10.3.2 User Newsletter

April 2, 2003

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This is the eighth issue of the user newsletter for 10.3.2. It's been quite a while since the seventh, and a lot has been happening and will happen. I'd like to take this time to welcome the new users who will be coming on next cycle.

Science

Thanks to all the users who have brought their work here and have been producing scientific results. Since much of it hasn't been published, I can't be too detailed about it, but I can mention some of the subject areas. As usual, there has been a lot of work on soils of various sorts, both contaminated and natural. Certain patterns of speciation are starting to build up. A growing segment of the work has concerned things that live in the soil, mainly hyperaccumulating plants and Mn-oxidizing bacteria. For example, a project of mine (in collaboration with F. Panfili and A. Manceau) involves looking at metal-bearing granules on root surfaces by combined EXAFS and XRD. There have also been several projects related to materials, such as the work of Grolimund, et. al. on model systems for waste disposal, and the CdSe nanoparticle work cited below.

Please, please keep me posted on any publications. I'm regularly asked about these, and they are the only evidence our management looks at to verify that the beamline remains productive and worth supporting. Unfortunately, DOE management subscribes to the theory that there are only three significant journals, *Science*, *Nature* and *Physical Review Letters*, so papers in these journals count more than papers in others, even in

flagship journals such as *Environmental Science and Technology.* I don't agree with this philosophy but it's what we have to work with.

A New Face

We have a new associate beamline scientist, replacing Bob Sublett, who quit to start his own aviation business. His name is Jamie Nasiatka (pronounced Na-SHOT-ka). He is a mechanical engineer by training. In the two weeks he's been here, he's really dug in and started to deal with the mechanics issues which have been piling up. For instance, he's working on the design of a new Peltier-cooled cold stage as well as sample holders for diffraction which are a little more elegant than the kludge we have now (not hard). He won't be full-time on 10.3.2, but instead will help out in various places where engineering is needed. It's expected that he will spend most of his time on upgrades to 7.3.3, but not until the immediate tasks are taken care of at 10.3.2.

Mirror

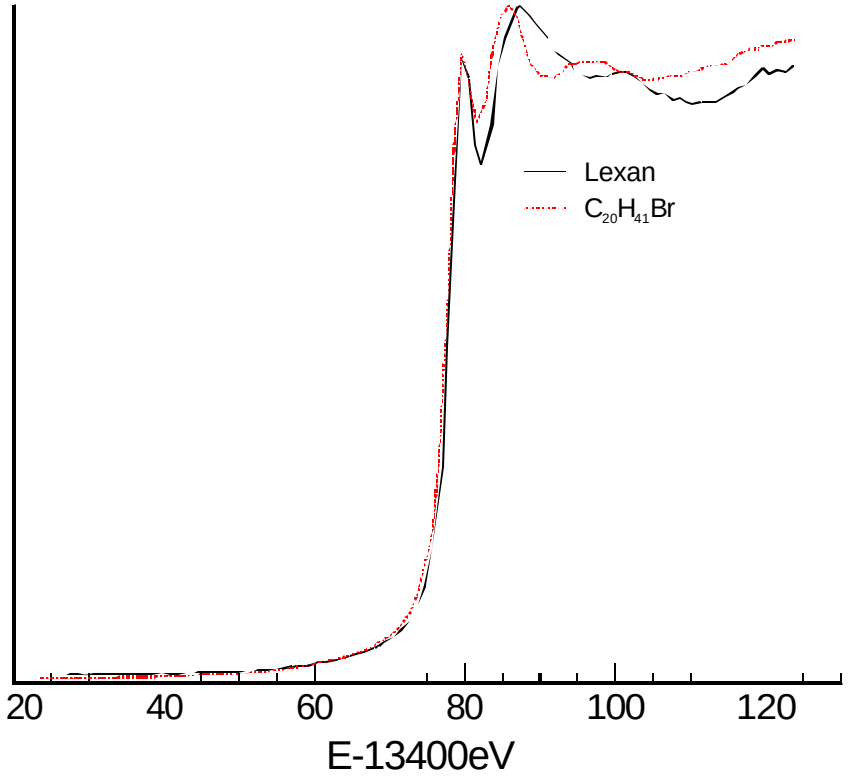
It's shutdown time, and once again, we're replacing M2. This time, it's a brand new mirror, on a bender which Jamie has assembled out of spares. The old bender will be removed so that if the mirror needs to be replaced yet again, we can pre-install the new mirror, get it bent, then plug in the whole assembly with minimum downtime. Another reason for building up a new bender assembly is that one of the picomotors on the old one has failed, so the assembly has to come out anyway.

The water-cooling lines on the mirrors have always been a source of problems with vacuum and for installation. They're also not even being used now. However, it seems reasonable that some form of temperature stabilization will be useful in keeping the beamline in tune. Thus, our latest plan is to make a block which mounts on the bar on

which M2-M4 mount and run water through that and only that. This step halves the number of fittings in the vacuum space and mechanically decouples the cooling lines from the mirrors, so that removing mirrors doesn't require undoing Gyro-Lok fittings. The hope is that the new lines will be vacuum-tight, so we can run water through them instead of pumping on them, as we do now.

Another part of our plan is to leak in some oxygen so as to provide an ozone clean. We figure something like 10^{-4}T will do it, and we hope that this low pressure won't result in ozone corrosion of wires and steel. We'll see! We'll use a leak valve so that it will be difficult to get anywhere near an atmosphere of O_2 in the box. Yes, I remember Apollo 1 and respect the dangers of pure oxygen.

Just before shutdown, I took a spectrum of the Br edge in some organo-Br samples. Organobromines show a distinct feature at the edge which was not resolved the first time we attempted it. It was well-resolved in this run:

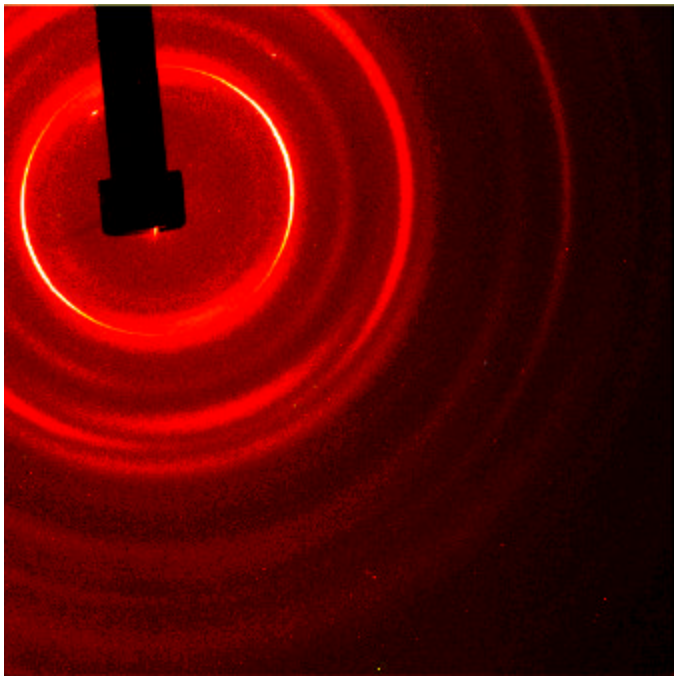


One of the samples is 1-bromo-n-eicosane, and the other is a random chunk of Lexan from the shop, both done in fluorescence with a 1eV step. It seems that Lexan and Plexiglas contain brominated chemicals, possibly as fire retardants. Thanks to Dave Edwards for pointing this out and providing the bromoalkane.

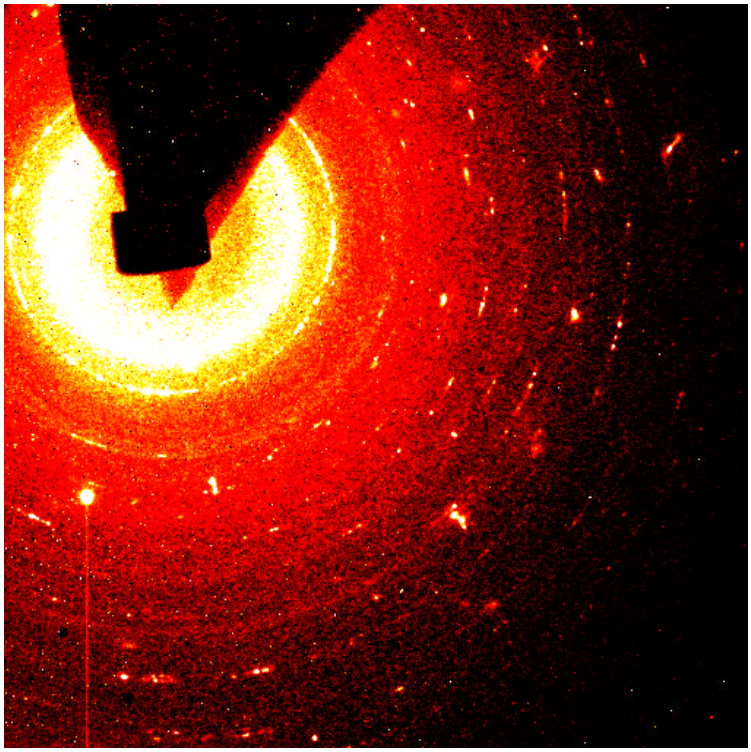
This result is significant because it gives us a clue as to how well M2 collimates the beam hitting the monochromator. When we come back up, we'll rerun that spectrum and see if it's still good. The Br edge is a good one for us to use for testing because it's at high energies, at which we're the most sensitive to angular divergence on the crystals.

Diffraction

We've made good use of the Bruker SMART6000 diffraction camera. We've used it for everything from CdSe nanoparticles to roots to magnetic semiconductors. Here's a pattern from CdSe nano-rods (M. A. Marcus, D. Michiko-Arguete, L. Li, unpublished). These rods form a nematic phase when suspended in sufficient concentration in a solvent. The idea is to do polarized EXAFS on the suspension, varying the angle between the rods and the electric field. We use the diffraction pattern to measure the orientation where the EXAFS is taken. The sharp reflections are the 002 basal-plane reflections of the wurzite structure. The *c*-axis is along the rods so these reflections have the least size broadening. The other reflections are broadened because the rods are only 3nm across.



This is an image taken from a root particle at 15keV for 60seconds. It's probably ZnO. The bright inner ring is due to the root body and is present if one moves off the particle. The spotty rings are due to the particle itself.



In both images, the dark shape which looks like the head of a bolt is the head of a bolt. It's used as a beamstop.

We've seen reflections with d-spacings to 1.4nm with 14keV radiation, by pulling the detector back to about 20cm from the sample.

It now appears that we'll have to trade the 6000 to another beamline. In exchange, we'll get a MAR detector, which has a larger active area and is said to be faster and more sensitive than the 6000. If all this is true, it will be a good thing indeed. We don't yet know if the trade is permanent, but it should last a while. One of Jamie's jobs is to build a new adaptor bracket for the MAR.

Software

The data-taking software has been pretty stable, with only minor, incremental changes. The analysis software has undergone a fair amount of continuous improvement and debugging. Unfortunately, the documentation has not kept pace. Among the

improvements: The FT program was cured of a bug which caused problems with the phase of the real and imaginary parts of the FT. The PCA program now lets you add and subtract spectra from the set being analyzed. The combination-fit program shows you the fit in Fourier as well as real space, which is very useful for deciding whether a set of reference really captures the important parts of what's in the unknown.

There are also new programs. On the mapping side, there's a program for correcting deadtime, using the same algorithm as the EXAFS Editor uses. There's also a program for registering maps to sub-pixel accuracy. This feature is needed for difference maps in which there is sometimes a drift of the sample position between maps. If this drift is not corrected, doing the difference results in edge detection and a '3D'-looking artifact. On the EXAFS end, there's a program for analyzing polarization effects, which was developed for the CdSe-nanoparticle project. This program is intended for samples of uniaxial symmetry. The idea is that one takes data with several values for the angle between the sample's axis and the polarization vector. Very general arguments then say that the EXAFS should follow a quadrupole form:

$$c(k) = c_0(k) + (c_0(k) - c_{90}(k))\cos^2 q \qquad (1)$$

where q is the angle between the two axes. The program looks at the data at each value of k and does a fit to the above equation. It then shows the fitted values for the two extreme signals $c_0(k)$ and $c_{90}(k)$ as well as 1-s confidence bands for the signals and their difference. One can thus see whether there's a polarization effect and how significant it is.

Website and documentation

You may have noticed some odd, up-and-down behavior from the website. This has to do with Bob having quit and me taking over (poorly) as Webmaster. I've gotten things a bit more under control, so things should be better. In particular, the analysis executables have been updated and should remain more current than they have been. I've also tracked down the cause of some misbehavior of the executables. There's a file called `lvanlys.dll` which needs to be in a directory called Data which must be a subdirectory of the one in which the executable resides. If this file is misplaced or missing, the executable may run, but not work correctly. That was a tough one to track down!

I have been remiss in updating documentation and writing manuals for the new programs. However, I have updated the troubleshooting guide. The one on the web may not be up to date, but the one on the beamline (accessible through a shortcut) is continually added to whenever a new problem is discovered. When you run into a problem on the beamline, read this, because someone else probably had the same problem and its description and solution are here.

Begging

All of us involved with the beamline would like to see it remain competitive as new lines are built elsewhere. One of the things which will eventually become necessary is to move the beamline to another source, such as a superbend or a wiggler. However, this takes money we don't have. We can't really go after grants ourselves, being part of DOE. However, there's nothing to prevent one or more user groups from getting together and finding a few hundred thousand dollars to give to our engineers. What this would

buy is improved flux and a useful energy range extending through the U L_3 edge at 17keV. As it is now, the flux is down two orders of magnitude at that energy, so little can be done up there. The flux would improve 10-fold or more at the Se-Y K-edges. I must admit that I don't really know how the politics works, but I gather that there could be an informal arrangement which would not be hampered by such things as some participants not being American, as can happen with other types of arrangement.

Schedule

The next cycle (June->December, 2003) is a long one, with the longest break being two weeks of 2-bunch time. However, we have a lot of user groups, each of which has its own constraints. The schedule is therefore going to be hard to change. Unfortunately for me, the User Office staff will not be able to continue managing the schedule for this cycle, so I'll have to do it. All I can suggest is that if any of you need to change your time, find someone to trade with, ask them, and if they're agreeable, then tell me about it. If you ask me to switch you, I'll just have to tell you to find a trading partner, because there's not much flexibility.

Until further notice, it looks as if I've finally achieved something I've wanted for two years now - a schedule without spillover into the next cycle. Let's see if we can keep it that way.

The January->May 2004 cycle will be a short one because there will be a long shutdown like the one we're in right now and for the same reasons. The current plan is to have such a shutdown every year for realignment of the whole ring. By doing this yearly, the hope is to keep the inevitable drifts from accumulating to the point where front-end components have to be moved.

Beam motion

I have some good news about beam motion. The machine physicists have put in new BPMs and correctors which allow feedback around all the undulators. This has cut way down on the drift we used to get between top and bottom of a fill, and also on the sensitivity to motions of the EPUs. We used to see non-normalizing signals whenever the Beamline 4 EPU was used for MCD measurement. We now occasionally see some small effects when it's doing MLD measurements, which involve a more-perturbing switching of the undulator. We still see effects in I_0, but most of that is due to changes in the beam size, about which we can't do much. There is a plan afoot to reduce the beam-size changes, but this hasn't happened yet. We just did some tests and found that the beam moved 1/4-1/2 micron under the worst-case manipulations of the undulators. Thanks to Christoph Steier and the rest of the staff for lending us some AP time and work to make these measurements.

The next step is to do some tests in which we artificially move the beam by swinging M1 and watch for the effect on the normalized EXAFS yield. The goal will be to see how significant such a source motion is. If we find that the measured motions are significant, then we have some ideas about X-ray BPMs and closed-loop feedback, which would perhaps get installed during the 2-bunch time in August.

Summary

It has been a productive half-year here, with new capabilities and new science. The beamline is well along on its transition from something on which to be experimented to something with which to do experiments. We are working on solving some long-

standing problems such as beam motion and sample manipulation. I would like to thank the users for their patience and participation, and my co-workers for their help.