International Tables for Crystallography (2012). Vol. F, Section 8.1.6, pp. 194–195.

Table 8.1.5.1
Structures in the Protein Data Bank (PDB) for which data were collected at the SRS

The data presented here were compiled in December 2009 (see http://biosync.rcsb.org/biosync_regions/SyncEurope.html#SRS) and are likely to be reasonably complete since the SRS closed operations in August 2008. The SRS has delivered 3.6% of the total of 38 650 macromolecular crystal structures determined using radiation from synchrotrons around the world as of December 2009. The ESRF third-generation source, in comparison, integrated over about half as many years, but about two to three times more beamlines, has delivered 15.6% of the structures, *i.e.* at a rate therefore about three to four times greater than the second-generation SRS.

	Station						
Year	10.1	14.1	14.2	7.2	9.5	9.6	Not known
1995	0	0	0	5	6	19	3
1996	0	0	0	6	11	33	0
1997	0	0	0	11	29	43	0
1998	0	0	0	26	32	35	0
1999	0	0	0	28	13	45	4
2000	0	1	0	28	17	60	3
2001	0	13	9	9	16	47	2
2002	0	13	21	7	7	59	2
2003	0	27	38	3	8	41	3
2004	3	40	42	5	2	36	2
2005	18	34	36	1	4	47	1
2006	22	32	22	1	1	23	0
2007	21	37	21	0	0	11	1
2008	51	15	15	2	0	20	1
2009	14	12	5	1	0	3	0
Total	129	224	209	133	146	522	22

documents and workshops, and the ESR Project (ESRP) led by B. Buras and based in Geneva at CERN, culminated in the so-called 'Red Book' in 1987, the *ESRF Foundation Phase Report* (1987), totalling some 1000 pages of machine, beamline and experimental specifications and costs. This, then, was the progenitor of the third-generation sources, characterized by their high energy and high spectral brightness, tailored to optimized undulator emission in the 1 Å range. Actually, the ESRF machine energy was initially set at 5 GeV, but increased to 6 GeV to optimize the production of 14.4 keV photons to better match the nuclear scattering experiments proposed initially by Mossbauer in 1975. Proposals for the US machine, the Advanced Photon Source at 7 GeV, and the Japanese 8 GeV SPring-8 machine followed, with the higher machine energy enhancing the X-ray tuning range of undulators. Thus, MAD tuning-based techniques were facilitated with these machines and studies involving yet-smaller samples (crystals, single fibres or tiny liquid aliquots) or very large unit cells were enabled. As a result, micron-sized protein crystals as well as huge multi-macromolecular biological structures (of large viruses, for example) also became routinely accessible.

8.1.5.4. New national SR machines

Today a variety of enhanced national SR machines have been built. In Switzerland there is the SLS, in the UK there is DIAMOND and in France there is SOLEIL. These machines are more tailored to the bulk of a country's user needs, distinct from the special provisions at the ESRF. The different countries' SR needs, of course, have many aspects in common, with some historical biases. The new sources are, in essence, characterized by high spectral brightness, *i.e.*, low emittance. The 2 GeV SR source ELETTRA in Trieste, the MAXII machine in LUND and the Brazilian Light Source are already operational. In many ways, national sources like the SRS, LURE, DORIS and so on fuelled the case and specification for the ESRF. Now the developments at the ESRF, including high harmonic emission of undulators *via* magnet shimming (Elleaume, 1989) and narrow-gap undulator operation (Elleaume, 1998), are fuelling ideas and the specification of what is possible in the new national SR sources.

8.1.5.5. X-ray free-electron lasers (XFELs)

In terms of the evolution of X-ray sources, X-ray FELs are being constructed at DESY in Hamburg (Brinkmann *et al.*, 1997), at SLAC (Winick, 1995) and at Spring8. Compared to SR, one will have a transversely fully coherent beam, a larger average spectral brightness and, in particular, pulse lengths of ~10 fs full width at half-maximum with eight to ten orders of magnitude larger peak spectral brightness. Such a machine is based on a linear accelerator (linac)-driven XFEL utilizing a linear collider installation (*e.g.*, for a high-energy physics centre-of-mass energy capability of 500 GeV). For this machine there is a 'switchyard' distributing the electrons in a beam to different undulators from which the X-rays are generated in the range 0.1 to ~12 keV. The anticipated r.m.s. opening angle would be 1 mrad and the source diameter would be 20 μm. This source of X-rays would then compete in time resolution with laser-pulse-generated X-ray beams [see Helliwell & Rentzepis (1997) for a survey of that work and a comparison with synchrotron radiation] and would also have higher pulse flux. Coherent methods in the X-ray sciences have been extensively reviewed by Nugent (2009).

8.1.6. SR instrumentation

The divergent continuum of X-rays from the source must be intercepted by the sample cross-sectional area. The crystal sample acceptance, as seen above, is a good way to illustrate to the machine designer the sort of machine emittances required. Likewise, the beamline optics, mirrors and monochromators should not degrade the X-ray beam quality. Mirror surface and shape finish have improved a great deal in the last few decades; slope errors of mirrors, even for difficult shapes like polished cylinders, which on bending give a toroidal reflecting surface, are now around 1 arc second (5.5 μrad) for a length of 1 m. Thus, over focusing distances of 10–20 m, say, the focal-spot smearing contribution from this is 55–110 μm, important for focusing onto small crystals. Further optics developments (*e.g.* Fresnel optics) have yielded micron focus beams and smaller, and are being applied to studying ever-smaller crystals in macromolecular crystallography and obviously have a variety of other diffraction and spectroscopy applications [for a review, see Riekel (2000)]. The choice of materials has evolved, too, from the relatively easy-to-work-with and -finish fused quartz to silicon; silicon having the advantageous property that at liquid-nitrogen temperature the expansion coefficient is zero (Bilderback, 1986). This has been of particular advantage in the cooling of silicon monochromators at the ESRF, where the heat loading on optics is very high. An alternative approach with the rather small X-ray beams from undulators is the use of transparent monochromator crystals made of diamond, which is a robust material with the additional advantage of transparency, thus allowing multiplexing of stations, one downstream from the other, fed by one straight section of one or more undulator designs. For a review of the ESRF beamline optics, see Freund (1996); for reviews of the macromolecular crystallography programmes at the ESRF, see Miller (1994), Branden (1994) and Lindley (1999), as well as the *ESRF Foundation Phase Report* (1987). See also Helliwell (1992), Chapter 5.

Detectors have improved enormously. The early days of SR use saw considerable reliance on photographic film, as well as single-counter four-circle diffractometers. Evolution of area detectors, in particular, has been considerable and impressive, and in a variety of technologies. Gas detectors, *i.e.*, the multiwire proportional chamber (MWPC), were invented and developed through various generations and types [Charpak (1970); for reviews of their use at SR sources, see *e.g.* Lewis (1994) and Fourme (1997)]. MWPCs have the best detector quantum efficiency (DQE) of the area detectors, but there are limitations on count rate (local and global) and their use at wavelengths less than ~1 Å is restricted due to geometric image parallax effects. The most popular devices at present are charge coupled devices (CCDs) [see Tate *et al.* (1995), Allinson (1994), Gruner & Ealick (1995) and Westbrook & Naday (1997) for details of their development]. Image plates (IPs) were popular during the late 1980s and early to mid-1990s, mainly, but not exclusively, with online scanners, notably the MAR Research devices. IPs are also used in a Weissenberg geometry [see Sakabe (1983, 1991) and Sakabe *et al.* (1995), and for a recent review see Amemiya (1997)]. IPs and CCDs are complementary in performance, especially with respect to size and duty cycle; IPs are larger, *i.e.*, with many resolution elements possible, but are slower to read out than CCDs. Both are capable of imaging well at wavelengths shorter than 1 Å and with high count rates. Both have overcome the tedium of chemical development of film. Other detectors needed for crystallography include those for monitoring the beam intensity; these must not interfere with the beam collimation, and yet must monitor the beam downstream of the collimator (Bartunik *et al.*, 1981); also needed are fluorescence detectors for setting the wavelength for optimized anomalous-scattering applications (see Cianci *et al.*, 2005).

Most recently, an area-detector development has been the so-called pixel detector. This is made of silicon cells, each 'bump bonded' onto associated individual electronic readout chains. Thus, extremely high count rates are possible. These devices can then combine the attributes of large image plate sensitive areas with the fast readout of CCDs, along with high count-rate capability and so on. Devices and prototypes have been developed at Princeton/Cornell (Eikenberry *et al.*, 1998), Berkeley/San Diego (Beuville *et al.* 1997) and Imperial College, London (Hall, 1995), and are now in use at the SLS (Broennimann *et al.*, 2006).

Provision of robotics for sample mounting on the synchrotron beamlines has been increasingly deployed in the last decade, improving efficiency and ease of use, often coupled with remote access (*e.g.* see Gonzalez *et al.*, 2005) and telepresence (*e.g.* see Warren *et al.*, 2008).

8.1.7. SR monochromatic and Laue diffraction geometry

In the utilization of SR, both Laue and monochromatic modes are important for data collection. The unique geometric and spectral properties of SR render the treatment of diffraction geometry different from that for a conventional X-ray source.

8.1.7.1. Laue geometry: sources, optics, sample reflection bandwidth and spot size

Laue geometry involves the use of the polychromatic SR spectrum as transmitted through the beryllium window that is used to separate the apparatus from the machine vacuum. There is useful intensity down to a wavelength minimum of $\sim\lambda_c/5$, where λ_c is the critical wavelength of the magnet source in the case of bending magnets (BMs) and wavelength shifters. The maximum wavelength is typically ≥ 3 Å; however, if the crystal is mounted in a capillary, then the glass absorbs the wavelengths beyond ~2.5 Å (Helliwell, 2004).

The bandwidth on BMs and wigglers can be limited by a reflecting mirror at grazing incidence, whereby the minimum wavelength in the beam can be sharply defined to aid the accurate definition of the Laue spot multiplicity. The mirror can also be used to focus the beam at the sample. The maximum-wavelength limit can be truncated by use of aluminium absorbers of varying thickness or a transmission mirror (Lairson & Bilderback, 1982; Cassetta *et al.*, 1993).

The measured intensity of individual Laue diffraction spots depends on the wavelength at which they are stimulated. The problem of wavelength normalization is treated by a variety of methods. These include:

(i) use of a monochromatic reference data set;
(ii) use of symmetry equivalents and multiple measurements in the Laue data set recorded at different wavelengths;
(iii) calibration with a standard sample, such as a silicon crystal.

Each of these methods produces a 'λ curve' describing the relative strength of spots measured at various wavelengths. Most Laue diffraction data are now recorded on CCDs or IPs. The greater sensitivity of these detectors (expressed as the DQE), especially for weak signals, has greatly increased the number of Laue exposures recordable per crystal (*e.g.* Nieh *et al.*, 1999). Thus, multiplet deconvolution procedures, based on the recording of reflections stimulated at different wavelengths and with different relative intensities, have become possible (Campbell & Hao, 1993; Ren & Moffat, 1995*b*; Nieh *et al.*, 1999). Data quality and completeness have improved considerably.

Narrow-bandpass beams, *e.g.* $\delta\lambda/\lambda \leq 0.2$, are used for enhancing the signal-to-noise ratio. Such bandwidths are produced generally by an X-ray undulator, whereby *e.g.* 10–100 periods should ideally yield a bandwidth behind a pinhole of $\delta\lambda/\lambda \simeq 0.1$–0.01. In these cases, wavelength normalization is more difficult, because the actual spectrum over which a reflection is integrated is rapidly varying in intensity; nevertheless, high-order Chebychev polynomials are successful (Ren & Moffat, 1995*a*; Artz *et al.*, 1999).

The spot bandwidth is determined by the mosaic spread and horizontal beam divergence (since $\gamma_H > \gamma_V$) as

$$(\delta\lambda/\lambda) = (\eta + \gamma_H)\cot\theta, \qquad (8.1.7.1)$$

where η is the sample mosaic spread, assumed to be isotropic, and

references