

BIODIFF: Diffractometer for large unit cells

Heinz Maier-Leibnitz Zentrum
Technische Universität München
Forschungszentrum Jülich, Jülich Centre for Neutron Science

Instrument Scientists:

- Andreas Ostermann, Heinz Maier-Leibnitz Zentrum (MLZ), Technische Universität München, Garching, Germany, phone: +49(0) 89 289 14702, email: andreas.ostermann@frm2.tum.de
- Tobias E. Schrader, Jülich Centre for Neutron Science (JCNS) at Heinz Maier-Leibnitz Zentrum (MLZ), Forschungszentrum Jülich GmbH, Garching, Germany, phone: +49(0) 89 289 10743, email: t.schrader@fz-juelich.de

Abstract: The single crystal diffractometer BIODIFF, which is jointly operated by the Technische Universität München and JCNS, Forschungszentrum Jülich, is designed to handle crystals with large unit cells and is dedicated to the structure determination of biological macromolecules. The main field of application is the neutron structure analysis of proteins, especially the determination of hydrogen atom positions.

1 Introduction

In biological macromolecules, like proteins and nucleic acids, hydrogen atoms play an important role. Hydrogen atoms take part in the substrate binding process and are essential for proton transfer reactions during the catalysis in many enzymes. Therefore, the knowledge about the protonation states of amino acid residues in the active centre of proteins is often crucial for the understanding of their reaction mechanisms. However, hydrogen atoms, especially rather flexible ones, are barely detectable in X-ray structure determinations of proteins. On the other hand, hydrogen atoms are clearly visible in neutron crystallography experiments even at moderate resolutions ($d_{\min} < 2.0 \text{ \AA}$).

BIODIFF is the first instrument along the cold neutron guide NL1 and is positioned in a distance of about 32.5 m from the cold source (Figure 1). Using a pyrolytic graphite monochromator PG(002) the diffractometer covers a tunable wavelength range of 2.4 Å to about 5.6 Å. Higher order wavelength contaminations are removed by a neutron velocity selector. The main detector of the diffractometer consists of a neutron imaging plate system in a cylindrical geometry to cover a large solid angle. A fast LiF/ZnS scintillator CCD camera is foreseen for additional detection abilities (compare Figure 2).



Figure 1: Instrument BIODIFF with the detector unit on the left side, monochromator shielding (blue) on the right side (Copyright by W. Schürmann, TUM).

The main advantage of this instrument is the possibility to adapt the wavelength to the size of the sample crystal's unit cell while operating with a clean monochromatic beam that keeps the background level low.

2 Typical Applications

The main field of application is the neutron structure analysis of proteins, especially the determination of hydrogen atom positions. Typical questions in this field of interest are:

- Enzymatic mechanism (protonation states of amino acids)
- Ligand binding mediated by hydrogen bonds
- Investigation of the hydration shell of proteins
- H/D-exchange pattern as a monitor of structural stability/flexibility

3 Sample Environment

Besides standard sample environment BIODIFF provides:

- Oxford Cryosystems Cryostream 700 plus with a temperature range of 90 K to 500 K
- Closed cycle cryostat 3.5 - 325 K

4 Technical Data

4.1 Primary beam

- Neutron guide NL1; supermirror $m = 2$
- Monochromator:
 - PG(002) mosaicity: $0.4 - 0.5^\circ$
- Higher order filter:
 - Astrum type velocity selector
 - transmission 87 % for $2.4 \text{ \AA}$

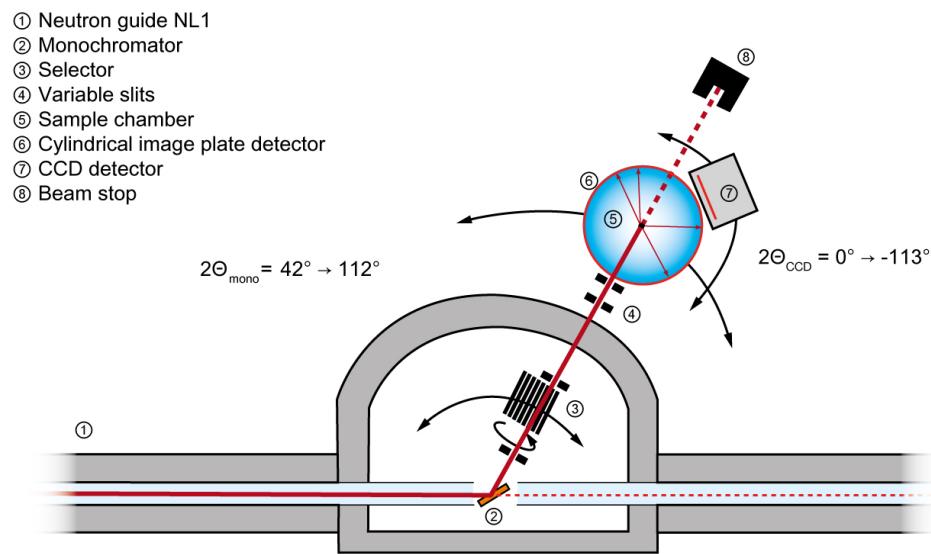


Figure 2: Schematic drawing of BIODIFF.

- Wavelength range:
2.4 – 5.6 Å with selector
2.4 – 6.1 Å without selector
- Collimation by adjustable slits down to $\varnothing = 1$ mm

4.2 Beam properties at the sample position

- Wavelength resolution at sample position: $\Delta\lambda/\lambda = 2.9\%$ at 2.4 Å
- Beam divergence (no slits)
0.8° FWHM horizontal
0.7° FWHM vertical

4.3 Main detector

Neutron image plate (cylindrical)

- BaFBr:Eu²⁺ mixed with Gd₂O₃
- Dimensions:
radius 200 mm
angular range $\pm 152^\circ$ horizontal
 $\pm 48^\circ$ vertical
- Pixel size (quadratic) 125, 250, 500 μm
- Readout time (with erasing): 5 min (for 500 μm pixel size)

4.4 Auxiliary detector

CCD camera with scintillator

- ZnS mixed with ^6LiF
- Dimensions:
 - Active scintillator area (flat) $200 \times 200 \text{ mm}^2$
 - Distance to sample 100 mm
- 2Θ -angle around sample position $0^\circ - 113^\circ$
- CCD chip with 2048×2048 pixels
- Pixel size: $13.5 \times 13.5 \mu\text{m}^2$
- Overall spatial resolution $\approx 300 \times 300 \mu\text{m}^2$ (limited by scintillator thickness)
- Minimum readout time $\approx 1 \text{ sec}$ (full resolution); $< 1 \text{ sec}$ (binning mode)