

Activity Report for the X-ray Structural Facility for FY 2007

*Paul D. Boyle
X-ray Structural Facility
Department of Chemistry
North Carolina State University
Raleigh, NC 27695-8204*

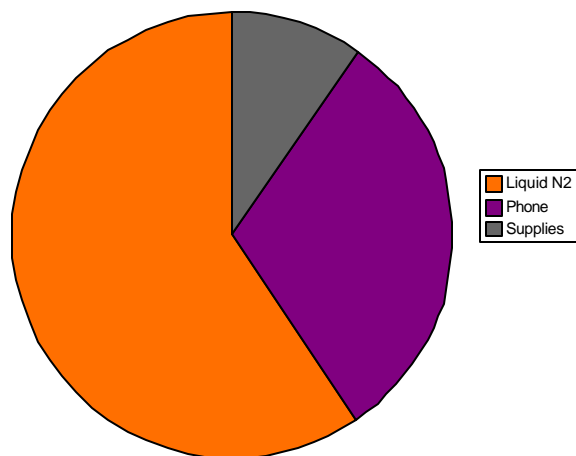
Introduction

This year the Faculty has seen a number of important changes and challenges. Most significantly, major software upgrades for the various diffractometer control computers were accomplished and a trial no usage fee scheme was adopted. The challenges included working through a number hardware and software problems with the CCD diffractometer.

Financial

In October 2006, the Department Chair gave the go ahead to stop charging for X-ray structure determinations for one year. This led to an increase in usage, including usage by groups which had not used the Facility very much in the past. The revenue generated thus far for FY2007 is \$2790. The expenses from 1 July 2006 until 30 April 2007 have been broken down into broad categories, as liquid nitrogen related expenses, \$1422.63; phone, \$742.51, and miscellaneous supplies, \$228.39, and is depicted graphically in Chart 1 below.

Facility Expenses FY2007

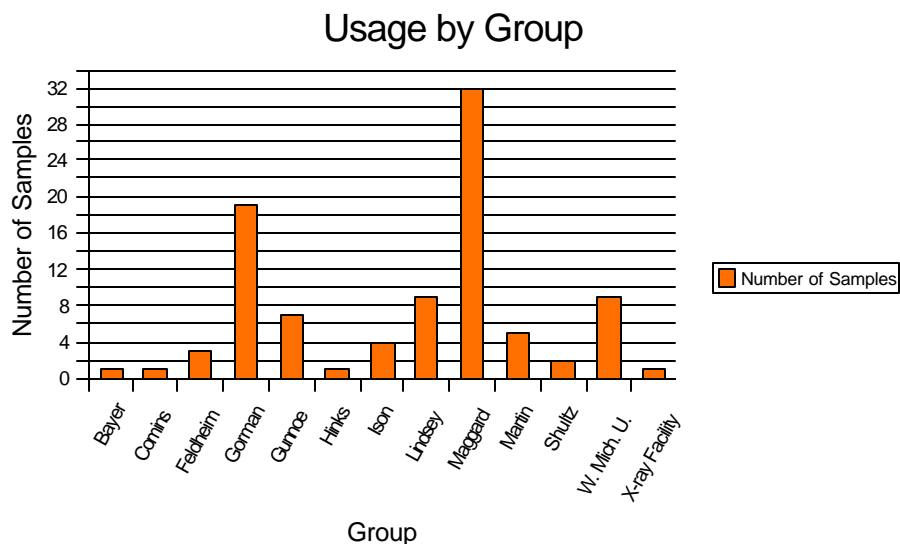


It should be noted that these figures do not include the approximately \$5500 expense for a service call for instrument repair which occurred in April 2007. The low temperature device is also due for a routine maintenance which will cost approximately \$1000.

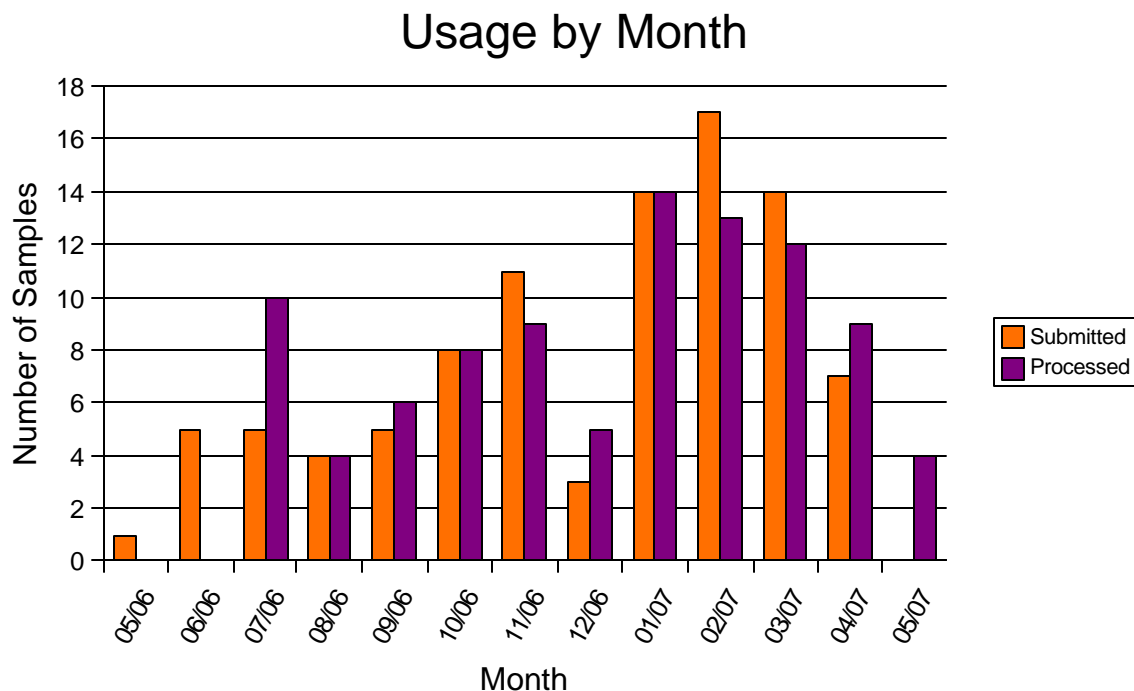
Usage

In the Faculty response to the X-ray Facility's self assessment in 2006, the stated desire was to have the Facility producing approximately 100 structures per year. For this to happen, the researchers in the Department should be submitting 200-300 samples per year. This is, on average, about 4 samples per

week. As mentioned above, the dropping of the fee structure has led to an increase in usage, including some groups which had not previously used Facility services in the past. The usage has been plotted by both research group and month. These charts are presented below.



As might be expected, the Maggard group has submitted the most samples, but surprisingly, the Gorman group has become a strong user of the Facility. I am pleased to see that organic chemists are making more use the Facility's services. Samples from the Lindsey group and the Department of Chemistry at Western Michigan University fills out the top three sample generators for the Facility.



The chart contains submitted entries for 05/06 and 06/06, these represent only the samples which were submitted during these months but were not processed (i.e. put on the instrument) until FY2007. As can be seen from the chart, there was a general increase in usage after the fee structure was dropped in

late October. In recent months usage has dropped a little, and this is primarily due to a number of instrumental problems (see the Instrument Maintenance and Downtime section below). I think these charts paint an optimistic picture of growing Facility usage. As the Ison group comes online, hopefully the sample submission levels will approach the 15-20 samples per month level. It is my hope that usage levels from other groups will increase as well. I recommend that the no fee for structure program be continued for another year.

Instrument Maintenance and Downtime

The CCD diffractometer's control computers underwent major upgrades in FY2007. Three different computers are used to control the instrument. The first is "the interface" which is used to drive the arcs and shutter mechanisms. The server computer provides a network communication interface and controls the CCD camera *per se*. The user control computer provides the user interface for data collection and data reduction of the frame data.

The user interface computer is a Linux workstation and was upgraded from a venerable Redhat 8.0 distribution to a modern Linux distribution (openSuSE 10.1), and the Apex2 diffractometer control software was upgraded to version 2.0 on this computer. A DVD burner was also added to this computer as well to allow for easier archiving of raw frame data.

The server computer also underwent very significant upgrades during FY2007. Because the Apex2 software was being upgraded on the user interface computer, Bruker suggested to upgrade the software on the Windows server computer as well. In addition, this computer always seemed unstable in that it would lock up at seemingly random times which caused data collections to halt.. Glenn Hennessee suggested it was due to a syndrome known as "Windows rot" where a Windows computer becomes increasingly unstable over time. Based on this suggestion, I made the decision to upgrade the operating system from Windows2000 to Windows XP as a prerequisite to the upgrade of the Bruker software. This, however, did not improve the stability of the computer. Glenn suggested that this was then indicative of a hardware problem. Eventually, I made an educated guess that the problem was due to a failing onboard network adapter. I installed a new ethernet card and the machine has been stable since then.

After Windows XP was successfully installed, the Bruker software was upgraded. However, due to a paucity of meaningful documentation, the upgrade proved problematic. Glenn and I tried upgrading the software, but were unsuccessful. A Bruker applications engineer then spent two weeks installing and getting the software working. Fortunately, this could be done via a program called Webex which allowed the applications engineer to work on our system remotely from Madison, Wisconsin.

There have been two problems with the instrument this fiscal year which have caused delays or disruptions in Facility operations. One problem was hardware related and the other was/is software related.

In late March and early April the instrument developed a shutter problem which inhibited data collection for a period of approximately three weeks. I reported the problem to Bruker on 30 March. I did some preliminary trouble shooting exercises given to me by Bruker service personnel over the phone and by email. Based on the results of that trouble shooting it was decided that a service call would be needed to resolve the problem. Unfortunately, it took some time for Bruker to schedule a visit by a service engineer. The service engineer was on site for two days and fixed the problem.

Since the software upgrades, I have experienced an intermittent software problem which causes an

interruption in the data collection. I am working with Bruker to resolve this problem. According to Bruker, I am not the first user to have seen this problem manifest. I suspect it has to do with bugs in the software regarding the synchronization of instrument data collection limits stored on different computers.

New Developments & Future Plans

In September I wrote a python program which takes CIF files and prepares nicely formatted tables of crystallographic data which can be easily imported into MS Word. Since September I have rewritten and expanded to software a series of class libraries which can be used to generate structure reports automatically from CIF files and user specified input. This set of libraries is being used to develop a GUI program which will generate reports. The same class libraries will be used to develop a web based report generation application.

In November 2006, presented a crystal growing workshop for graduate students and postdocs. I covered a number of topics designed to give the participants an understanding of what constitutes a suitable sample, general considerations in growing crystals, detailed explanations of various crystal growing techniques, followed up by a question and answer session. The workshop was geared toward researchers growing molecular crystals (i.e. organic, organometallic and coordination compounds) rather than crystallization of extended covalent structures. I made the decision to focus on this because it seemed that is where the largest gaps in knowledge needed to be filled. The workshop was relatively well attended with approximately 10 graduate students participating.

For FY2008, I would like to dispense with paper "Structure Determination Request" forms, and have users submit requests for structure determinations via the Facility's website. The web interface would enter the request into a queue. The queue could then be looked at by users to see when their sample will be run and the status of their request. One issue which would need to be resolved is uploading a chemical diagram to accompany the request. What would the file format need to be, and how to implement it. I welcome user comments on what they would like to see for such an online request system.

In FY2008, I plan to do another workshop. This could be a repeat the crystal growing workshop or on a different topic such as editing CIF's or how to interpret crystallographic output which comes as a report from structure determinations performed at NCSU. I am open to input from Faculty as to what they think would be most beneficial for their students.