

8. SYNCHROTRON CRYSTALLOGRAPHY

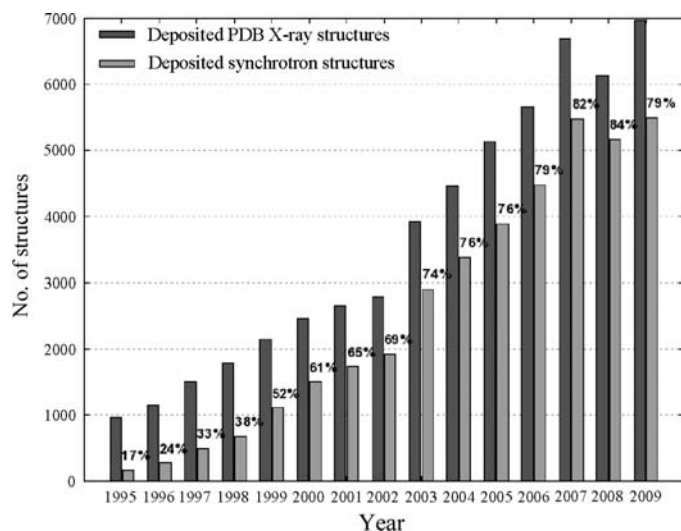


Figure 8.1.9.1

Synchrotron structures deposited in the PDB versus all PDB deposited structures (from <http://biosync.rcsb.org/BiosyncStat.html>) as of December 2009, i.e. a majority of all determined macromolecular crystal structures are now synchrotron-radiation derived.

discovery [for an early description see Bugg *et al.* (1993), for an example of pharmaceutical company collaboration see the Industrial Macromolecular Crystallography Association (IMCA) Collaborative Access Team at APS in Chicago (<http://www.imca.aps.anl.gov/>) and for a recent European perspective see *e.g.* Maclean *et al.* (2006).]

8.1.8.6. Radiation damage

The successes of these sources in macromolecular crystallography have been spectacular, so much so that provision of such beamlines now accounts for ~40% of the APS and ESRF insertion-device sectors. Nevertheless, X-radiation damage has been a continuing concern not least as the ‘cutting edge’ of capability to study ever-smaller crystals and ever-larger unit cells at synchrotron-radiation sources has been improved and optimized. A notable development has been a series of International Workshops on X-ray Damage to Crystalline Biological Samples. Most of these workshops have resulted in special issues of the *Journal of Synchrotron Radiation* (Volume 9 part 6, Volume 12 part 3, Volume 14 part 1 and Volume 16 part 2). The latest of these has a mini-review by Garman & Nave (2009).

8.1.9. Concluding remarks

SR and crystallography are now intricately intertwined in their scientific futures and in facilities provision (see *e.g.* Helliwell, 1998; Dauter, 2006; Fig. 8.1.9.1). Jiang & Sweet (2004) have given a systematic analysis of the impact of SR on macromolecular crystallography capabilities.

The new XFELs have produced very novel possibilities for 3D structure determination not only of non-crystallizable proteins but also whole cells (Neutze *et al.*, 2000; Miao *et al.*, 2001; Sayre, 2008; Shapiro, 2008). Whilst these approaches have attracted controversy, they represent a bold new push of X-ray diffraction methods towards widening capabilities in important frontiers in structural biology and structural cellular biology.

References

Abrahams, J. P. & Leslie, A. G. W. (1996). *Methods used in the structure determination of bovine mitochondrial F₁ ATPase*. *Acta Cryst.* **D52**, 30–42.

- Abrahams, J. P., Leslie, A. G. W., Lutter, R. & Walker, J. E. (1994). *Structure at 2.8 Å resolution of F₁-ATPase from bovine heart mitochondria*. *Nature (London)*, **370**, 621–628.
- Acharya, R., Fry, E., Stuart, D., Fox, G., Rowlands, D. & Brown, F. (1989). *The 3-dimensional structure of foot and mouth disease virus at 2.9 Å resolution*. *Nature (London)*, **337**, 709–716.
- Allinson, N. M. (1994). *Development of non-intensified charge-coupled device area X-ray detectors*. *J. Synchrotron Rad.* **1**, 54–62.
- Amemiya, Y. (1997). *X-ray storage phosphor imaging plate detectors: high sensitivity X-ray area detector*. *Methods Enzymol.* **276**, 233–243.
- Andrews, S. J., Hails, J. E., Harding, M. M. & Cruickshank, D. W. J. (1987). *The mosaic spread of very small crystals deduced from Laue diffraction patterns*. *Acta Cryst.* **A43**, 70–73.
- Arndt, U. W., Greenough, T. J., Helliwell, J. R., Howard, J. A. K., Rule, S. A. & Thompson, A. W. (1982). *Optimised anomalous dispersion crystallography: a synchrotron X-ray polychromatic simultaneous profile method*. *Nature (London)*, **298**, 835–838.
- Arnold, E., Vriend, G., Luo, M., Griffith, J. P., Kamer, G., Erickson, J. W., Johnson, J. E. & Rossmann, M. G. (1987). *The structure determination of a common cold virus, human rhinovirus 14*. *Acta Cryst.* **A43**, 346–361.
- Arzt, S., Campbell, J. W., Harding, M. M., Hao, Q. & Helliwell, J. R. (1999). *LSCALE – the new normalization, scaling and absorption correction program in the Daresbury Laue software suite*. *J. Appl. Cryst.* **32**, 554–562.
- Baker, P. J., Farrants, G. W., Stillman, T. J., Britton, K. L., Helliwell, J. R. & Rice, D. W. (1990). *Isomorphous replacement with optimized anomalous scattering applied to protein crystallography*. *Acta Cryst.* **A46**, 721–725.
- Ban, N., Freeborn, B., Nissen, P., Penczek, P., Grassucci, R. A., Sweet, R., Frank, J., Moore, P. B. & Steitz, T. A. (1998). *A 9 Å resolution X-ray crystallographic map of the large ribosomal subunit*. *Cell*, **93**, 1105–1115.
- Barber, J. (2009). *Photosynthetic energy conversion: natural and artificial*. *Chem. Soc. Rev.* **38**, 185–196.
- Bartunik, H. D., Clout, P. N. & Robrahn, B. (1981). *Rotation data collection for protein crystallography with time-variable incident intensity from synchrotron radiation sources*. *J. Appl. Cryst.* **14**, 134–136.
- Beuville, E., Beche, J. F., Cork, C., Douence, V., Earnest, T., Millaud, J., Nygren, D., Padmore, H., Turko, B., Zizka, G., Datte, P. & Xuong, N. H. (1997). *A 16 × 16 pixel array detector for protein crystallography*. *Nucl. Instrum. Methods*, **395**, 429–434.
- Bilderback, D. H. (1986). *The potential of cryogenic silicon and germanium X-ray monochromators for use with large synchrotron heat loads*. *Nucl. Instrum. Methods*, **246**, 434–436.
- Blakeley, M. P. (2009). *Neutron macromolecular crystallography*. *Crystallogr. Rev.* **15**, 157–218.
- Blewett, J. P. (1946). *Radiation losses in the induction electron accelerator*. *Phys. Rev.* **69**, 87–95.
- Blow, D. M. (2002). *Rearrangement of Cruickshank’s formulae for the diffraction-component precision index*. *Acta Cryst.* **D58**, 792–797.
- Bonse, U., Materlik, G. & Schröder, W. (1976). *Perfect-crystal monochromators for synchrotron X-radiation*. *J. Appl. Cryst.* **9**, 223–230.
- Bourenkov, G. P., Popov, A. N. & Bartunik, H. D. (1996). *A Bayesian approach to Laue diffraction analysis and its potential for time-resolved protein crystallography*. *Acta Cryst.* **A52**, 797–811.
- Bourgeois, D. & Weik, M. (2009). *Kinetic protein crystallography: a tool to watch proteins in action*. *Crystallogr. Rev.* **15**, 87–118.
- Brammer, R., Helliwell, J. R., Lamb, W., Liljas, A., Moore, P. R., Thompson, A. W. & Rathbone, K. (1988). *A new protein crystallography station on the SRS wiggler beamline for very rapid Laue and rapidly tunable monochromatic experiments: I. Design principles, ray tracing and heat calculations*. *Nucl. Instrum. Methods A*, **271**, 678–687.
- Branden, C. I. (1994). *The new generation of synchrotron machines*. *Structure*, **2**, 5–6.
- Brinkmann, R., Materlik, G., Rossbach, J., Schneider, J. R. & Wilk, B. H. (1997). *An X-ray FEL laboratory as part of a linear collider design*. *Nucl. Instrum. Methods*, **393**, No. 1–3, 86–92.
- Broennimann, Ch., Eikenberry, E. F., Henrich, B., Horisberger, R., Huelsen, G., Pohl, E., Schmitt, B., Schulze-Briese, C., Suzuki, M., Tomizaki, T., Toyokawa, H. & Wagner, A. (2006). *The PILATUS 1M detector*. *J. Synchrotron Rad.* **13**, 120–130.
- Bugg, C. E., Carson, W. M. & Montgomery, J. A. (1993). *Drugs by design*. *Sci. Am.* **269**, 60–66.

8.1. SYNCHROTRON RADIATION

- Campbell, J. W. & Hao, Q. (1993). *Evaluation of reflection intensities for the components of multiple Laue diffraction spots. II. Using the wavelength-normalization curve.* *Acta Cryst.* **A49**, 889–893.
- Caspar, D. L. D., Clarage, J., Salunke, D. M. & Clarage, M. (1988). *Liquid-like movements in crystalline insulin.* *Nature (London)*, **352**, 659–662.
- Cassetta, A., Deacon, A., Emmerich, C., Habash, J., Helliwell, J. R., McSweeney, S., Snell, E., Thompson, A. W. & Weisgerber, S. (1993). *The emergence of the synchrotron Laue method for rapid data collection from protein crystals.* *Proc. R. Soc. London Ser. A*, **442**, 177–192.
- Cauchois, Y., Bonnelle, C. & Missoui, G. (1963). *Rayonnement électromagnétique – premiers spectres X du rayonnement d'orbite du synchrotron de Frascati.* *C. R. Acad. Sci. Paris*, **257**, 409–412.
- Charpak, G. (1970). *Evolution of the automatic spark chambers.* *Annu. Rev. Nucl. Sci.* **20**, 195–254.
- Chayen, N. E., Boggon, T. J., Cassetta, A., Deacon, A., Gleichmann, T., Habash, J., Harrop, S. J., Helliwell, J. R., Nieh, Y. P., Peterson, M. R., Raftery, J., Snell, E. H., Hädener, A., Niemann, A. C., Siddons, D. P., Stojanoff, V., Thompson, A. W., Ursby, T. & Wulff, M. (1996). *Trends and challenges in experimental macromolecular crystallography.* *Q. Rev. Biophys.* **29**, 227–278.
- Chayen, N. E. & Helliwell, J. R. (1998). *Protein crystallography: the human genome in 3-D.* *Phys. World*, **11**, No. 5, 43–48.
- Cianci, M., Antonyuk, S., Bliss, N., Bailey, M. W., Buffey, S. G., Cheung, K. C., Clarke, J. A., Derbyshire, G. E., Ellis, M. J., Enderby, M. J., Grant, A. F., Holbourn, M. P., Laundry, D., Nave, C., Ryder, R., Stephenson, P., Helliwell, J. R. & Hasnain, S. S. (2005). *A high-throughput structural biology/proteomics beamline at the SRS on a new multipole wiggler.* *J. Synchrotron Rad.* **12**, 455–466.
- Cianci, M., Helliwell, J. R. & Suzuki, A. (2008). *The interdependence of wavelength, redundancy and dose in sulfur SAD experiments.* *Acta Cryst.* **D64**, 1196–1209.
- Cianci, M., Rizkallah, P. J., Olczak, A., Raftery, J., Chayen, N. E., Zagalsky, P. F. & Helliwell, J. R. (2001). *Structure of lobster apocrustacyanin A, using softer X-rays.* *Acta Cryst.* **D57**, 1219–1229.
- Colapietro, M., Cappuccio, G., Marcianite, C., Pifferi, A., Spagna, R. & Helliwell, J. R. (1992). *The X-ray diffraction station at the ADONE wiggler facility: preliminary results (including crystal perfection).* *J. Appl. Cryst.* **25**, 192–194.
- Cruickshank, D. W. J. (1999). *Remarks about protein structure precision.* *Acta Cryst.* **D55**, 583–601.
- Cruickshank, D. W. J., Helliwell, J. R. & Johnson, L. N. (1992). *Editors. Time-resolved Macromolecular Crystallography.* Oxford: OUP/The Royal Society.
- Cruickshank, D. W. J., Helliwell, J. R. & Moffat, K. (1987). *Multiplicity distribution of reflections in Laue diffraction.* *Acta Cryst.* **A43**, 656–674.
- Cruickshank, D. W. J., Helliwell, J. R. & Moffat, K. (1991). *Angular distribution of reflections in Laue diffraction.* *Acta Cryst.* **A47**, 352–373.
- Dauter, Z. (2006). *Current state and prospects of macromolecular crystallography.* *Acta Cryst.* (2006). **D62**, 1–11.
- Dauter, Z., Lamzin, V. S. & Wilson, K. S. (1997). *The benefits of atomic resolution.* *Curr. Opin. Struct. Biol.* **7**, 681–688.
- Deacon, A., Gleichmann, T., Kalb (Gilboa), A. J., Price, H., Raftery, J., Bradbrook, G., Yariv, J. & Helliwell, J. R. (1997). *The structure of concanavalin A and its bound solvent determined with small-molecule accuracy at 0.94 Å resolution.* *J. Chem. Soc. Faraday Trans.* **93**, 4305–4312.
- Doniach, S., Hodgson, K., Lindau, I., Pianetta, P. & Winick, H. (1997). *Early work with synchrotron radiation at Stanford.* *J. Synchrotron Rad.* **4**, 380–395.
- Doucet, J. & Benoit, J. P. (1987). *Molecular dynamics by analysis of the X-ray diffuse scattering from lysozyme crystals.* *Nature (London)*, **325**, 643–646.
- Dumas, C., Duquerroy, S. & Janin, J. (1995). *Phasing with mercury at 1 Å wavelength.* *Acta Cryst.* **D51**, 814–818.
- Eikenberry, E. F., Barna, S. L., Tate, M. W., Rossi, G., Wixted, R. L., Sellin, P. J. & Gruner, S. M. (1998). *A pixel-array detector for time-resolved X-ray diffraction.* *J. Synchrotron Rad.* **5**, 252–255.
- Einspahr, H., Suguna, K., Suddath, F. L., Ellis, G., Helliwell, J. R. & Papiz, M. Z. (1985). *The location of manganese and calcium ion cofactors in pea lectin crystals by use of anomalous dispersion and tuneable synchrotron X-radiation.* *Acta Cryst.* **B41**, 336–341.
- Ellemaume, P. (1989). *Special synchrotron radiation sources. Part 1: conventional insertion devices.* *Synchrotron Radiat. News*, **1**, 18–23.
- Ellemaume, P. (1998). *Two-plane focusing of 30 keV undulator radiation.* *J. Synchrotron Rad.* **5**, 1–5.
- ESRF Foundation Phase Report (1987). Grenoble: ESRF.
- Ferreira, K. N., Iverson, T. M., Maghlaoui, K., Barber, J. & Iwata, S. (2004). *Architecture of the photosynthetic oxygen-evolving center.* *Science*, **303**, 1831–1838.
- Fourme, R. (1997). *Position-sensitive gas detectors: MWPCs and their gifted descendants.* *Nucl. Instrum. Methods A*, **392**, 1–11.
- Fourme, R., Kahn, R., Mezouar, M., Girard, E., Hoerentrup, C., Prangé, T. & Ascone, I. (2001). *High-pressure protein crystallography (HPPX): instrumentation, methodology and results on lysozyme crystals.* *J. Synchrotron Rad.* **8**, 1149–1156.
- Freund, A. K. (1996). *Third-generation synchrotron radiation X-ray optics.* *Structure*, **4**, 121–125.
- Garman, E. F. & Nave, C. (2009). *Radiation damage in protein crystals examined under various conditions by different methods.* *J. Synchrotron Rad.* **16**, 129–132.
- Girard, E., Dhaussy, A.-C., Couzinet, B., Chervin, J.-C., Mezouar, M., Kahn, R., Ascone, I. & Fourme, R. (2007). *Toward fully fledged high-pressure macromolecular crystallography.* *J. Appl. Cryst.* **40**, 912–918.
- Glover, I. D., Harris, G. W., Helliwell, J. R. & Moss, D. S. (1991). *The variety of X-ray diffuse scattering from macromolecular crystals and its respective components.* *Acta Cryst.* **B47**, 960–968.
- Gonzalez, A., Cohen, A., Eriksson, T., McPhillips, S., Moorhead, P., Narevicius, J., Sharp, K., Smith, C., Song, J. & Soltis, S. M. (2005). *Facility updates: remote access to the SSRL macromolecular crystallography beamlines.* *Synchrotron Radiat. News*, **18**, 36–39.
- Greenhough, T. J. & Helliwell, J. R. (1982). *Oscillation camera data processing: reflecting range and prediction of partiality. II. Monochromatised synchrotron X-radiation from a singly bent triangular monochromator.* *J. Appl. Cryst.* **15**, 493–508.
- Grimes, J. M., Burroughs, J. N., Gouet, P., Diprose, J. M., Malby, R., Zientara, S., Mertens, P. P. C. & Stuart, D. I. (1998). *The atomic structure of the bluetongue virus core.* *Nature (London)*, **395**, 470–478.
- Grimes, J. M., Jakana, J., Ghosh, M., Basak, A. K., Roy, P., Chiu, W., Stuart, D. I. & Prasad, B. V. V. (1997). *An atomic model of the outer layer of the bluetongue virus core derived from X-ray crystallography and electron cryomicroscopy.* *Structure*, **5**, 885–893.
- Gruner, S. M. & Ealick, S. E. (1995). *Charge coupled device X-ray detectors for macromolecular crystallography.* *Structure*, **3**, 13–15.
- Guillot, B., Jelsch, C., Podjarny, A. & Lecomte, C. (2008). *Charge-density analysis of a protein structure at subatomic resolution: the human aldose reductase case.* *Acta Cryst.* **D64**, 567–588.
- Guss, J. M., Merritt, E. A., Phizackerley, R. P., Hedman, B., Murata, M., Hodgson, K. O. & Freeman, H. C. (1988). *Phase determination by multiple wavelength X-ray diffraction – crystal structure of a basic blue copper protein from cucumbers.* *Science*, **241**, 806–811.
- Habash, J., Raftery, J., Nuttall, R., Price, H. J., Wilkinson, C., Kalb (Gilboa), A. J. & Helliwell, J. R. (2000). *Direct determination of the positions of the deuterium atoms of the bound water in concanavalin A by neutron Laue crystallography.* *Acta Cryst.* **D56**, 541–550.
- Habash, J., Raftery, J., Weisgerber, S., Cassetta, A., Lehmann, M. S., Hoghoj, P., Wilkinson, C., Campbell, J. W. & Helliwell, J. R. (1997). *Neutron Laue diffraction study of concanavalin A: the proton of Asp28.* *J. Chem. Soc. Faraday Trans.* **93**, 4313–4317.
- Hädener, A., Matzinger, P. K., Battersby, A. R., McSweeney, S., Thompson, A. W., Hammersley, A. P., Harrop, S. J., Cassetta, A., Deacon, A., Hunter, W. N., Nieh, Y. P., Raftery, J., Hunter, N. & Helliwell, J. R. (1999). *Determination of the structure of selenomethionine-labelled hydroxymethylbilane synthase in its active form by multi-wavelength anomalous dispersion.* *Acta Cryst.* **D55**, 631–643.
- Hajdu, J., Acharya, K. R., Stuart, D. I., McLaughlin, P. J., Barford, D., Oikonomakos, N. G., Klein, H. & Johnson, L. N. (1987). *Catalysis in the crystal – synchrotron radiation studies with glycogen phosphorylase.* *EMBO J.* **6**, 539–546.
- Hajdu, J., Machin, P. A., Campbell, J. W., Greenhough, T. J., Clifton, I. J., Zurek, S., Gover, S., Johnson, L. N. & Elder, M. (1987). *Millisecond X-ray diffraction and the first electron density map from Laue photographs of a protein crystal.* *Nature (London)*, **329**, 178–181.
- Hall, G. (1995). *Silicon pixel detectors for X-ray diffraction studies at synchrotron sources.* *Q. Rev. Biophys.* **28**, 1–32.
- Harmsen, A., Leberman, R. & Schultz, G. E. (1976). *Comparison of protein crystal diffraction patterns and absolute intensities from synchrotron and conventional X-ray sources.* *J. Mol. Biol.* **104**, 311–314.

8. SYNCHROTRON CRYSTALLOGRAPHY

- Hart, M. (1971). *Bragg reflection X-ray optics*. *Rep. Prog. Phys.* **34**, 435–490.
- Haslegrove, J. C., Faruqi, A. R., Huxley, H. E. & Arndt, U. W. (1977). *The design and use of a camera for low-angle X-ray diffraction experiments with synchrotron radiation*. *J. Phys. E*, **10**, 1035–1044.
- Hastings, J. B. (1977). *X-ray optics and monochromators for synchrotron radiation*. *J. Appl. Phys.* **48**, 1576–1584.
- Hastings, J. B., Kincaid, B. M. & Eisenberger, P. (1978). *A separated function focusing monochromator system for synchrotron radiation*. *Nucl. Instrum. Methods*, **152**, 167–171.
- Hedman, B., Hodgson, K. O., Helliwell, J. R., Liddington, R. & Papiz, M. Z. (1985). *Protein micro-crystal diffraction and the effects of radiation damage with ultra high flux synchrotron radiation*. *Proc. Natl Acad. Sci. USA*, **82**, 7604–7607.
- Helliwell, J. R. (1977). *Application of synchrotron radiation to protein crystallography: preliminary experiments on 6PDGH crystals using the NINA synchrotron, Daresbury, UK*. DPhil thesis, University of Oxford, Appendix 1.
- Helliwell, J. R. (1979). *Optimisation of anomalous scattering and structural studies of proteins using synchrotron radiation*. In *Proceedings of the Study Weekend. Applications of SR to the Study of Large Molecules of Chemical and Biological Interest*, edited by R. B. Cundall & I. H. Munro, pp. 1–6. DL/SCI/R13. Warrington: Daresbury Laboratory.
- Helliwell, J. R. (1984). *Synchrotron X-radiation protein crystallography: instrumentation, methods and applications*. *Rep. Prog. Phys.* **47**, 1403–1497.
- Helliwell, J. R. (1985). *Protein crystallography with synchrotron radiation*. *J. Mol. Struct.* **130**, 63–91.
- Helliwell, J. R. (1988). *Protein crystal perfection and the nature of radiation damage*. *J. Crystal Growth*, **90**, 259–272.
- Helliwell, J. R. (1992). *Macromolecular Crystallography with Synchrotron Radiation*. Cambridge University Press.
- Helliwell, J. R. (1993). *The choice of X-ray wavelength in macromolecular crystallography*. In *Computational Aspects of Data Collection*, compiled by S. Bailey, pp. 80–88. DL/SCI/R34. Warrington: Daresbury Laboratory.
- Helliwell, J. R. (1997a). *Overview on synchrotron radiation and application in macromolecular crystallography*. *Methods Enzymol.* **276**, 203–217.
- Helliwell, J. R. (1997b). *Neutron Laue diffraction does it faster*. *Nature Struct. Biol.* **4**, 874–876.
- Helliwell, J. R. (1998). *Synchrotron radiation and crystallography: the first fifty years*. *Acta Cryst. A54*, 738–749.
- Helliwell, J. R. (2004). *Overview and new developments in softer X-ray ($2 \text{ \AA} < \lambda < 5 \text{ \AA}$) protein crystallography*. *J. Synchrotron Rad.* **11**, 1–3.
- Helliwell, J. R., Ealick, S., Doing, P., Irving, T. & Szebenyi, M. (1993). *Towards the measurement of ideal data for macromolecular crystallography using synchrotron sources*. *Acta Cryst. D49*, 120–128.
- Helliwell, J. R., Habash, J., Cruickshank, D. W. J., Harding, M. M., Greenhough, T. J., Campbell, J. W., Clifton, I. J., Elder, M., Machin, P. A., Papiz, M. Z. & Zurek, S. (1989). *The recording and analysis of synchrotron X-radiation Laue diffraction photographs*. *J. Appl. Cryst.* **22**, 483–497.
- Helliwell, J. R., Nieh, Y. P., Raftery, J., Cassetta, A., Habash, J., Carr, P. D., Ursby, T., Wulff, M., Thompson, A. W., Niemann, A. C. & Hädener, A. (1998). *Time-resolved structures of hydroxymethylbilane synthase (Lys59Gln mutant) as it is loaded with substrate in the crystal determined by Laue diffraction*. *J. Chem. Soc. Faraday Trans.* **94**, 2615–2622.
- Helliwell, J. R., Papiz, M. Z., Glover, I. D., Habash, J., Thompson, A. W., Moore, P. R., Harris, N., Croft, D. & Pantos, E. (1986). *The wiggler protein crystallography workstation at the Daresbury SRS; progress and results*. *Nucl. Instrum. Methods A*, **246**, 617–623.
- Helliwell, J. R. & Rentzepis, P. M. (1997). *Editors. Time-resolved Diffraction*. Oxford University Press.
- Helliwell, J. R. & Wilkinson, C. (1994). *X-ray and neutron Laue diffraction*. In *Neutron and Synchrotron Radiation for Condensed Matter Studies*, Vol. 3, edited by J. Baruchel, J. L. Hodeau, M. S. Lehmann, J. R. Regnard & C. Schlenker, ch. 12. Berlin: Springer Verlag.
- Hendrickson, W. A. (1985). *Analysis of protein structure from diffraction measurements at multiple wavelengths*. *Trans. Am. Crystallogr. Assoc.* **21**, 11–21.
- Hendrickson, W. A., Horton, J. R. & LeMaster, D. M. (1990). *Selenomethionyl proteins produced for analysis by multi-wavelength anomalous diffraction (MAD) – a vehicle for direct determination of 3-dimensional structure*. *EMBO J.* **9**, 1665–1672.
- Hendrickson, W. A., Pahler, A., Smith, J. L., Satow, Y., Merritt, E. A. & Phizackerley, R. P. (1989). *Crystal structure of core streptavidin determined from multi-wavelength anomalous diffraction of synchrotron radiation*. *Proc. Natl Acad. Sci. USA*, **86**, 2190–2194.
- Herzenberg, A. & Lau, H. S. M. (1967). *Anomalous scattering and the phase problem*. *Acta Cryst.* **22**, 24–28.
- Holmes, K. C. (1998). *A molecular model for muscle contraction*. *Acta Cryst. A54*, 789–797.
- Hope, H., Frolow, F., von Böhlen, K., Makowski, I., Kratky, C., Halfon, Y., Danz, H., Webster, P., Bartels, K. S., Wittmann, H. G. & Yonath, A. (1989). *Cryocrystallography of ribosomal particles*. *Acta Cryst. B45*, 190–199.
- Hoppe, W. & Jakubowski, V. (1975). *The determination of phases of erythrocrucorin using the two-wavelength method with iron as an anomalous scatterer*. In *Anomalous Scattering*, edited by S. Ramaseshan & S. C. Abrahams, pp. 437–461. Copenhagen: Munksgaard.
- Huxley, H. E. & Holmes, K. C. (1997). *Development of synchrotron radiation as a high-intensity source for X-ray diffraction*. *J. Synchrotron Rad.* **4**, 366–379.
- Iwanenko, D. & Pomeranchuk, I. (1944). *On the maximal energy attainable in a betatron*. *Phys. Rev.* **65**, 343.
- Jakoncic, J., Di Michiel, M., Zhong, Z., Honkimaki, V., Jouanneau, Y. & Stojanoff, V. (2006). *Anomalous diffraction at ultra-high energy for protein crystallography*. *J. Appl. Cryst.* **39**, 831–841.
- Jiang, J. & Sweet, R. M. (2004). *Protein Data Bank depositions from synchrotron sources*. *J. Synchrotron Rad.* **11**, 319–327.
- Kahn, R., Fourme, R., Bosshard, R., Chiadmi, M., Risler, J. L., Dideberg, O. & Wery, J. P. (1985). *Crystal structure study of opsin-tau parvalbumin by multi-wavelength anomalous dispersion*. *FEBS Lett.* **170**, 133–137.
- Karle, J. (1967). *Anomalous scatterers in X-ray diffraction and the use of several wavelengths*. *Appl. Opt.* **6**, 2132–2135.
- Karle, J. (1980). *Some anomalous dispersion developments for the structure investigation of macromolecular systems in biology*. *Int. J. Quantum Chem. Symp.* **7**, 357–367.
- Karle, J. (1989). *Macromolecular structure from anomalous dispersion*. *Phys. Today*, **42**, 22–29.
- Karle, J. (1994). *Developments in anomalous scattering for structure determination*. In *Resonant Anomalous X-ray Scattering*, edited by G. Materlik, C. J. Sparks & K. Fischer, pp. 145–158. Amsterdam: North Holland.
- Kohra, K., Ando, M., Matsushita, T. & Hashizume, H. (1978). *Design of high resolution X-ray optical system using dynamical diffraction for synchrotron radiation*. *Nucl. Instrum. Methods*, **152**, 161–166.
- Korszun, Z. R. (1987). *The tertiary structure of azurin from pseudomonas-denitrificans as determined by Cu resonant diffraction using synchrotron radiation*. *J. Mol. Biol.* **196**, 413–419.
- Lairson, B. M. & Bilderback, D. H. (1982). *Transmission X-ray mirror – a new optical element*. *Nucl. Instrum. Methods*, **195**, 79–83.
- Lemonnier, M., Fourme, R., Rousseaux, F. & Kahn, R. (1978). *X-ray curved-crystal monochromator system at the storage ring DCI*. *Nucl. Instrum. Methods*, **152**, 173–177.
- Lewis, R. (1994). *Multiwire gas proportional counters: decrepit antiques or classic performers?* *J. Synchrotron Rad.* **1**, 43–53.
- Liddington, R. C., Yan, Y., Moulai, J., Sahli, R., Benjamin, T. L. & Harrison, S. C. (1991). *Structure of simian virus-40 at 3.8 Å resolution*. *Nature (London)*, **354**, 278–284.
- Liljas, A., Liljas, L., Piskur, J., Lindblom, G., Nissen, P. & Kjeldgaard, M. (2009). *Textbook of Structural Biology*. Singapore: World Scientific.
- Lindley, P. F. (1999). *Macromolecular crystallography with a third-generation synchrotron source*. *Acta Cryst. D55*, 1654–1662.
- Liu, Y., Ogata, C. & Hendrickson, W. (2001). *Multiwavelength anomalous diffraction analysis at the M absorption edges of uranium*. *Proc. Natl Acad. Sci. USA*, **98**, 10648–10653.
- Lovelace, J., Snell, E. H., Pokross, M., Arvai, A. S., Nielsen, C., Xuong, N.-H., Bellamy, H. D. & Borgstahl, G. E. O. (2000). *BEAM-ish: a graphical user interface for the physical characterization of macromolecular crystals*. *J. Appl. Cryst.* **33**, 1187–1188.
- Luger, K., Mader, A. W., Richmond, R. K., Sargent, D. F. & Richmond, T. J. (1997). *Crystal structure of the nucleosome core particle at 2.8 Å resolution*. *Nature (London)*, **389**, 251–260.

8.1. SYNCHROTRON RADIATION

- Luger, P. (2007). *Fast electron density methods in the life sciences – a routine application in the future?* *Org. Biomol. Chem.* **5**, 2529–2540.
- McDermott, G., Prince, S. M., Freer, A. A., Hawthornthwaite-Lawless, A. M., Papiz, M. Z., Cogdell, R. J. & Isaacs, N. W. (1995). *Crystal structure of an integral membrane light harvesting complex from photosynthetic bacteria.* *Nature (London)*, **374**, 517–521.
- Maclean, E. J., Rizkallah, P. J. & Helliwell, J. R. (2006). *Protein crystallography and synchrotron radiation; current status and future landscape.* *Eur. Pharm. Rev.* **2**, 71–76.
- Mayans, O. & Wilmanns, M. (1999). *X-ray analysis of protein crystals with thin-plate morphology.* *J. Synchrotron Rad.* **6**, 1016–1020.
- Miao, J., Hodgson, K. O. & Sayre, D. (2001). *An approach to three-dimensional structures of biomolecules by using single-molecule diffraction images.* *Proc. Natl Acad. Sci. USA*, **98**, 6641–6645.
- Miller, A. (1994). *Advanced Synchrotron Sources – Plans at ESRF in SR in Biophysics*, edited by S. S. Hasnain. Chichester: Ellis Horwood.
- Mills, D. M., Helliwell, J. R., Kvik, Å., Ohta, T., Robinson, I. A. & Authier, A. (2005). *Report of the Working Group on Synchrotron Radiation Nomenclature – brightness, spectral brightness or brilliance?* *J. Synchrotron Rad.* **12**, 385.
- Moffat, K., Szebenyi, D. & Bilderback, D. H. (1984). *X-ray Laue diffraction from protein crystals.* *Science*, **223**, 1423–1425.
- Mukherjee, A. K., Helliwell, J. R. & Main, P. (1989). *The use of MULTAN to locate the positions of anomalous scatterers.* *Acta Cryst.* **A45**, 715–718.
- Munro, I. H. (1997). *Synchrotron radiation research in the UK.* *J. Synchrotron Rad.* **4**, 344–358.
- Neutze, R. & Hajdu, J. (1997). *Femtosecond time resolution in X-ray diffraction experiments.* *Proc. Natl Acad. Sci. USA*, **94**, 5651–5655.
- Neutze, R., Wouts, R., Spoel, D., Weckert, E. & Hajdu, J. (2000). *Potential for biomolecular imaging with femtosecond X-ray pulses.* *Nature (London)*, **406**, 752–757.
- Nieh, Y. P., Raftery, J., Weisgerber, S., Habash, J., Schotte, F., Ursby, T., Wulff, M., Hädener, A., Campbell, J. W., Hao, Q. & Helliwell, J. R. (1999). *Accurate and highly complete synchrotron protein crystal Laue diffraction data using the ESRF CCD and the Daresbury Laue software.* *J. Synchrotron Rad.* **6**, 995–1006.
- Niemann, A. C., Matzinger, P. K. & Hadener, A. (1994). *A kinetic analysis of the reaction catalysed by hydroxymethylbilane synthase.* *Helv. Chim. Acta*, **77**, 1791–1809.
- Noller, H. F. (2005). *RNA structure: reading the ribosome.* *Science*, **309**, 1508–1514.
- Nugent, K. (2009). *Coherent methods in the X-ray sciences.* *Adv. Phys.* **59**, 1–99.
- Okaya, Y. & Pepinsky, R. (1956). *New formulation and solution of the phase problem in X-ray analysis of non-centric crystals containing anomalous scatterers.* *Phys. Rev.* **103**, 1645–1647.
- Parratt, L. G. (1959). *Use of synchrotron orbit-radiation in X-ray physics.* *Rev. Sci. Instrum.* **30**, 297–299.
- Pebay-Peyroula, E., Rummel, G., Rosenbusch, J. P. & Landau, E. M. (1997). *X-ray structure of bacteriorhodopsin at 2.5 Å from microcrystals grown in lipidic cubic phases.* *Science*, **277**, 1676–1681.
- Perrakis, A., Cipriani, F., Castagna, J.-C., Claustre, L., Burghammer, M., Riek, C. & Cusack, S. (1999). *Protein microcrystals and the design of a microdiffractometer: current experience and plans at EMBL and ESRF/ID13.* *Acta Cryst.* **D55**, 1765–1770.
- Peterson, M. R., Harrop, S. J., McSweeney, S. M., Leonard, G. A., Thompson, A. W., Hunter, W. N. & Helliwell, J. R. (1996). *MAD phasing strategies explored with a brominated oligonucleotide crystal at 1.65 Å resolution.* *J. Synchrotron Rad.* **3**, 24–34.
- Phillips, J. C. & Hodgson, K. O. (1980). *The use of anomalous scattering effects to phase diffraction patterns from macromolecules.* *Acta Cryst.* **A36**, 856–864.
- Phillips, J. C., Templeton, L. K., Templeton, D. H. & Hodgson, K. O. (1978). *L-III edge anomalous X-ray scattering by cesium measured with synchrotron radiation.* *Science*, **201**, 257–259.
- Phillips, J. C., Wlodawer, A., Goodfellow, J. M., Watenpugh, K. D., Sieker, L. C., Jensen, L. H. & Hodgson, K. O. (1977). *Applications of synchrotron radiation to protein crystallography. II. Anomalous scattering, absolute intensity and polarization.* *Acta Cryst.* **A33**, 445–455.
- Phillips, J. C., Wlodawer, A., Yevitz, M. M. & Hodgson, K. O. (1976). *Applications of synchrotron radiation to protein crystallography: preliminary results.* *Proc. Natl Acad. Sci. USA*, **73**, 128–132.
- Polikarpov, I., Teplyakov, A. & Oliva, G. (1997). *The ultimate wavelength for protein crystallography?* *Acta Cryst.* **D53**, 734–737.
- Ren, Z. & Moffat, K. (1995a). *Quantitative analysis of synchrotron Laue diffraction patterns in macromolecular crystallography.* *J. Appl. Cryst.* **28**, 461–481.
- Ren, Z. & Moffat, K. (1995b). *Deconvolution of energy overlaps in Laue diffraction.* *J. Appl. Cryst.* **28**, 482–493.
- Riek, C. (2000). *New avenues in X-ray microbeam experiments.* *Rep. Prog. Phys.* **63**, 233–262.
- Riek, C., Burghammer, M. & Schertler, G. (2005). *Protein crystallography microdiffraction.* *Curr. Opin. Struct. Biol.* **15**, 556–562.
- Rosenbaum, G., Holmes, K. C. & Witz, J. (1971). *Synchrotron radiation as a source for X-ray diffraction.* *Nature (London)*, **230**, 434–437.
- Rossmann, M. G., Arnold, E., Erickson, J. W., Frankenberger, E. A., Griffith, J. P., Hecht, H. J., Johnson, J. E., Kamer, G., Luo, M., Mosser, A. G., Rueckert, R. R., Sherry, B. & Vriend, G. (1985). *Structure of a human common cold virus and functional relationship to other picornaviruses.* *Nature (London)*, **317**, 145–153.
- Rossmann, M. G. & Erickson, J. W. (1983). *Oscillation photography of radiation-sensitive crystals using a synchrotron source.* *J. Appl. Cryst.* **16**, 629–636.
- Sakabe, N. (1983). *A focusing Weissenberg camera with multi-layer-line screens for macromolecular crystallography.* *J. Appl. Cryst.* **16**, 542–547.
- Sakabe, N. (1991). *X-ray diffraction data collection system for modern protein crystallography with a Weissenberg camera and an imaging plate using synchrotron radiation.* *Nucl. Instrum. Methods A*, **303**, 448–463.
- Sakabe, N., Ikemizu, S., Sakabe, K., Higashi, T., Nagakawa, A. & Watanabe, N. (1995). *Weissenberg camera for macromolecules with imaging plate data collection at the Photon Factory – present status and future plan.* *Rev. Sci. Instrum.* **66**, 1276–1281.
- Sayre, D. (2008). *Report on a project on three-dimensional imaging of the biological cell by single-particle X-ray diffraction.* *Acta Cryst.* **A64**, 33–35.
- Schiltz, M. & Bricogne, G. (2009). *Instrument-independent specification of the diffraction geometry and polarization state of the incident X-ray beam.* *J. Appl. Cryst.* **42**, 101–108.
- Schiltz, M., Kvik, Å., Svensson, O. S., Shepard, W., de La Fortelle, E., Prangé, T., Kahn, R., Bricogne, G. & Fourme, R. (1997). *Protein crystallography at ultra-short wavelengths: feasibility study of anomalous-dispersion experiments at the xenon K-edge.* *J. Synchrotron Rad.* **4**, 287–297.
- Schwinger, J. (1949). *On the classical radiation of accelerated electrons.* *Phys. Rev.* **75**, 1912–1925.
- Shapiro, D. A. (2008). *Report on a project on three-dimensional imaging of the biological cell by single-particle X-ray diffraction.* *Addendum.* *Acta Cryst.* **A64**, 36–37.
- Snell, E. H., Weisgerber, S., Helliwell, J. R., Weckert, E., Hölzer, K. & Schroer, K. (1995). *Improvements in lysozyme protein crystal perfection through microgravity growth.* *Acta Cryst.* **D51**, 1099–1102.
- Srajer, V., Teng, T. Y., Ursby, T., Pradervand, C., Ren, Z., Adachi, S., Schildkamp, W., Bourgeois, D., Wulff, M. & Moffat, K. (1996). *Photolysis of the carbon monoxide complex of myoglobin: nanosecond time-resolved crystallography.* *Science*, **274**, 1726–1729.
- Stuhrmann, H. B. & Lehmann, M. S. (1994). *Anomalous dispersion of X-ray scattering from low Z elements.* In *Resonant Anomalous X-ray Scattering*, edited by G. Materlik, C. J. Sparks & K. Fischer, pp. 175–194. Berlin: Springer Verlag.
- Sugahara, M., Asada, Y., Shimada, H., Taka, H. & Kunishima, N. (2009). *HATODAS II – heavy-atom database system with potentiality scoring.* *J. Appl. Cryst.* **42**, 540–544.
- Tate, M. W., Eikenberry, E. F., Barna, S. L., Wall, M. E., Lowrance, J. L. & Gruner, S. M. (1995). *A large-format high-resolution area X-ray detector based on a fiber-optically bonded charge-coupled device (CCD).* *J. Appl. Cryst.* **28**, 196–205.
- Templeton, D. H. & Templeton, L. K. (1985). *X-ray dichroism and anomalous scattering of potassium tetrachloroplatinate(II).* *Acta Cryst.* **A41**, 365–371.
- Templeton, D. H., Templeton, L. K., Phillips, J. C. & Hodgson, K. O. (1980). *Anomalous scattering of X-rays by cesium and cobalt measured with synchrotron radiation.* *Acta Cryst.* **A36**, 436–442.
- Templeton, L. K., Templeton, D. H., Phizackerley, R. P. & Hodgson, K. O. (1982). *L₃-edge anomalous scattering by gadolinium and samarium measured at high resolution with synchrotron radiation.* *Acta Cryst.* **A38**, 74–78.

8. SYNCHROTRON CRYSTALLOGRAPHY

- Teplyakov, A., Oliva, G. & Polikarpov, I. (1998). *On the choice of an optimal wavelength in macromolecular crystallography*. *Acta Cryst.* **D54**, 610–614.
- Tsukihara, T., Aoyama, H., Yamashita, E., Tomizaki, T., Yamaguchi, H., Shinzawa-Itoh, K., Nakashima, R., Yaono, R. & Yoshikawa, S. (1996). *The whole structure of the 13-subunit oxidized cytochrome c oxidase at 2.8 Å*. *Science*, **272**, 1136–1144.
- Usha, R., Johnson, J. E., Moras, D., Thierry, J. C., Fourme, R. & Kahn, R. (1984). *Macromolecular crystallography with synchrotron radiation: collection and processing of data from crystals with a very large unit cell*. *J. Appl. Cryst.* **17**, 147–153.
- Warren, J. E., Diakun, G., Bushnell-Wye, G., Fisher, S., Thalal, A., Helliwell, M. & Helliwell, J. R. (2008). *Science experiments via telepresence at a synchrotron radiation source facility*. *J. Synchrotron Rad.* **15**, 191–194.
- Webb, N. G., Samson, S., Stroud, R. M., Gamble, R. C. & Baldeschwieler, J. D. (1977). *A focusing monochromator for small-angle diffraction studies with synchrotron radiation*. *J. Appl. Cryst.* **10**, 104–110.
- Weckert, E. & Hümmel, K. (1997). *Multiple-beam X-ray diffraction for physical determination of reflection phases and its applications*. *Acta Cryst.* **A53**, 108–143.
- Westbrook, E. M. & Naday, I. (1997). *Charge-coupled device-based area detectors*. *Methods Enzymol.* **276**, 244–268.
- Wilson, K. S., Stura, E. A., Wild, D. L., Todd, R. J., Stuart, D. I., Babu, Y. S., Jenkins, J. A., Standing, T. S., Johnson, L. N., Fourme, R., Kahn, R., Gadet, A., Bartels, K. S. & Bartunik, H. D. (1983). *Macromolecular crystallography with synchrotron radiation. II. Results*. *J. Appl. Cryst.* **16**, 28–41.
- Wimberley, B. T., Brodersen, D. E., Clemons, W. M., Morgan-Warren, R. J., Carter, A. P., Vonrhein, C., Hartsch, T. & Ramakrishnan, V. (2000). *Structure of the 30S ribosomal subunit*. *Nature (London)*, **407**, 327–339.
- Winick, H. (1995). *The linac coherent light source (LCLS): a fourth-generation light source using the SLAC linac*. *J. Electron Spectrosc. Relat. Phenom.* **75**, 1–8.
- Yano, J., Kern, J., Irrgang, K. D., Latimer, M. J., Bergmann, U., Glatzel, P., Pushkar, Y., Biesiadka, J., Loll, B., Sauer, K., Messinger, J., Zouni, A. & Yachandra, V. K. (2005). *X-ray damage to the Mn4Ca complex in single crystals of photosystem II: a case study for metalloprotein crystallography*. *Proc. Natl Acad. Sci. USA*, **102**, 12047–12052.
- Yonath, A. (1992). *Approaching atomic resolution in crystallography of ribosomes*. *Annu. Rev. Biophys. Biomol. Struct.* **21**, 77–93.
- Yonath, A., Harms, J., Hansen, H. A. S., Bashan, A., Schlünzen, F., Levin, I., Koelln, I., Tocilj, A., Agmon, I., Peretz, M., Bartels, H., Bennett, W. S., Krumbholz, S., Janell, D., Weinstein, S., Auerbach, T., Avila, H., Piolletti, M., Morlang, S. & Franceschi, F. (1998). *Crystallographic studies on the ribosome, a large macromolecular assembly exhibiting severe nonisomorphism, extreme beam sensitivity and no internal symmetry*. *Acta Cryst.* **A54**, 945–955.