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A new multipurpose diffractometer PILATUS@SNBL

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The diffraction beamline BM01A at the European Synchrotron Radiation Facility (CRG Swiss–Norwegian beamlines) has been successfully operational for 20 years. Recently, a new multifunctional diffractometer based on the Dectris Pilatus 2M detector has been constructed, commissioned and offered to users. The diffractometer combines a fast and low-noise area detector, which can be tilted and moved horizontally and vertically, together with flexible goniometry for sample positioning and orientation. The diffractometer is controlled by a user-friendly and GUI-based software *Pylatus* which is also used to control various auxiliary equipment. The latter includes several heating and cooling devices, *in situ* cells and complimentary spectroscopic tools.

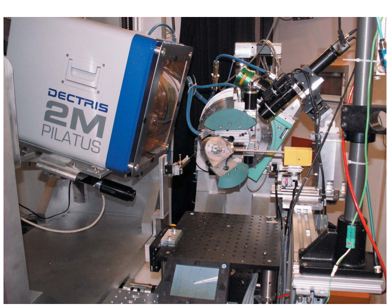
1. Introduction

X-ray diffraction is a well established experimental technique which recently celebrated its 100 year anniversary. Nevertheless, diffractometers are still amongst the most-requested instruments at large synchrotron facilities. The advent of fast and nearly noise-free pixel area detectors, in combination with bright synchrotron light, opens up new opportunities for diffraction. The short time of data collection enables kinetic experiments to be performed, and the low background makes it possible for weak signals such as diffuse scattering and satellite reflections to be detected.

Here we describe a diffractometer based on the Pilatus 2M pixel area detector (Broennimann, 2008). The diffractometer is installed on the BM01A beamline of the European Synchrotron Radiation Facility (CRG Swiss–Norwegian beamlines). This versatile diffractometer has been offered to the broad user community of chemists, physicists and material scientists for the last two years. At the moment, this apparatus is heavily overbooked at the ESRF, in spite of the fact that it is installed on a bending magnet source rather than an insertion device beamline.

2. Beamline overview

The Swiss–Norwegian Beamlines SNBL is a split beamline on a bending magnet source at the ESRF. The branch line BM01A is a multi-purpose diffraction beamline. The optical scheme consists of a conventional arrangement of a pair of collimating and vertically focusing rhodium-coated X-ray mirrors and a sagitally focusing double-crystal Si(111) monochromator. The performance of the optics has been optimized for an energy range of 10–20 keV. The main scientific activity is single-crystal diffraction, but *in situ* powder diffraction, high-pressure (diamond anvil cell) and thin film experiments can also be performed.



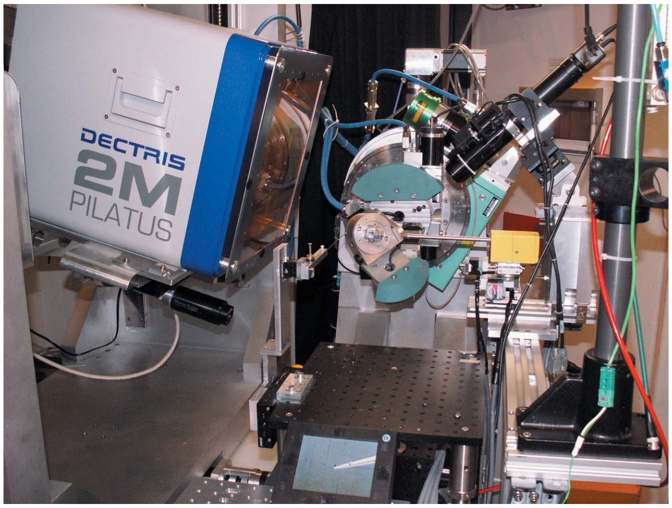


Figure 1
The diffractometer PILATUS@SNBL with the tilted Pilatus 2M detector and the Huber mini-kappa goniometer.

Two separate diffractometers within the same experimental hutch have been used in the past: a multi-circle heavy-duty KM6 diffractometer equipped with an Onyx CCD detector (Oxford Diffraction/Agilent) and a MAR345 image-plate detector used in combination with a single rotation axis for the sample (MarXperts GmbH). This pair of instruments has now been replaced by a single diffractometer based around the Pilatus 2M detector. The combination of large area detector and flexible goniometer provides a very versatile diffraction platform (Fig. 1). The design and construction of this platform was carried out in collaboration with Instrument Design Technology (IDT) Ltd (IDT, 2012).

3. Diffractometer PILATUS@SNBL

3.1. Detector, goniometry and auxiliary equipment

The Pilatus 2M detector (Broennimann, 2008) has been selected as a compromise between active area and weight. The latter parameter is instrumental for the optimization of the detector positioning mechanics. The detector support consists of:

(i) A thick aluminium frame housing the detector arm and a counterweight, the frame can be translated horizontally.

(ii) The detector support holding the detector and the detector's horizontal translation table. The support can be moved vertically inside the frame and also allows the detector to be tilted in the direction of the incoming beam.

The distance from a sample to the detector can be varied between 146 and 700 mm, the detector height can be set between 0 (the beam hits nearly the center of the detector) and 500 mm, while the detector tilt is currently limited to 35°. The maximal angle covered by the diffractometer $2\Theta = 78°$. The angular resolution depends on the beam focusing, sample-to-detector distance and sample and beam size. In a typical powder experiment with a 0.1 mm-thick glass capillary, 0.7 Å X-ray wavelength and the shortest sample-to-detector distance (146 mm), the diffractometer offers a resolution of $\Delta d/d \simeq 10^{-3}$; $\Delta d/d \simeq 10^{-4}$ can be reached at the maximal sample-to-detector distance (700 mm) with beam focused on the detector.

The kappa goniometer is fixed onto a support stand which allows vertical and horizontal translation of the goniometer normal to the beam over a 100 mm range. The support has a slow but precise encoded rotary table designed for thin film and similar experiments (ω' table). Another rotary table (ω) can be installed for conventional data collections, with maximum speed of 10° s^{-1}. Alternatively, a mini-kappa setup [ω, φ and κ rotary tables supplied by Huber (Huber, 2012)] can be installed to increase completeness and redundancy of data collections.

The detector support and the sample positioning unit are mounted on a substantial marble table that is, in turn, motorized for precise positioning in horizontal and vertical directions normal to the beam. The setup is complemented by a Huber slit system (Huber, 2012) and an adjustable collimator or focusing device support stand that finalize incoming beam conditioning.

A small CCD camera (Photonic Science Ltd) is fixed underneath the Pilatus 2M detector and serves as an X-ray eye to control beam focusing and alignment. Together with an optical microscope, the X-ray eye is used for alignment of a broad range of samples such as single crystals, capillaries, thin films, diamond anvil pressure cells, *etc*.

A growing list of auxiliary equipment is available for users. Currently, it contains a modified-by-ESRF Helijet open-flow helium blower (temperature range 5–35 K) (van der Linden *et al.*, 2013), a CryoVac cryostat with windows modified for diffraction experiment (4.5–300 K), an Oxford Cryostream 700+ nitrogen blower (80–500 K) and a locally developed nitrogen hot blower (300–900 K). High-pressure diamond anvil cells are frequently used covering a range 0.1–50 GPa. The low-pressure range 0–0.15 GPa is covered by a gas loading system (Jensen *et al.*, 2010). Any user-designed cell which fits the geometrical restrictions can be mounted [see an electrochemical cell (Maity *et al.*, 2015), gas cells (Jensen *et al.*, 2010) and an electric field cell (Vergentev *et al.*, 2015) as examples].

3.2. Control software and *Pylatus*

The control of the optics and motors of the diffractometer is based on the standard ESRF controllers IcePAPs and SPEC environment (Janvier *et al.*, 2013). The detector is operated by the original Dectris software *Camserver* supporting compact mini-CBF file format (Dectris, 2013).

For user-friendly and easy control of the diffractometer a special program *Pylatus* (Fig. 2) has been developed to:

(i) Synchronized triggering of the detector and the motion of all motors required for a given experiment.

(ii) Manipulate the acquired images and provide information about the experiment in the file headers.

(iii) Control auxiliary equipment, such as heat blowers, Oxford Cryostream coolers, a Helijet blower or a cryostat operated by the Lakeshore temperature controller.

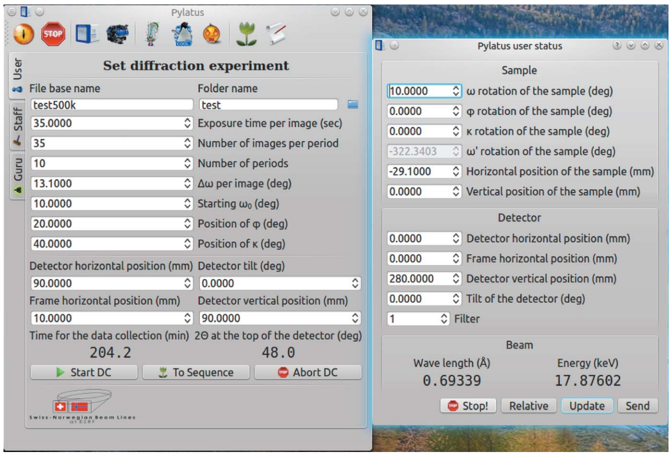


Figure 2
Screenshot of the *Pylatus* main windows. The left window allows the user to set up the diffraction experiment. The right window shows the current status of motors involved in a typical experiment.

(iv) Offer a range of experimental configurations for powder, single-crystal and thin-film diffraction experiments.

(v) Make possible a sequence of data collections at different detector or sample positions as a function of external parameters (temperature, pressure, *etc*).

(vi) Synchronize triggering of an external equipment such as an *in situ* gas system, an electric field cell, or Raman and UV–VIS spectrometers together with a data collection.

(vii) Perform various combinations of the above items.

Together with the programmable ESRF MUSST card (Bouchenoire *et al.*, 2010), *Pylatus* can be used for data collection synchronized with periodical perturbation of a sample, *e.g.* in a stroboscopic mode or for the modulation-enhanced diffraction (Chernyshov *et al.*, 2011).

Pylatus is written in the Python programming language (van Rossum, 1995) using open source components such as GUI toolkit Qt with its binding to Python PyQt4 and SpecClient library developed at the ESRF. The mercurial repository for *Pylatus* is freely available at http://hg.3lp.cx/pylatus.

4. Data manipulation

4.1. Big data for small molecular crystallography

A huge amount of data can be harvested in a relatively short time with the Pilatus 2M detector. For example, a full-sphere single-crystal Bragg data collection with 0.1° ω-slicing performed at the maximum speed of 10° s^{-1} (assuming a well scattering crystal) would take 36 s and produce 3600 frames (9 GB of compressed CBF data; this corresponds to 36 GB after decompression in, for example, the ESRF data format EDF). Special care has, therefore, to be taken of the data transfer and storage. The beamline, therefore, provides a separate data storage server with 26 TB of space connected to the control PC by an optical link. All the new data are copied into the data storage and kept for at least six months.

For many experiments it is vital to be able to analyze the diffraction data as soon as they are measured in order to adapt and optimize the data collection strategy. Apart from the measurement computer and the storage server, the users have another PC connected by a fast 10 Gb link to process their data without interfering with the data collection.

4.2. *SNBL ToolBox*

In order to simplify the further use of commercial or freely available software for data analysis and based on user requests we offer to users an in-house-developed package *SNBL ToolBox* (Fig. 3). The package currently contains a number of utilities:

(i) *Crysis* reads headers of a data collection and produces the files needed for the Rigaku/Oxford Diffraction *Crysalis* software (Rigaku, 2015); furthermore, the data are processed smoothly as if they were native for *Crysalis*. Complex multi-run scans with kappa geometry are fully supported.

(ii) *Slovio* converts Pilatus CBF frames into a standard Esperanto format processed by *Crysalis* natively. It should be noted that the format is unpacked and it needs at least four times more disc space than the Pilatus mini-CBF.

(iii) *Converter* transforms frames from Pilatus into EDF (ESRF data format) accepted by *Fit2d* (Hammersley *et al.*, 1996); it can also bin data frames within a run or between many runs; the binned data are stored either as EDF or CBF.

(iv) *σ-scaler* normalizes to monitor or background one-dimensional data previously integrated by *Fit2d* and calculates error bars based on the pixel statistics and the number of pixels per powder ring.

(v) *HeadEx* extracts information from the frame headers (such as sample temperature, monitor counts, *etc*).

(vi) *Sleuth* allows a user to perform a fast inspection of diffracting intensity in a selected volume of reciprocal space in a sequence of data collections, *e.g.* for on-line monitoring of the scattering intensity as a function of temperature, time, pressure, *etc*.

(vii) *Hroerekr* inspects whether a user needs to perform ψ-scans for a given single-crystal data collection.

Figure 3
Screenshot of the *SNBL ToolBox* main window.

4.3. Powder data processing with *Bubble*

Although the diffractometer has predominantly been developed for single-crystal diffraction, it is actively used for *in situ* powder diffraction experiments. Therefore, we have provided a processing tool for powder data (*Bubble*) that performs azimuthal integration of raw images using the pyFAI library (Kieffer & Karkoulis, 2013).

The software consists of two parts: client and server. The server is a daemon, it performs the integration and can be used as a standalone program receiving commands in JSON format through a standard network socket connection (for example, directly from a beamline software). The server runs in a twisted event loop and integrates images in separate threads using the twisted thread pool.

The client is a GUI program based on the Qt4 framework and its Python binding PyQt4 (Fig. 4). A user interacts with the main window specifying integration parameters such as a folder where the raw images are stored, calibration [so-called pyFAI poni-file (Kieffer & Karkoulis, 2013)], background, mask, flat field and geometrical distortion corrections (the last two are for CCD detectors), and other corrections if needed. When a user starts a new integration process, the client sends all the parameters to the server, the server reads images from the specified folder, applies all the provided corrections and transmits the final data array to the pyFAI integration procedure. The client polls the server receiving current results of the processing (such as number of integrated files, and one-dimensional and two-dimensional graphs of the last integrated files). When all the data have been processed, the server waits for new images. Thus, *Bubble* can be used as an on-line integrator, which is very important for fast *in situ* experiments when a user may need to quickly optimize experimental strategies.

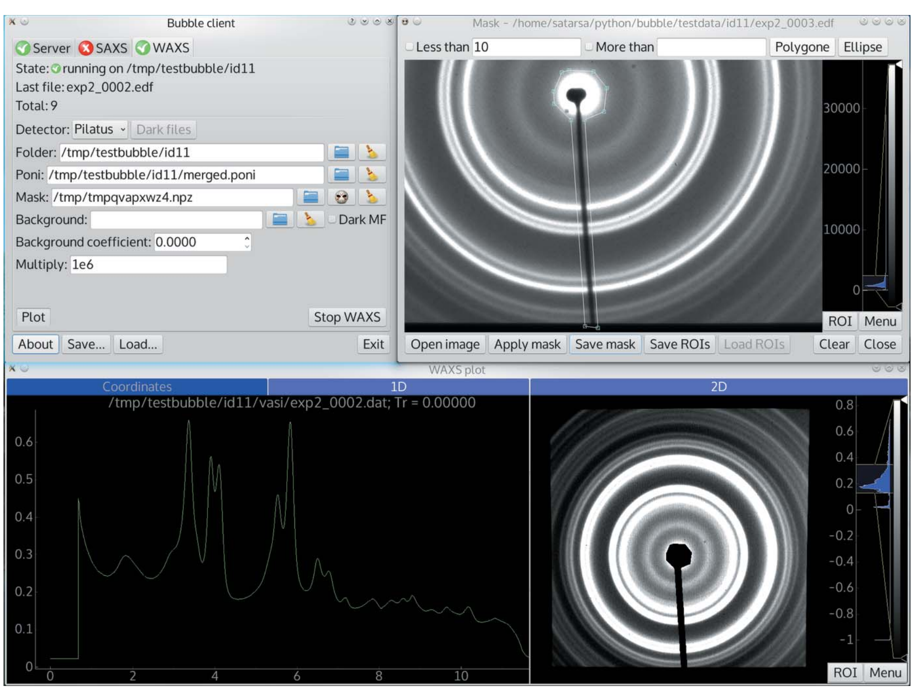


Figure 4
Screenshot of the *Bubble* client. The top left window allows users to set integration parameters. The top right window is used to produce a mask. The bottom window shows integration results.

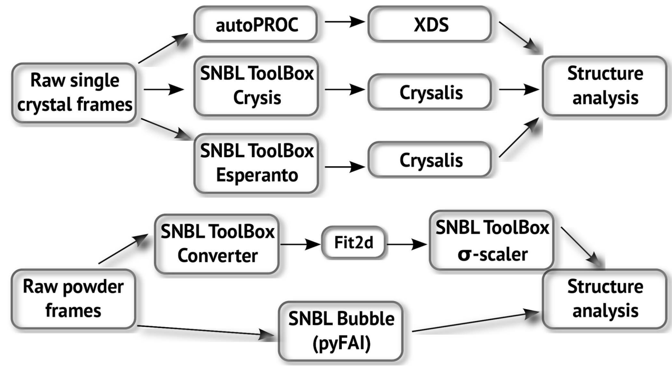


Figure 5
Typical scheme of data flow with the PILATUS@SNBL diffractometer.

4.4. Data flow

Data flow variants typical for single-crystal and powder diffraction data are shown in Fig. 5. Raw single-crystal data can be sent either *via autoProc* to *XDS* (Kabsch, 2010) or *via SNBL ToolBox* to *Crysalis* software (Rigaku, 2015).

Raw powder data can be either converted by the *SNBL ToolBox* and processed by *Fit2D*, or directly integrated to powder pattern by the PyFAI-based tool *Bubble*.

SNBL ToolBox and *Bubble* are open source programs and their mercurial repositories are freely available at http://hg.3lp.cx/snbltb and http://hg.3lp.cx/bubble, respectively.

5. Summary

A new multipurpose diffractometer has recently been built at the bending magnet beamline BM01A (SNBL at ESRF). The diffractometer combines the flexible kappa-goniometry with the fast hybrid-pixel detector Pilatus 2M that can be translated in two directions and rotated. Such a combination makes possible a large variety of diffraction experiments such as single-crystal thin-film powder diffraction with an optimized angular resolution and coverage of reciprocal space. It also makes possible various *in situ* experiments where the change of experimental conditions can be synchronized with diffraction data acquisition. All the options listed above are easily available *via* a user-friendly software *Pylatus*, which also provides a tool for complex sequences of experimental scenarios and combines an easy-to-use graphical interface with an editor for Python-based macros. Some examples of published work carried out with the new diffractometer can be found in a recent review article (Chernyshov, 2015).

The control software interacts *via* the MUSST card developed by the ESRF with a variety of external equipment. It allows the user to manipulate gas pressure in a gas rig (Jensen *et al.*, 2010) or electric field strength and polarity in the electric cell (Vergentev *et al.*, 2015) or perform an external control of any user supplied equipment. Such a scheme supports triggering of external devices in a mode synchronized or delayed with respect to the data acquisition by the Pilatus 2M detector *via* the *Pylatus* macros. As a result, complex combined experiments, *e.g.* diffraction + Raman spectroscopy + UV–VIS as a function of temperature or pressure, become easily automated.

Users are also provided with a number of tools which are used to process and analyze acquired data. Together with the easy-to-use control software and short data acquisition time, these tools (*SNBL ToolBox* and *Bubble*) enable a fast inspection of the results and add more flexibility to optimize experimental strategies.

The PILATUS@SNBL diffraction platform is now one of the most requested material science instruments at the ESRF. The technical and programming solutions described above could also be useful for other diffractometers based on Dectris Pilatus detectors. All the source code of the developed software is freely available *via* the links cited in the text.

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