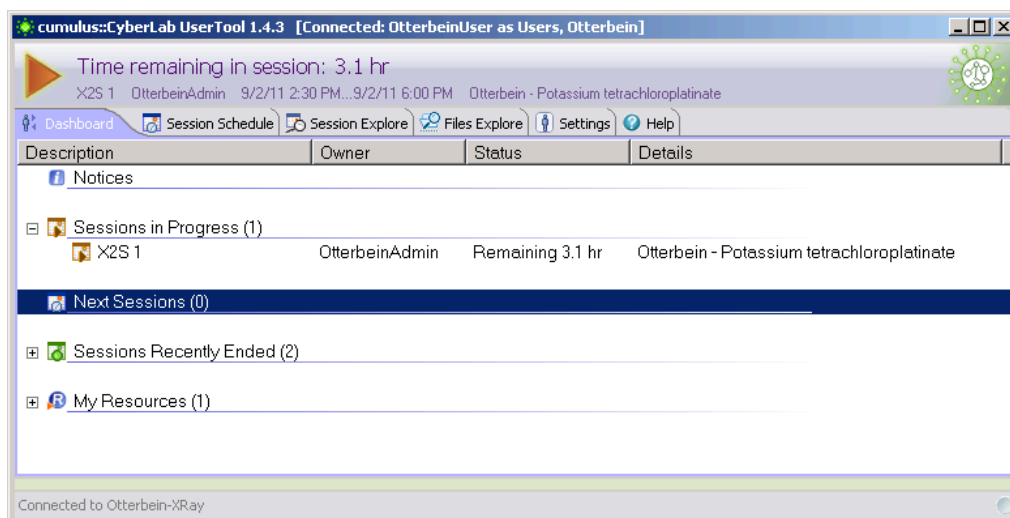
Logging in gives a window similar to that shown below:



In this case, a session has been created for OtterbeinUser. Clicking on the large orange arrow will open a view/control session window. Instructions for viewing a session can be found in the **SMART X2S: Remote Viewing / Remote Control** instruction sheet.

SMART X2S: Remote Viewing / Remote Control

When you log in to the CyberLab remote access system, you will only see those sessions for which you have been given permission to join. If you have scheduled a session and do not see the large orange arrow, please contact Dean Johnston. Here is the window for OtterbeinUser with an active session:



Clicking on the large orange arrow brings up a new VNC session with information about the current session. Click the small green and white X (⊗) in the top right corner to hide the information screen. Shown below is a screenshot from an active data collection session:



You can use your mouse to control the instrument as the data collection is running. Simply close the window to stop the remote viewing session. See Chapter 4 of the **Bruker SMART X2S User Manual** for more information about automated data collection and structure solution.

SMART X2S Remote Access: Accessing Files

Once the data collection and structure solution process is complete, the data files are burned to a CD (optional, but recommended) and archived to the data storage node. The data generated by the experiment and processing are archived into three zip files:

- **frames.zip** – the raw image files or “image frames”
- **work.zip** – the data processing files generated during indexing, integration, absorption correction, structure solution and structure refinement
- **report.zip** – the final report, generated as an HTML page with embedded *Jmol* figure

In most cases, when a satisfactory solution is found, you need only to download the final report, i.e., the **report.zip** file. This zip file contains both the full report (**report.html**) and the structure information (**report.jmol**). The **report.jmol** file is a crystallographic information (CIF) file, even though it is given a “.jmol” extension.

Sample Naming Convention

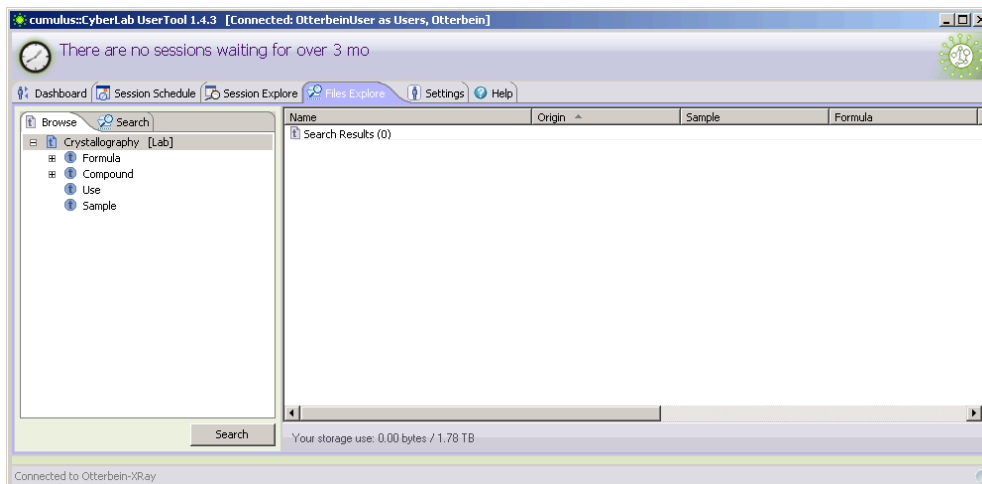
The following convention will typically be used for all samples:

- The first two letters represent the organization / institution
 - OT = Otterbein University
 - DU = Denison University
 - OW = Ohio Wesleyan University
 - WT = Wittenberg University, etc.
- The next six characters represent the date in YYMMDD format
- The next character is an A, B, C, etc. for each sample collected in the same day

Example – the first Otterbein sample collected on September 2, 2011 would be named **OT110902A**. Note that the three files archived always have the same *file* names. But they will use the above convention for the **sample** name. Additional information is associated with each file, including **formula**, **date**, and even the final **R-factor**.

Exploring Archived Files

Log into the SMART X2S Remote Access node as described in the **SMART X2S Remote Access: Logging In** instruction sheet. Once logged in, click on the **Files Explore** tab.

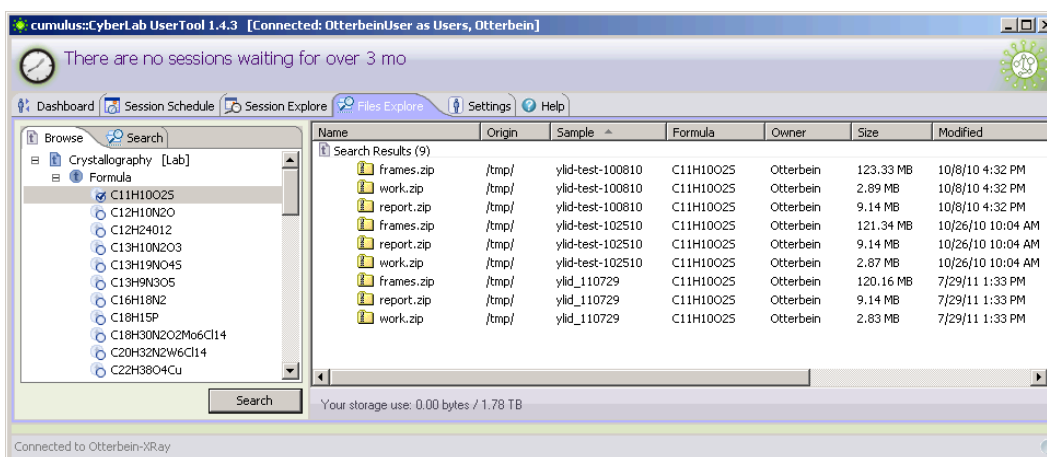


You can *browse* by **Formula** or **Sample (Name)**, or you can *search* by **Date Modified/Stored**. (Note: searching by Name, Origin or Owner is not very useful since those parameters are essentially the same for every experiment.)

Example #1 – Finding and Downloading Data for an “Ylid” Sample

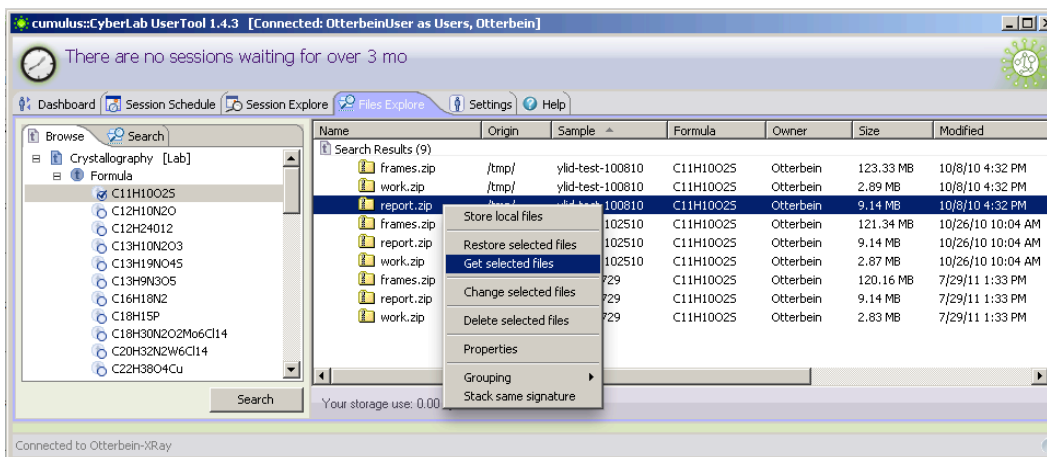
The reference sample for the instrument is 2-dimethylsulfuranylidene-1,3-indanedione ($C_{11}H_{10}O_2S$), and is commonly called the “ylid” sample. This sample gets run periodically to check instrument performance.

- In the **Files Explore** window, click on the plus sign to the left of the **Formula** item to expand the list of formulas entered for the various compounds.
- Double-click on the (first) entry with the formula $C_{11}H_{10}O_2S$ – the symbol to the left of the formula changes to a check mark.
- Click on the **Search** button at the bottom of the window – a list of all files with the selected formula is generated (shown below)



The window above shows a total of three different experiments, each generating three files (frames.zip, work.zip, report.zip). We will select one experiment and download just the report.

- Right-click on the first **report.zip** file and select **Get selected files** (see below). The file will be downloaded to the location you choose.



After expanding the **report.zip** file (creates a report directory), opening the **report.html** file in a browser shows the full structure report.

Structure determination of ylid-test-100810 with the Bruker SMART X2S benchtop crystallographic system

A colorless crystal of ylid-test-100810 with the dimensions of 0.50 mm x 0.50 mm x 0.50 mm mounted on a Mitegen Micromount was automatically centered on a Bruker SMART X2S benchtop crystallographic system. Intensity measurements were performed using monochromated (doubly curved silicon crystal) Mo-K α -radiation (0.71073 Å) from a sealed microfocus tube. Generator settings were 50 kV, 1 mA. Data collection temperature was 27°C.

Data were acquired using three sets of Omega scans at different Phi settings. The frame width was 0.5° with an exposure time of 5.0 s.

The detailed data collection strategy was as follows:
Detector distance: 40 mm
Detector swing angle (fixed 2 Theta): -20°.

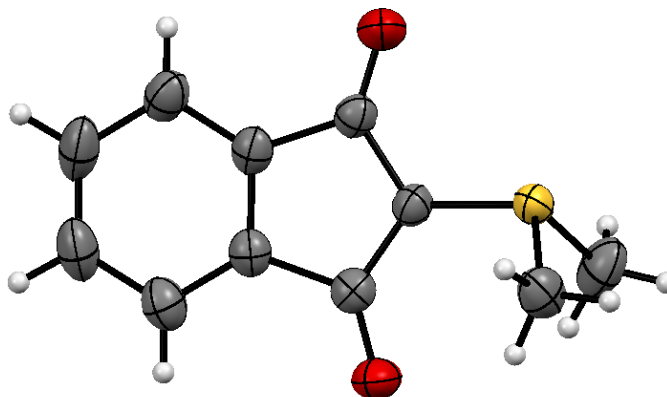
Run	Omega (start)	Omega (end)	Phi	Frames
1	-20.0	160.0	0.0	360
2	-20.0	100.0	120.0	240
3	-20.0	40.0	240.0	120

APEX2 software was used for preliminary determination of the unit cell. Determination of integrated intensities and unit cell refinement were performed using SAINT. The integration of the data yielded a total of 6285 reflections to a maximum θ angle of 25.08° (0.84 Å resolution). The constants for the orthorhombic unit cell are $a = 5.9501(6)$ Å, $b = 9.0360(8)$ Å, $c = 18.3522(18)$ Å, $V = 986.71(16)$ Å³. They are based upon the refinement of the XYZ-centroids of 3671 reflections above 20.0 I/ σ (I) with $2.51^\circ \leq \theta \leq 25.08^\circ$. Data were corrected for absorption effects with SADABS using the multiscan technique. The ratio of minimum to maximum apparent transmission is 93.5:100. The average residual for symmetry equivalent reflections is $R_{\text{int}} = 1.89\%$ and $R_{\sigma} = 1.78\%$. XPREP determined the space group to be **P 21 21 21**, with $Z = 4$ for the formula unit, **C₁₁H₁₀O₂S**.

The structure was solved with XS and subsequent structure refinements were performed with XL. The final anisotropic full-matrix least-squares refinement on F_o^2 with 129 variables converged at $R_1 = 2.59\%$ for the observed data and $wR_2 = 6.54\%$ for all data. The goodness-of-fit was

Jmol script terminated

Opening up the **report.jmol** file in a program like Mercury to generate ORTEP representations (below), packing diagrams, etc.



Example #2 – Finding Data By Date Stored

You can also search for files by the date they were originally stored on the storage node.

- In the **Files Explore** window, click on the **Search** tab.
- Click the checkbox next to the **Stored** item.
- Select or enter the date or date range of interest. (In this case we'll see what data was collected on September 2, 2011)
- Click on the **Search** button at the bottom of the window – a list of all files stored on September 2, 2011 is generated (shown below)

The screenshot shows the 'cumulus: CyberLab UserTool 1.4.3' interface. The 'Files Explore' tab is active, and the 'Search' sub-tab is selected. The search criteria are set to 'Stored' for the date '9/ 2/2011'. The search results table is as follows:

Name	Origin	Sample	Formula	Owner	Size	Modified	Stored
frames.zip	/tmp/	OT110902A	C8H8N2O4	Otterbein	124.66 MB	9/2/11 10:42 AM	9/2/11 10:42 AM
report.zip	/tmp/	OT110902A	C8H8N2O4	Otterbein	9.14 MB	9/2/11 10:42 AM	9/2/11 10:42 AM
work.zip	/tmp/	OT110902A	C8H8N2O4	Otterbein	3.81 MB	9/2/11 10:42 AM	9/2/11 10:42 AM
frames.zip	/tmp/	OT110902B	K2PtCl4	Otterbein	148.04 MB	9/2/11 5:21 PM	9/2/11 5:21 PM
work.zip	/tmp/	OT110902B	K2PtCl4	Otterbein	1.75 MB	9/2/11 5:21 PM	9/2/11 5:21 PM

The interface also shows a 'Search' button at the bottom left and a status bar at the bottom indicating 'Your storage use: 0.00 bytes / 1.78 TB'.

In this example, the second sample (OT110902B) did not generate a **report.zip** file because the experiment was aborted before the data collection and processing was completed. It is, however, still possible to download the “raw” data and fully process it using the APEX2 software suite (see the **Bruker APEX2 User’s Manual** for details).

SMART X2S: Summary of Generated Files

Archive File	Description	File Purpose	File Names / Extensions
frames.zip:	<i>"raw" image frames generated during data collection</i>	image frames	matrix_0x_yyyy.sfrm – <i>survey frames used to evaluate crystal and determine unit cell</i> scan_0x_yyyy.sfrm – <i>detailed frames used for full analysis</i>
work.zip:	<i>files generated by the indexing, integration, absorption correction, and structure solution and refinement are stored</i>	generated by SAINT (integration)	.raw, .mul, .p4p
		generated by scaling / SADABS (absorption correction)	.abs, .hkl – <i>lists each hkl index and reflection intensity</i>
		generated / used by XPREP, SHELXS, SHELXL	.ins – <i>instruction file</i> .res – <i>results file</i> .hkl – <i>merged hkl file</i> .lst – <i>listing (log) file</i>
		generated by SHELXL	.cif – <i>crystallographic information file, contains info about the final structure</i>
		image files (diagnostic)	.eps, .png
report.zip:	<i>files needed to display a web page with the full report and a Jmol view of the structure</i>	generated automatically	report.html – <i>the final report</i> report.jmol – <i>the CIF (crystallographic information file) file containing all the structure information</i>