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The MX Beamlines BL14.1-3 at BESSY II

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Abstract: The Macromolecular Crystallography (MX) group at the Helmholtz-Zentrum Berlin (HZB) is operating three state-of-the-art synchrotron beamlines for MX at BESSY II in Berlin (Heinemann et al., 2003; Mueller et al., 2012, 2015). The radiation source for all three beamlines BL14.1-3 is a superconducting 7T-wavelength shifter. Currently, the three beam lines are the most productive stations for MX in Germany, with about 250 PDB depositions per year and over 1500 PDB depositions in total (Status 10/2015). BL14.1 and BL14.2 are energy tuneable in the range 5.5-15.5 keV, while beam line BL14.3 is a fixed-energy side station operated at 13.8 keV. The HZB-MX beamlines are in regular user operation providing close to 200 beam days per year and about 600 user shifts to approximately 100 research groups across Europe. Additional user facilities include office space adjacent to the beam lines, a sample preparation laboratory, a biology laboratory (safety level 1) and high-end computing resources.

1 Introduction

BL14.1. is a tuneable energy beamline dedicated to MX (Fig 1). The beamline is highly automated and users can access the whole energy range without assistance by beamline staff. The endstation features an MD2 microdiffractometer with a very small sphere-of-confusion of 1 μm. This setup can support X-ray diffraction experiments of small crystals down to 15 μm in size. A mini-kappa goniometer is permanently installed for special applications. Samples may be mounted using a CATS sample changer robot (IRELEC, France). Rapid data collection experiments are possible with the PILATUS 6M pixel-detector (DECTRIS, Switzerland). At this station the whole range of MX experiments is possible, from rapid crystal screening to MAD data collection and long-wavelength applications. A special feature of

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Figure 1: Experimental station of beamline BL14.1

this beamline is that it is equipped with a pulsed UV-laser for radiation-damage induced phase determination experiments.

BL14.2. is a tuneable energy beamline dedicated to MX (Fig. 2). Just like on BL14.1 (see previous paragraph) the whole energy range is easily accessible. This beamline has just undergone a complete upgrade. It features a nano-diffractometer, which has been built in-house in collaboration with DESY, and on-axis sample viewing. Samples may be mounted from a large dewar, which can house up to 300 samples, using a GROB sample changer robot (NatX-ray, France). Rapid data collection experiments are possible with the PILATUS3S 2M pixel-detector (DECTRIS, Switzerland). It is planned to develop this beamline in terms of experiment automation further in order to optimally support fragment-screening experiments. The possibility to collect microspectrophotometric data from crystals is also foreseen on this beamline.

BL14.3. is a fixed-energy beamline dedicated to MX (Fig. 3). Unlike BL14.1 and BL14.2 this beamline is presently not fully automated. The endstation consists of a MAR-dtb goniometer (marXperts, Germany) and an MX225 CCD-detector (Rayonix, USA). A specialty of this beamline is the possibility to carry out crystal dehydration experiments using an HC1c crystal humidity controller (Arinax, France) (Bowler et al., 2015).

2 Instrument applications

The HZB-MX beamlines (Heinemann et al., 2003; Mueller et al., 2012, 2015) are designed and built to support all possible MX experiments in a reliable and user friendly way:

- Standard MX data collection
- Kappa-based data collection for low symmetry space groups
- MAD/SAD data collection for de novo structure determination
- Long wavelength data collection for native SAD phasing purposes

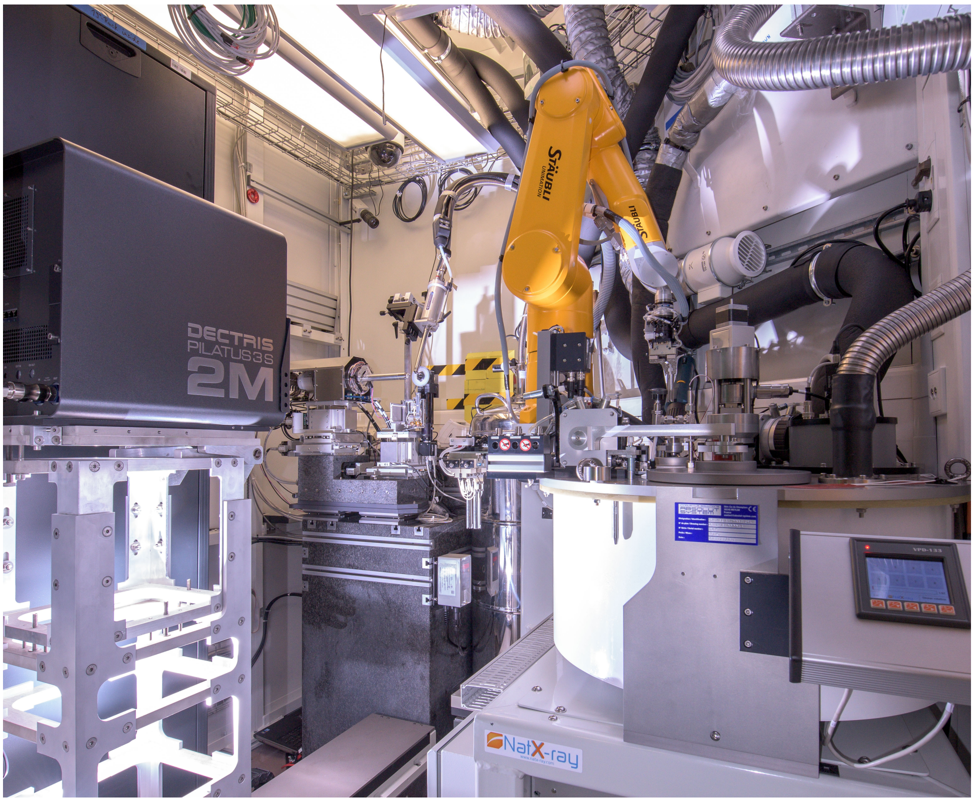

Figure 2: Experimental station of beamline BL14.2

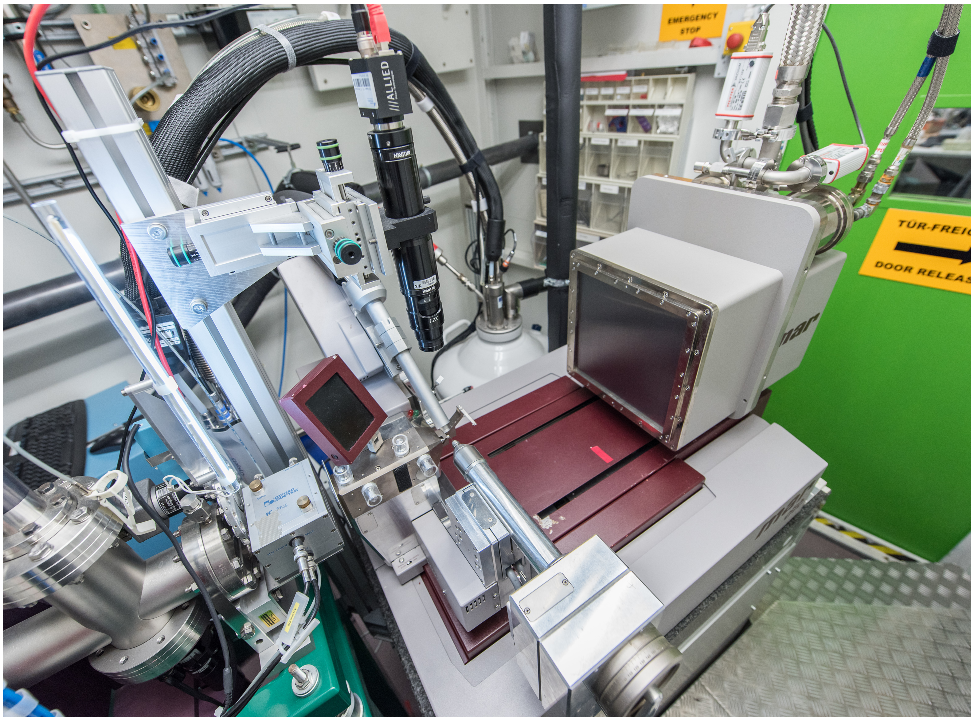

Figure 3: Experimental station of beamline BL14.3

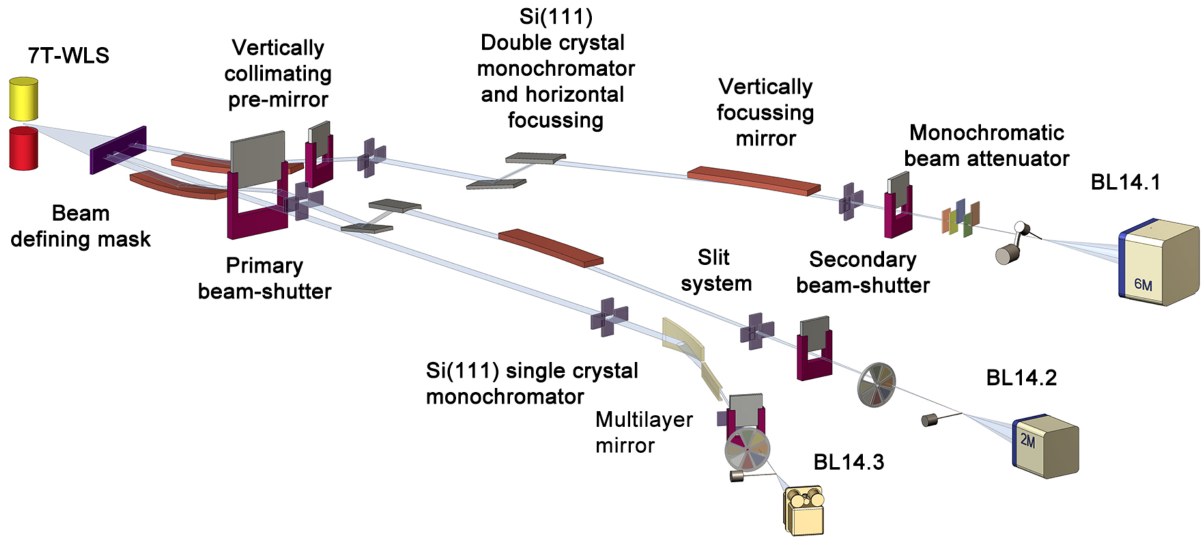

Figure 4: Optical layout of the HZB-MX-beam lines BL14.1-3.

- Ultra-high resolution data collection
- Fragment-screening experiments for drug discovery
- High throughput crystal screening
- UV radiation damage phasing
- Noble gas based phasing and solvent channel mapping
- Qualitative Element analysis by X-ray fluorescence
- Room temperature data collection
- Crystal dehydration (Bowler et al., 2015).

3 Source

The insertion device is the superconducting 7T wavelength shifter 7T-WLS-2 (Budker Institute of Nuclear Physics, Novosibirsk, Russia) with the following parameters:

Type	7 T Superconducting WLS
Location	Low beta-section 14
Periods	3 n

Table 1: Parameters of the 7T-WLS-2.

4 Optical Design

All three beamlines BL14.1-3 are fed from the same insertion device; hence they share the same optics hutch (Fig. 4). Both tunable energy beamlines BL14.1 and BL14.2 consist of two front-end sections, which include a vertically collimating Si-mirror coated with a 50 nm Rh-layer and a primary beam-shutter. In the joint optics hutch an indirectly water-cooled Si(111)-double crystal monochromator with horizontal focusing and a vertically focusing mirror coated with a 50 nm Rh-layer are installed. BL14.3 shares within the front-end section the primary beam-shutter with BL14.2. Further downstream the beam is deflected and horizontally focussed by an asymmetrically cut Si(111)-single crystal monochromator and vertically focussed by a lateral gradient cylindrically shaped multi-layer-mirror. A secondary beamshutter completes the set-up.

5 Technical Data

Location	15.2		
Source	7T-WLS-2		
Beamline	BL14.1	BL14.2	BL14.3
Monochromator	Si111-DCM with sagital bender	Si111-DCM with sagital bender	Si111 Single crystal monochromator
Energy range	5 - 15.5 keV	5 - 15.5 keV	13.8 keV
Energy resolution	<2 eV	<2 eV	<5 eV
Flux	1.4×10^{11} (Photons/s/100 mA)	2.0×10^{11} (Photons/s/100 mA)	5×10^{10} (Photons/s/100 mA)
Polarization	Horizontal	Horizontal	Horizontal
Divergence horizontal	1.5 mrad	1.5 mrad	0.5 mrad
Divergence vertical	0.5 mrad	0.5 mrad	0.5 mrad
Focus size (hor. x vert.)	150x100 μm^2	150x100 μm^2	200x100 μm^2
Experiment in vacuum	No	No	No
Temperature range	90 – 293 K	90 - 293 K	90 - 293 K
Detector(s)	•X-ray fluorescence detector: Amptek XR123 SDD •Pixel-detector: Pilatus 6M	•X-ray fluorescence detector: Amptek XR123 SDD •Pixel-detector: Pilatus 2M	•X-ray fluorescence detector: Amptek XR123 SDD •MARCCD-225 mm
Sample manipulators	CATS sample changer	GROB sample change	None
Goniometer	Microdiffractometer MD2 with Minikappa goniometer MK3	Nanodiffractometer	MAR-dtb

Table 2: Parameters of the 7T-WLS-2.

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