01.1–7 DATA COLLECTION FACILITY FOR STUDY OF BIOLOGICAL MACROMOLECULES USING SYNCHROTRON RADIATION. By R.M. Sweet, J.B. Hastings, W.C. Thomlinson, D.S. Wise, and B.P. Schoenborn, Department of Biology and National Synchrotron Light Source, Brookhaven National Laboratory, Upton, NY 11973.

A data collection facility is being constructed at the National Synchrotron Light Source (NSLS) x-ray storage ring. Characteristics of this facility are as follows. X-ray Source: The NSLS storage ring is designed to be operated at 2.5 GeV, 500 mA, with a critical energy of 4.2 keV for the bending magnet source. The electron beam dimensions will be only a few tenths of a millimeter at the tangent point. Monochromatization: Double single crystal monochromator of Ge or Si. Will be automated for rapid selection of wavelength for use of resonant scattering phenomena. Beam focussing: A single cylindrically cut (radius = 27 mm) longitudinally bent (radius = 3 km) mirror lies 9 m from the electron beam tangent point, 9 m from specimen. Collimation: The natural dimensions of the focussed beam will be 0.2 – 0.3 mm. Slits are used to limit the beam to smaller dimensions. The natural crossfire of the beam will be 0.25 mr in the vertical and 3 mr in the horizontal. Detector: X-ray film will be mounted either in flat cassettes with the beam normal to the plane (12.5 cm square) or in cylindrical cassettes with the axis of 10 cm radius curvature lying normal to the beam (12.5 x 18 cm). Detector distance: Flat film may be as close as 5.5 cm, cylindrical film as close as 10 cm. Data processing: Both film-scanning hardware and rotation-film scanning software are available within the Biology Department. This facility may be used by visitors to assess data quality, but most data processing will be done in visitors' home laboratories.

01.2–1 X-RAY CRYSTALLOGRAPHIC STUDIES ON LIPOAMIDEDEHYDROGENASE FROM AZOTOBACTER VINELANDII. By A.J.Schierbeek, J.Drenth and W.G.J.Hol,Lab.of Chem.Physics,Nijenborgh 16, 9747 AG Groningen, Netherlands.

Lipoamidedehydrogenase is an FAD containing enzyme which is part of the pyruvatedehydrogenase and α-ketoglutaric-acid dehydrogenase complex. In A.vinelandii it is a dimer with Mr= 112.000.
It catalyses the reoxidation of dihydrolipoic acid which is covalently bound in amide linkage to the core enzyme of the complex:

$$NAD^+ + \text{dihydrolipoamide} \rightleftarrows NADH + H^+ + \text{lipoamide}$$

It crystallizes as thin plates from solutions containing 10 mg/ml protein in 0.1 M K-phosphate pH 7.5 , 0.1 mM EDTA and 20% PEG 4000 in spacegroup $P2_12_12_1$, with a= 64.0 ,b = 83.8 and c = 193 Å and one dimer per asymmetric unit. The plates are enlarged by seeding the small crystals up to 1.0 x 0.6 x 0.2 mm^3.

The crystals are quite sensitive to radiation damage. The effective lifetime of the crystals could be prolonged with synchrotron radiation. In combination with cooling down to -10° C the resolution was increased up to 2.4 Å . Therefore a native dataset will be collected at DESY,EMBL at Hamburg using synchrotron radiation.

Lipoamidedehydrogenase displays extensive homology with Glutathionreductase. The threedimensional structure of the latter is known. Elucidation of the structure of lipoamidedehydrogenase will be essential to explain the functional differences between the enzyme and Glutathion reductase.

01.2–2 CRYSTAL STRUCTURE OF AZOTOBACTER CYTOCHROME C_5 AT 2.5 Å. By C. D. Stout, D. C. Carter, and S. O'Donnell, Dept. of Crystallography, University of Pittsburgh, Pittsburgh, PA 15260 USA.

The crystal structure of the dimeric cytochrome c_5 from Azotobacter vinelandii has been solved from a 2.5 Å resolution MIR map with figure of merit 0.71. The space group is C2 with a = 45.73, b = 37.56, c = 42.55 Å, β = 111.3°, where the dimer is superposed on the crystallographic twofold axis. The iodine derivative prepared with N-iodosuccinimide has two sites 1.7 and 2.0 Å from the 3' and 5' carbons of tyrosine 18. The platinum derivative prepared with $[PtI_6]^{2-}$ has three sites at the dimer interface. A model comprising 576 atoms was built into the MIR map and has been partially refined to R = 0.36 with 2.5 Å data.

The heme ligands are cysteines 19 and 22, and the Fe ligands are the heme, histidine 23 and methionine 63. The heme planes of the dimer form a 67° angle and the Fe-Fe distance is 16.4 Å across the dimer interface, which is comprised of hydrophobic contacts. Dimerization partially occludes the heme edge normally exposed to solvent in bacterial c-type cytochromes. However, both propionate groups of the heme are exposed to solvent on a face of the molecule containing an arginine and several lysines, and there is a marked clustering of aspartates and a glutamate on the opposite solvent-exposed face of the molecule. The overall polypeptide fold is homologous to cytochrome c_{551} (R.E.Dickerson, et al.), but the molecule is more compact, has a higher α-helical content, and contains a disulfide between cysteines 69 and 72.

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01.2–3 THE USE OF OPTIMISED ANOMALOUS DISPERSION IN PROTEIN CRYSTALLOGRAPHY FOR HEAVY ATOM LOCATION AND REFINEMENT, AND REFLECTION PHASING. By M Z Papiz[1], J R Helliwell[2], and D W J Cruickshank[2].

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The wavelength tunability of synchrotron x-radiation allows the scattering of specific atoms to be varied according to the standard relation for the atomic scattering factor

$$f = f_o + f'(\lambda) + if''(\lambda)$$

In protein crystallography the use of wavelengths close to the absorption edges of bound metal atoms allow the f' and f" coefficients to be optimised and varied in a controlled way. The changes induced in the diffraction pattern can be used to locate the anomalously scattering atom and to phase each reflection. In the case of two different metal atoms, eg Mn and Ca, the different values of f', f" for a given λ for each element allow the specific identification of the atoms bound to the protein and a better discrimination than would be available simply from the different atomic numbers of each atom.

To illustrate the accuracy of data required for the methods, results will be discussed on Fe:cytochrome c_4 (Sawyer, Harding, Gould, Damas, Papiz and Helliwell (Abstracts this meeting)) and Mn + Ca:pea lectin (Einspahr, Suguna, Suddath, Ellis, Helliwell and Papiz (Acta Cryst. submitted)) using a variety of wavelengths provided by the experimental workstation for protein crystallography on beam line 7 of the Daresbury SRS.