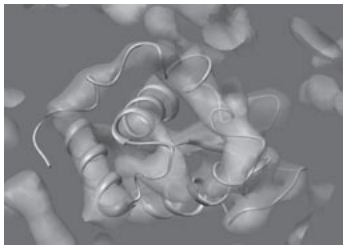
crystallography. Recently, an interest has grown for protein powder diffraction which is becoming a well-established method in the field of structure refinement and molecular replacement. With the use of two examples it is shown that de novo solutions to the phase problem can be obtained at low resolution via phasing methods such as the isomorphous replacement method. Using synchrotron radiation, high quality protein powder patterns have been collected in which pH- and radiation-induced anisotropic lattice changes were exploited in order to reduce the challenging and powder specific problem of overlapping reflections. The Single Isomorphous Replacement method enabled the computation of molecular envelopes and the mapping out of the solvent channels in the crystal. Electron density maps in which features of the secondary structure of the lysozyme protein molecule can be discerned, were then obtained using the Multiple Isomorphous Replacement method (as illustrated in the image).



Keywords: powder diffraction, protein structure determination, isomorphous replacement

MS.09.5

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Characterization of spider silks weaved by different species living in the Black sea region of Turkey

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Spider silks have attracted many researchers on account of their superior (Physical, chemical, biological and pharmacological) properties [1-4]. Our research group has begun to investigate spider silks with a project (TUBITAK, TBAG-107T017) after pre-studies[5]. The aims of this project are structural investigation of spider silk samples (in dragline and coccon forms) and obtaining some systematical information according to the different species(habitats, feeding and type of silk, etc.) First of all, our biologist group started to look for endemic species in The Backsea region of Turkey. They are indicating that, this geographic region is including very different spider species. During these researches, a lot of species, their draglines and egg-coccons were collected from their natural habitats. On the other hand, several living cavities (simulating their natural habitats) for the collected species were also constructed by our biologists in laboratory conditions. After these studies, a lot of silk samples produced by different species from Araneidae and Gnaphosidae familia were available. With this presentation, we would like to summarize our studies about structural investigation and characterization of the mentioned silks. Approximately, 15 samples were chosen and studied by using SEM, TEM, SWAXS, and XRD experimental methods. Alanine and glycine regions can be detected in XRD and SWAXS patterns due to their nanostructured and crystalline aggregations. XRD and SWAXS data have shown that the majority of these silks contain beta-plated sheet crystals that form from bilateral repeated aminoacid sequences rich in small aminoacid residues. At the end of the presentation, structural views, stabilizations and crystalinities of the samples will be compared.

Keywords: spider silk, Black Sea region, nanostructures, SWAXS, XRD, Araneidae, Gnaphosidae

MS.10.1

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Automation of the APS 11-BM high-resolution and high-throughput powder diffractometer

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The 11-BM powder diffractometer at the Argonne Advanced Photon Source operates with resolution on the order of $2x10^{-4}$ delta-Q/Q and collects an average diffraction pattern in ~1 hour. Equipped with a robotic sample changer, we expect this instrument to collect 20 or more datasets per day, primarily for remote users. Managing this level of use with minimal staffing has required that we optimize handling of sample metadata, as well as instrument control and data reduction. This talk will outline the database and web interface for 11-BM, which interfaces to the APS proposal and safety approval systems, as well as the instrument control system. A description of how users supply sample information and retrieve diffraction data via the web will be presented. The current instrument control software, which automatically calibrates the instrument, as well as streamlines data reduction, will also be discussed. Other topics to be presented include our current development plans, which will implement publication tracking and simplify sample storage and return/disposal. Progress on methods for automated review of diffraction data for internal consistency will also be presented.

Keywords: synchrotron powder diffraction, robots, automated data collection

MS.10.2

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Start to finish: Algorithms and parameters for successful robotic data collection

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Data acquisition automation implies robotics, and the appearance of new commercial robotic systems (such as the Rigaku ACTOR-SM) for small-molecule samples now requires algorithms that are robust and reliable but also flexible. To achieve fully automated crystal mounting, centering, data collection and processing (and optionally structure solution and refinement), a large number of parameters are necessary. Also, the system should be able to make intelligent decisions about whether to keep a given sample or to move on to the next one. These decisions can be made by applying the technique of *ranking* in order to aid in the decision process, for example to choose the best crystal from a group or to avoid wasting time collecting data on a sample that is unlikely to provide viable results. The choice of a minimum rank will depend on the purpose of the experiment;

a crystal of lower quality can be accepted if only a connectivity structure is needed but the requirements are more strict when a fully publishable result is required. The number of parameters considered in determining "rank" can be large. Algorithms may include the following parameters: resolution limits, $I/\sigma(I)$, spot sharpness, indexing quality, refinement quality, mosaicity, ice-ring detection, and others. The success of the experiment can be bolstered by the amount of information provided to the system, but a truly automatic system should be able to provide the necessary results with little or no human intervention. The ability to screen large numbers of crystals also allows tests for polymorphism, autoresolution of enantiomers, phase transformations, etc.

Keywords: robotics, automation algorithms, ranking

MS.10.3

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True walk-away automation in chemical crystallography

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The recent trend in the analytical instruments market towards compact benchtop systems spawned the development of fully automated low-cost X-ray diffractometers. This was made possible in part through advances in the analytical software, specifically new and improved algorithms and decision making expert systems. This paper presents an overview of the decision tree implemented in one such system and the individual steps involved in producing publication quality structures from single-crystals without user intervention. The design of the fully automated system is targeted primarily towards users who are not expert crystallographers. In addition to all decisions being made autonomously, this means keeping the user informed about the progression of the experiment in an easily comprehensible way. Equally important is to suggest remedies in case a problem is encountered that cannot be tackled in software, such as a poorly centered crystal, radiation induced crystal decay, or a temperature induced phase transition. The expert system proceeds through the following stages: quantify the crystal quality, determine the unit cell and the crystal symmetry, select a data collection strategy, acquire and reduce the diffraction data, scale the diffraction data, determine the space group, solve the phase problem, refine and validate the molecular structure, and finally generate a report. The results are provided as a Crystallographic Information File (CIF) and as a Hypertext Markup Language (HTML) report.

Keywords: automation, chemical crystallography, software

MS.10.4

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Rapid synchrotron X-ray crystallography for drug discovery using the SGX-CAT beamline at the APS

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SGX Pharmaceuticals, Inc., a San Diego-based oncology drug discovery company, designed, built, and operates a protein crystallography beamline (SGX-CAT) at the Advanced Photon Source (APS). This insertion device beamline operates exclusively as a mail-in facility, providing high-throughput X-ray crystallographic data collection for SGX, its corporate partners, and APS General Users. Screening of crystal quality and diffraction data collection at SGX-CAT utilize a series of automated processes, including crystal screening and data collection, sample loop visualization/centering, removal of surface ice from the sample, crystal quality scoring, and diffraction data processing/reduction. For most samples, crystal quality is evaluated and diffraction data are recorded without human intervention. All operations are tracked using a custom database, which links beamline operations to those elsewhere in the company and provides real-time access to information from the beamline. Expert systems evaluate the contents of the database, determine the next experiments to be performed, and, via web-based displays, summarize results therefrom. With this high-throughput approach, SGX makes routine use of synchrotron-based crystallography for de novo structure determination and for its FAST fragment-based, structure-guided drug discovery platform. During 2005, 2006, and 2007, 9379, 9934, and 10895 crystals were automatically screened at SGX-CAT, respectively. The number of diffraction datasets recorded at SGX-CAT averaged in excess of 4000/annum during the same period. Rapid access to crystallographic data proved critical for discovery of SGX523, a potent, selective, and orally-bioavailable c-MET inhibitor now in Phase I clinical trials for treatment of cancer patients.

Keywords: drug discovery and design, data collection methods, protein kinases

MS.10.5

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The interdependence of wavelength, redundancy and dose on a sulfur sad experiment

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In the last decade popularity of sulfur SAD anomalous dispersion experiments spread rapidly among synchrotron users as a quick and streamlined way for solving phase problem in macromolecular crystallography. On beam line 10 at SRS (Daresbury, UK) data sets have been collected on HEWL at six wavelengths between 0.979 Å and 2.290 Å to evaluate the importance and the interdependence of experimental variables like Bijvoet ratio, wavelength, resolution limit, redundancy, and absorbed dose in the sample per image. All the crystals used for the experiments were of high quality, so that the results could be interpreted independently of that. A feature of these experiments was the use of the detector tilt capability of the beam line to preserve high diffraction resolution even at the long wavelengths we tested. Radiation damage was found to affect disulfide bridges after the crystals have been given a total dose 2.5 $\times$ 10^6 Gγ. However with such a total dose, for all the data sets, it was possible to find a strategy to collect data to determine the sulfurs sub-structure and produce good quality phases by choosing the optimum combination of wavelength, exposure time and redundancy. $<|\delta_{ano}|/\sigma_{ano}>$ bigger than 1.0 for all resolution shells was a necessary requirement for a successful sulfur SAD substructure location. This work expands the general vision of a single wavelength anomalous