

The year 2020 of remote operation at P11

**Current and future trends in Macromolecular Crystallography experiments:
Focus on Automation, high data rate analysis and User Interfaces**

18.03.21

Guillaume Pompidor



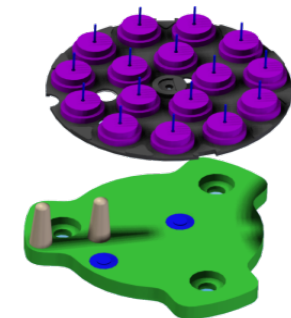
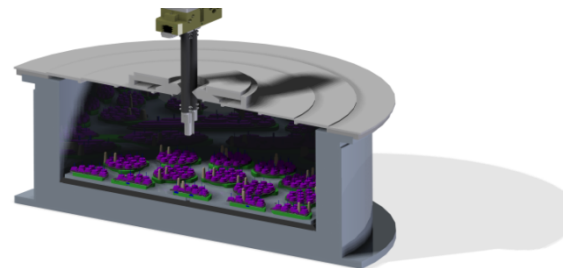
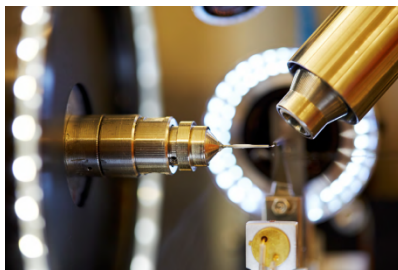
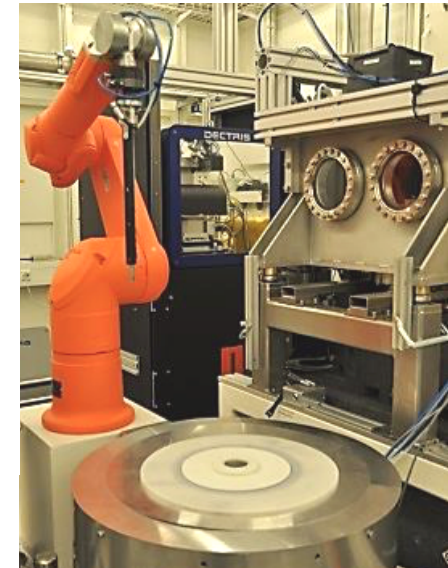
Outline

- **The P11 beamline at PETRA III**
- **User Interface**
- **Remote data collection**
- **The 2020 year**

P11 – High Throughput MX beamline at PETRA III

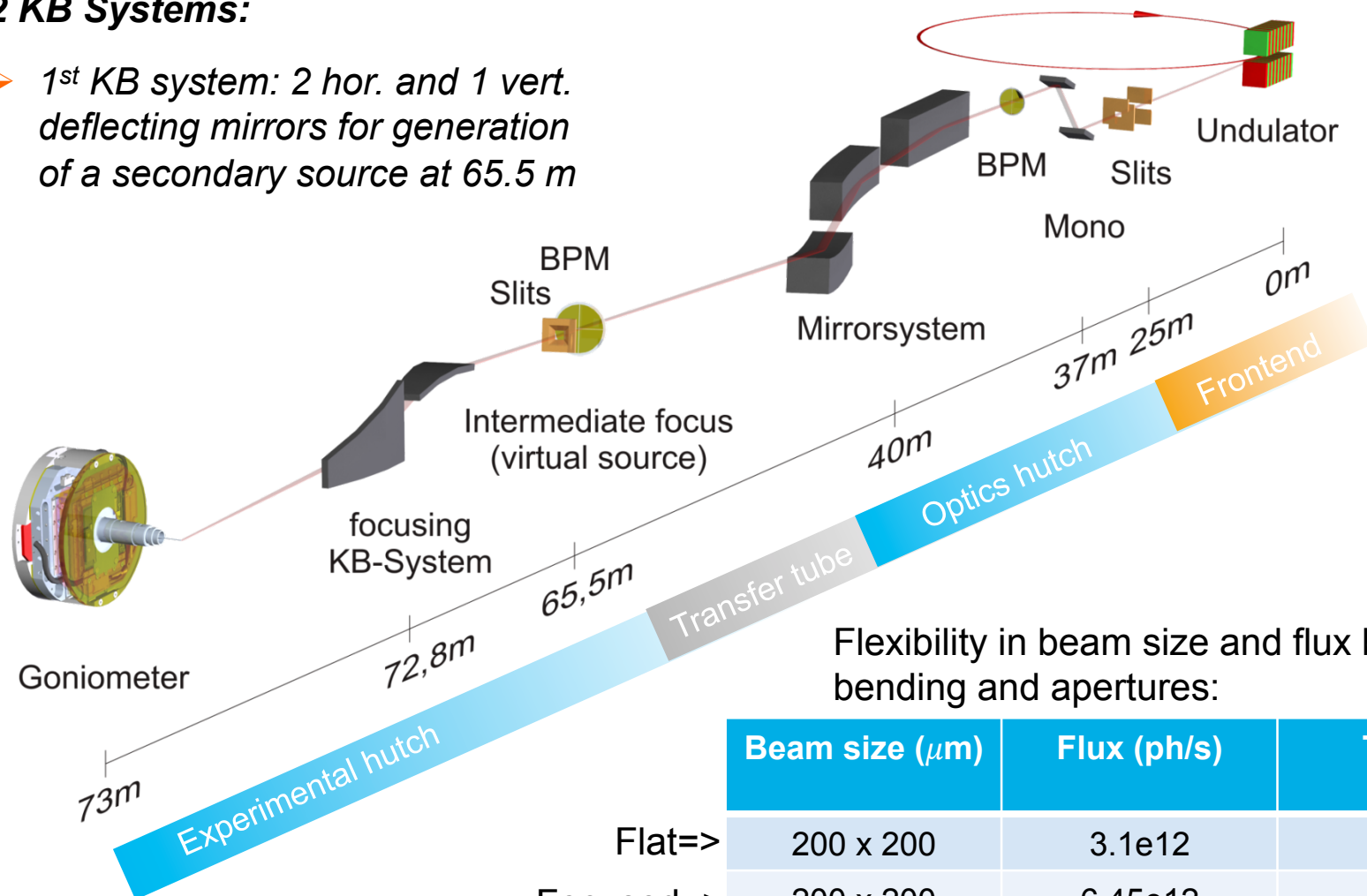
1) Crystallography experiment

- In user operation since 2013
- Broad energy range: 5.5 - 28 keV
- High-speed sample changer with capacity (23 unipcuks = 364 samples)
- Various focusing options (beamsize from 4 x 9 to 200 x 200 μm)
- Pilatus 6M, Eiger2 16M (permanent)
- XRF for experimental phasing (SAD/MAD) and element analysis
- Serial crystallography



2 KB Systems:

- 1st KB system: 2 hor. and 1 vert. deflecting mirrors for generation of a secondary source at 65.5 m

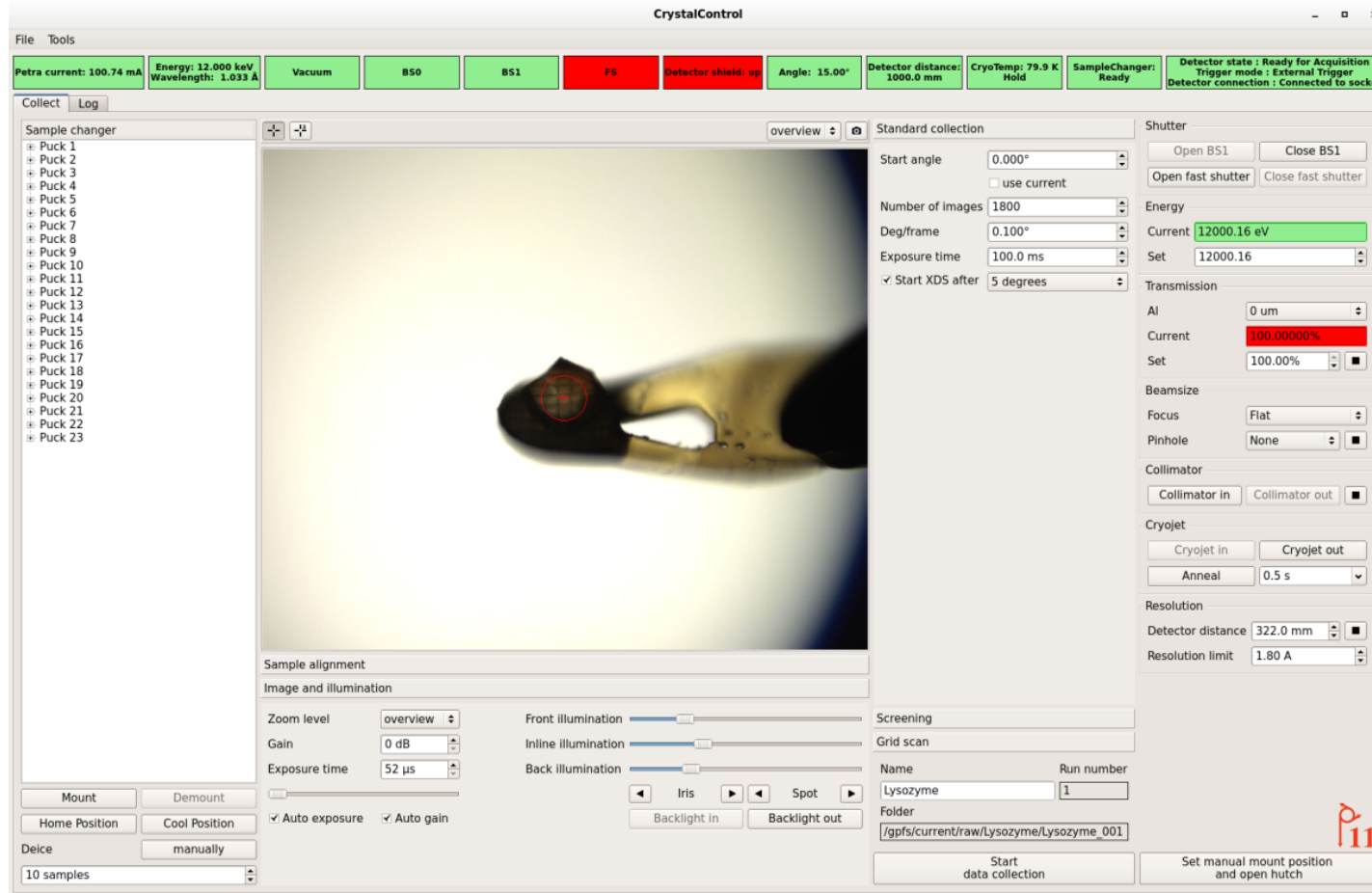


Flexibility in beam size and flux by mirror bending and apertures:

	Beam size (μm)	Flux (ph/s)	T_{xtal} (s)
Flat=>	200 x 200	3.1e12	800
Focused=>	200 x 200	6,45e12	360
	100 x 100	1e13	40
	50 x 50	1e13	10
	20 x 20	1e13	1,6
	4 x 9	1e13	0,17

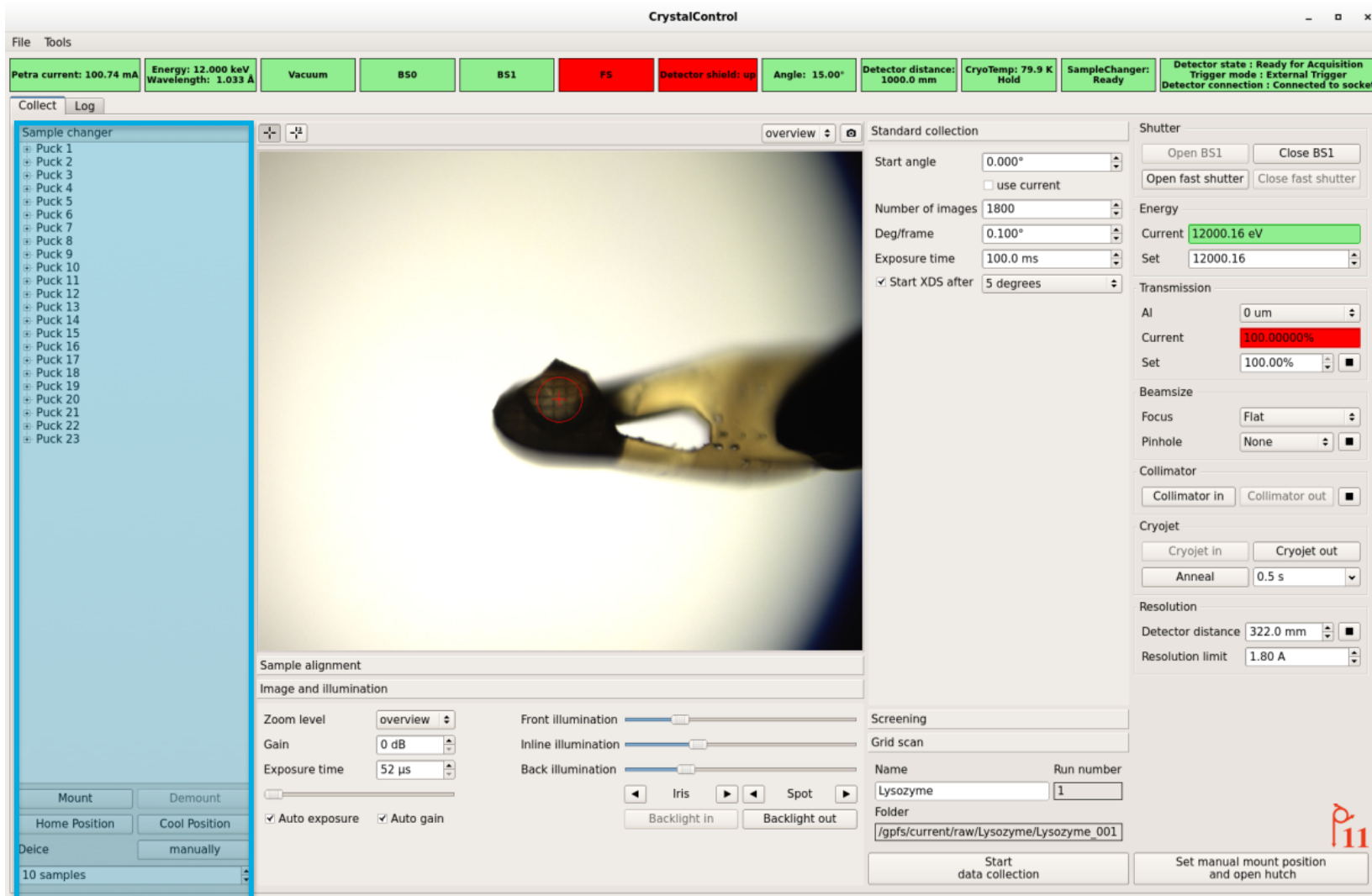
CrystalControl

- Inhouse-developed Python-based GUI for standard MX applications including SAD/MAD phasing



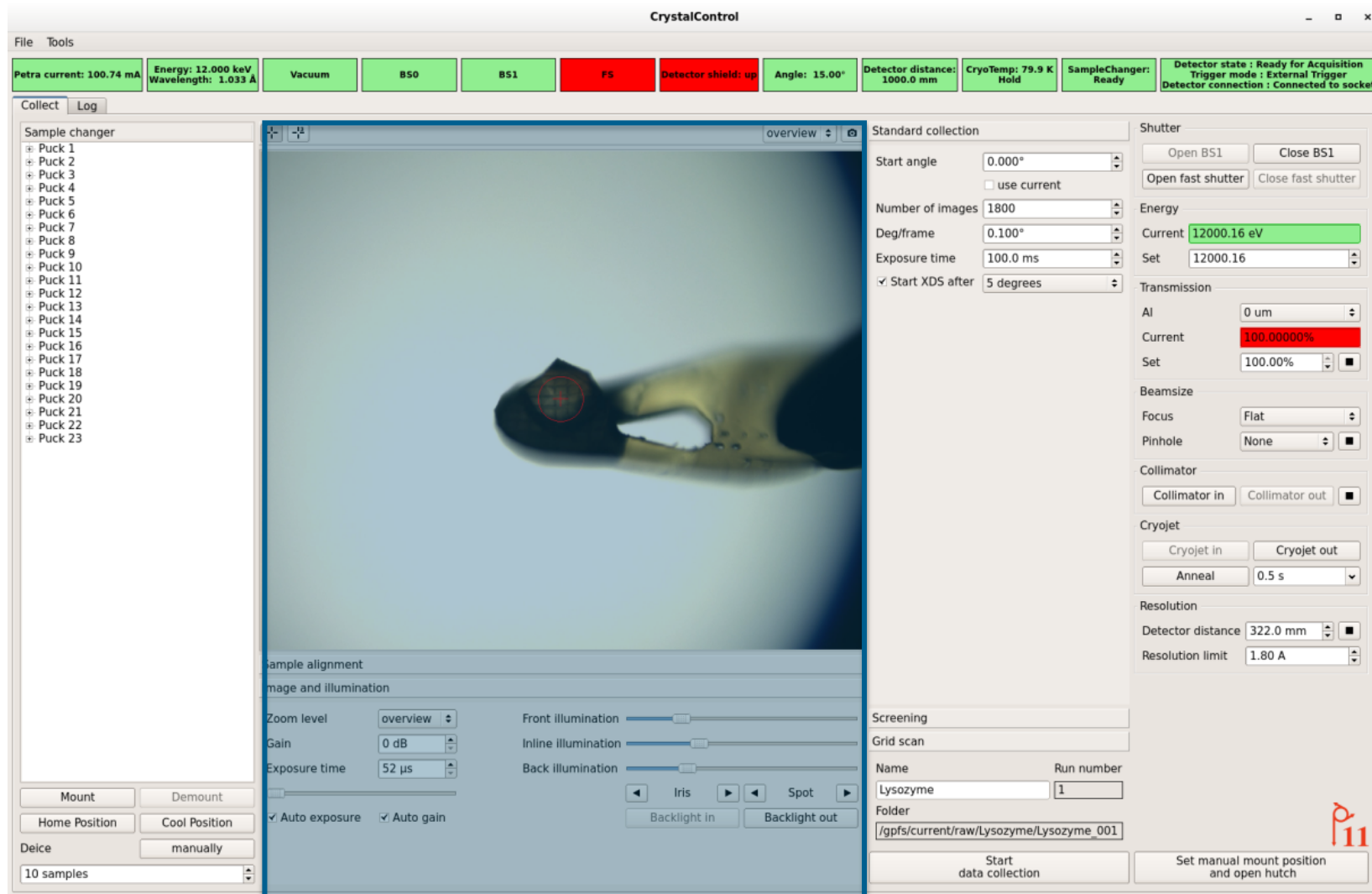
- Expert mode for beamline support (apertures, rotation axis, beam position...)

CrystalControl



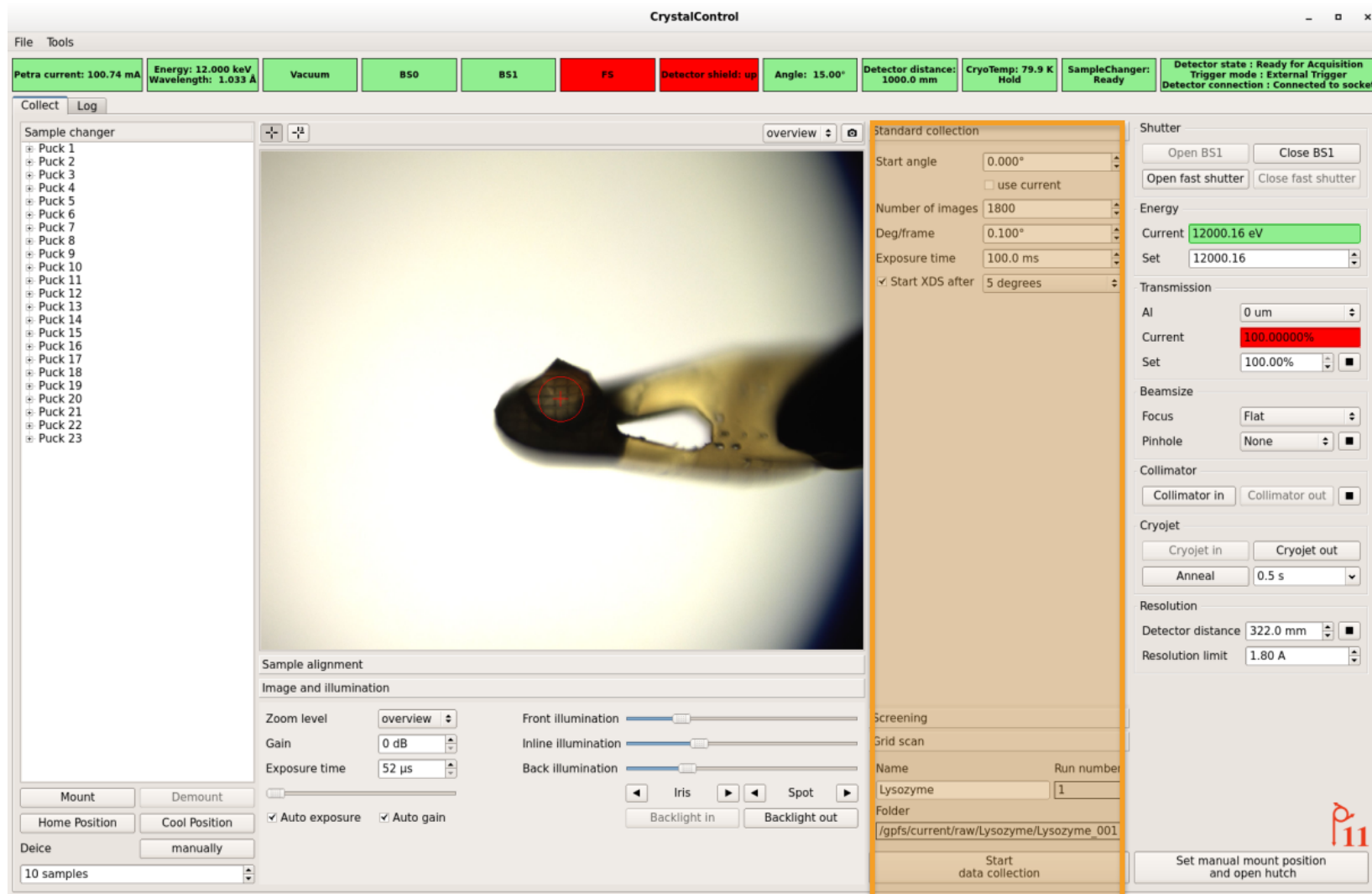
Sample changer : Puck and sample selection

CrystalControl



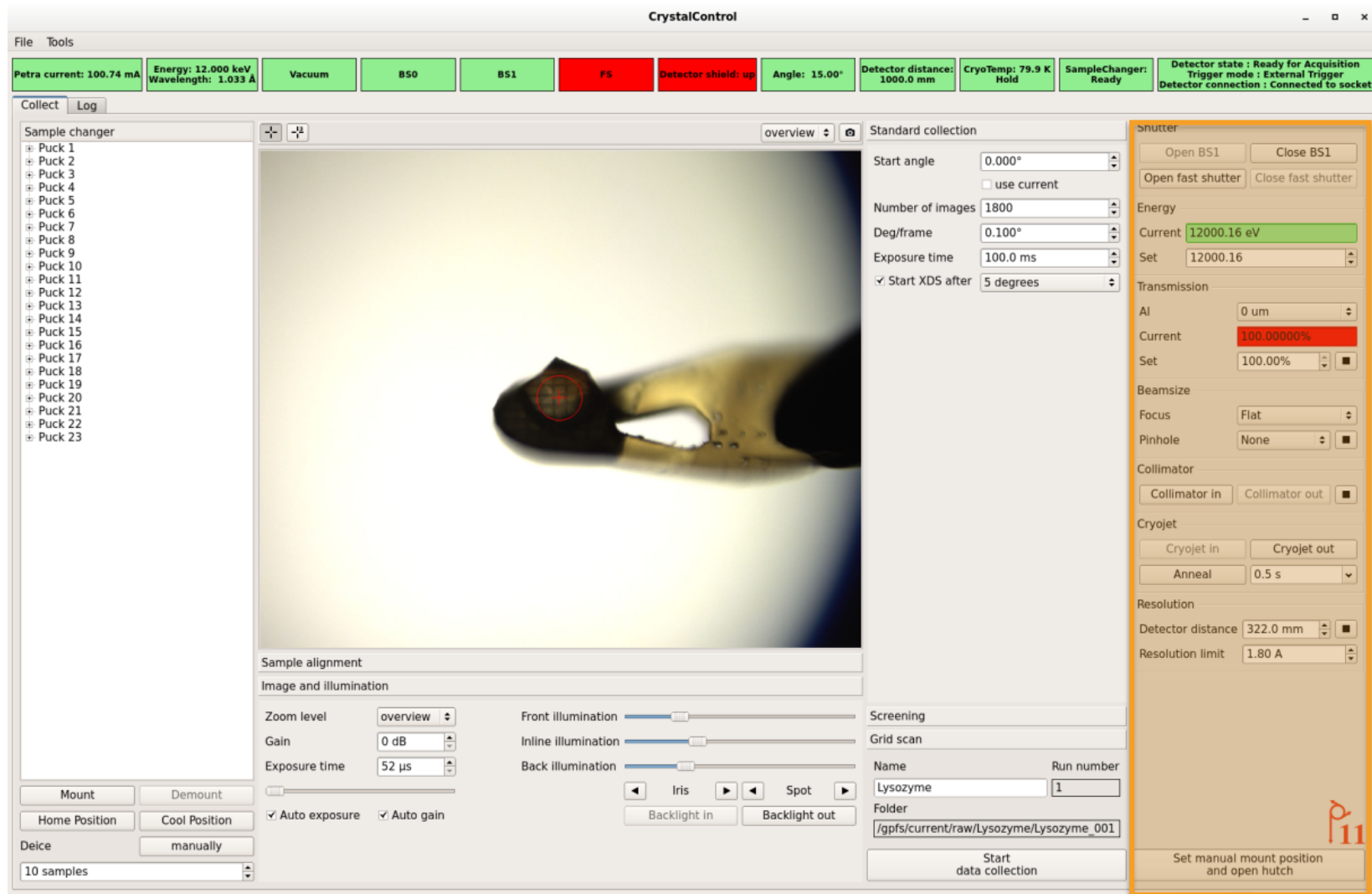
Crystal View: zoom selection and 3 clicks centering

CrystalControl



Data collection: Number of frames, exposure time, angular step

CrystalControl



Data collection: Energy, Focus selection, Pinholes, transmission, resolution (det. distance)

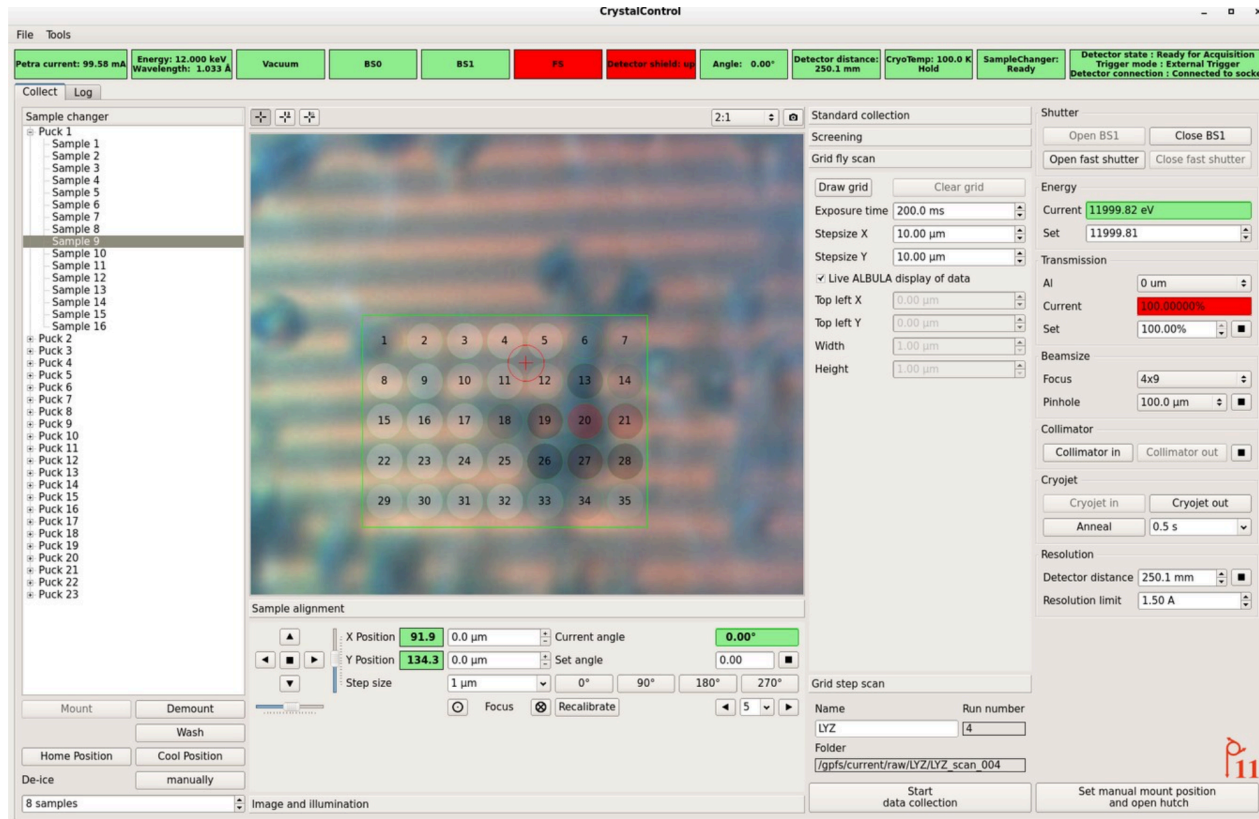
CrystalControl

- SAD/MAD phasing



CrystalControl

- ISMO scantool: GUI for fixed-target SSX, data collection with (rotation wedges) and without ϕ rotation (still images)



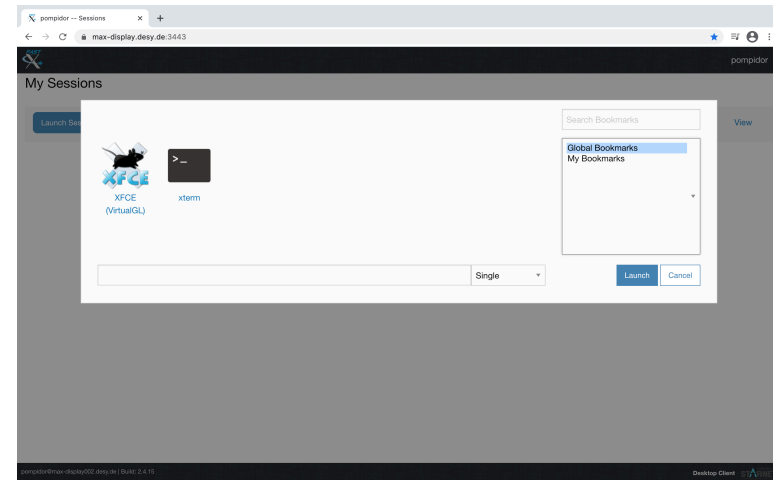
- Heat map overlayed on the sample view

Implementation of remote data collection

- Beamline control by one user only
- Access to needed functionalities only
- Monitoring of user actions
- Possibility to take back the control at any time for the local contacts
- No need of special hardware, software or OS dependencies for the user community
- Authentication against personalized account
- Remote access dependent on valid safety training

Technical Implementation (Spring 2020)

- **FastX**
- Commercial software for Linux remote desktop
- X server from a web browser
- Similar to Maxwell Cluster (computing at DESY)
- Sessions between several users with only one controlling
- Running on a dedicated host



Remote identification via user database: Door

User view

Staff view

ACCELERATORS | PHOTON SCIENCE | PARTICLE PHYSICS
Deutsches Elektronen-Synchrotron
A Research Centre of the Helmholtz Association

DESY PHOTON SCIENCE »

DOOR HOME | ROLE SELECTION | CONTACT

DOOR

DESY Online Office for Research with Photons

BEAMLINE SCIENTIST P11

- Applications P11
- Schedule of P11
- Declarations P11
- Manage P11 setups
- Log off

MANAGE REMOTE ACCESS

GENERAL INFORMATION

Project Number: I-20191071 EC
Beamtime/Application ID: 11009066
Project Leader: Dr. Dolittle
PI/Applicant: Dr. Don Carlo
E-Mail: d.carlo@downstream.de
Phone: +49 30-3289 2381
Beamline: P11
Setup/Instrument: Crystallography (1-axis goniostat)
Local contact: Dr. Johanna Hakanpaee
Startdate: 12-May-2020
Enddate: 12-May-2020

REGISTERED USERS

Dr. Dolittle ☐ on-site ☒ remote access
Mr. Mouse ☐ on-site ☒ remote access

REMOTE ACCESS SESSION KEY

eyJpZC16InQzNGIyNDI0ZDQzOTQ3NDQ5NjA3NzBkZDI3OGQxZjkiIiwia2V5Ijo1MDh0ZC0yYTNhLTN3ZGEtNjM5ZSIsInRlcm1zIjpbImdyYW50b3R1X000=

(*) these fields are mandatory.

CHECK LINK: **CONNECT**

MAIN SAVE REVOKE ACCESS LIST

HELMHOLTZ RESEARCH FOR GRAND CHALLENGES

Contact | DESY Data Privacy Policy | DOOR Data Privacy Policy | Imprint
© 2018 Deutsches Elektronen-Synchrotron DESY

DOOR user name: yefanov
Umbrella user name: DOOR account not linked with an Umbrella account

NEXT SCHEDULED SHIFTS

Proposal	Beamline	Start	End	Proof of registration
I-20191422	P11	09-May-2020 09:00	10-May-2020 09:00	not registered as participant
I-20170004	P11	20-Apr-2020 09:00	23-Apr-2020 09:00	pdf
I-20191422	P11	28-Jun-2020 09:00	29-Jun-2020 09:00	not registered as participant
P-20010197	P11	31-Mar-2020 09:00	07-Apr-2020 10:00	pdf
I-20191422	P11	31-May-2020 09:00	01-Jun-2020 09:00	not registered as participant

REMOTE SESSIONS

Proposal	Beamline	Start	End	Remote Session
P-20010197	P11	31-Mar-2020 09:00	07-Apr-2020 10:00	start remote session

PROPOSALS - BEAMTIME APPLICATIONS - EXPERIMENT SPECIFICATIONS

Submit a new proposal
New proposals may be submitted here. If you leave DOOR before completing the submission, your entries are saved and may be edited and completed later (see below).

Edit/Delete a partially completed proposal
If you have not completed the submission procedure (see above), you may edit/delete/delete your entries here. Once a proposal is submitted, it cannot be modified or deleted by yourself. In this case you must contact the beamline manager.

Follow-up applications for beamtime for a PETRA III long-term project
If you have a valid PETRA III long-term proposal, you can apply for beamtime here. Please note: Applications may be submitted only during calls for PETRA III proposals.

Confirm FLASH application for beamtime
If your FLASH proposal has been accepted, please confirm your session for beamtime here.

Users

- ☐ Sofiane
- ☒ yefanov
- ☐ p11user

- Selection of 'remote', 'onsite' or 'data-access' during user registration
- Automated e-mail notification once the remote is open
- All participants can log-in at the same time from different locations, one has control over the experiment, can be swapped independently among the users
- Detailed instructions on our public webpage for shipment, remote session and data processing
- Support via zoom
- Successful operation also with users who were never onsite; connection stable and shipments running e.g. to China and India

Remote data collection

Mozilla Firefox (Private Browsing)

Live view - AXIS P5534 | x | AXIS P5534 PTZ Dom | x | Network cameras | A x | PTZ cameras | Axis C x | AXIS P54 PTZ Networ | x | AXIS P5415-E at Duck | x | DESY Photon Science | x | p11remote.desy.de/s | x

https://p11remote.desy.de/share#eyJpZCI6ImQzMGlYNDI3ZDQ3NDQ5NjA3NzBkZDI3OGQxZjJkxliwI2V5Ij0= 67% P5534

Most Visited ebiss Lagerkatalog Kantine Cafe CFEL PETRA - Logbuch PETRA-3 Infoscreen

File Tools

Current: 100.83 mA Energy: 12.000 keV Wavelength: 1.033 Å Vacuum B50 B51 PS Detector shield: down Angle: 270.00° Detector distance: 198.9 mm CryoTemp: 99.9 K Hold SampleChanger: Ready Detector state: Ready for Acquisition Trigger mode: External Trigger Detector connection: Connected to socket

Collect Log

Sample changer

- Puck 1
- Puck 2
- Puck 3
- Puck 4
- Puck 5
- Puck 6
- Puck 7
- Puck 8
- Puck 9
- Puck 10
- Puck 11
- Puck 12
- Puck 13
- Puck 14
- Puck 15
- Puck 16
- Puck 17
- Puck 18
- Puck 19
- Puck 20
- Puck 21
- Puck 22
- Puck 23

Sample 1

Sample 2

Sample 3

Sample 4

Sample 5

Sample 6

Sample 7

Sample 8

Sample 9

Sample 10

Sample 11

Sample 12

Sample 13

Sample 14

Sample 15

Sample 16

Sample alignment

X Position: 94.2 0.0 µm

Y Position: -88.7 0.0 µm

Step size: 1 µm

Current angle: 270.00°

Set angle: 0.00

Focus

Recalibrate

Grid scan

Name: 20200512_1433s_p65_LvD27

Run number: 1

Folder: /rgf/rgf/current/raw/20200512_1433s_p65_LvD27/20200512_1433s_p65_LvD27_001

Start data collection

DataCollection

Time remaining: 00:00:54

Speed: 5.00 °/s

Number of images: 1080

g/frame: 0.250°

Collection time: 00:00:54

Exposure time: 50.0 ms

Start XDS after: 5 degrees

Shutter

Open B51

Close B51

Open fast shutter

Close fast shutter

Energy: 12000.22 eV

Current: 12000.22 eV

Set: 12000.22

Transmittance

AI: 0.00

Current: 100.83 mA

Set: 100.83

Beamstop

Focus: Flat

Pinhole: 100.0 µm

Collimator

Collimator in

Collimator out

Cryojet

Cryojet in

Cryojet out

Anneal: 0.5 s

Resolution

Detector distance: 198.9 mm

Resolution limit: 1.30 Å

DETAIS

Keys Tools

View

Halogram

Intensity: 0.00

Background: 0.8217

Foreground: 114.4015

Pixels: 5 BG: 0

BG: 4 # Pixels: 589953

Pixels: 2 FG: 1334

High Dynamic Range

Grey Scale

Heat

Invert Color

Plot 3D

Show Resolution Rings

PETRA Energy: 6.084 GeV Lifetime: 0.00 h Current: 100.8 mA

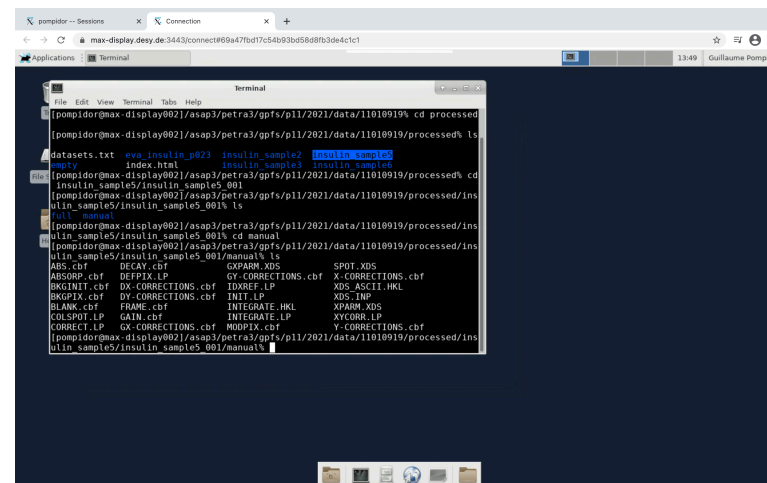
Beam Current

PU	Gap	PU	Gap
01a	13.06	21a	09.97
01b	12.00	21b	07.29
02	10.45	22	10.30
03	10.00	23	13.00
04	12.75	24	12.68
05	10.29	62	216.99
06	10.64	64	24.06
07	07.00	65	23.67
08	13.00		
09	10.02	PW	Gap
10	13.44	61	24
11	12.59		
12	21.10		
13	11.06		

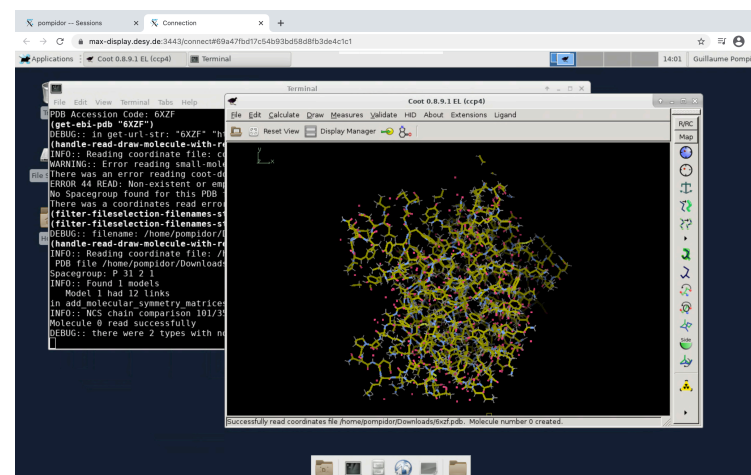
16:39

Remote data processing on Maxwell Cluster

- Computing cluster for DESY
- Access via scientific account
- Backup (or via Gamma portal)
- Data processing
 - Albula, adxv
 - XDS, imosflm
 - ccp4, Phenix
 - Coot, Chimera

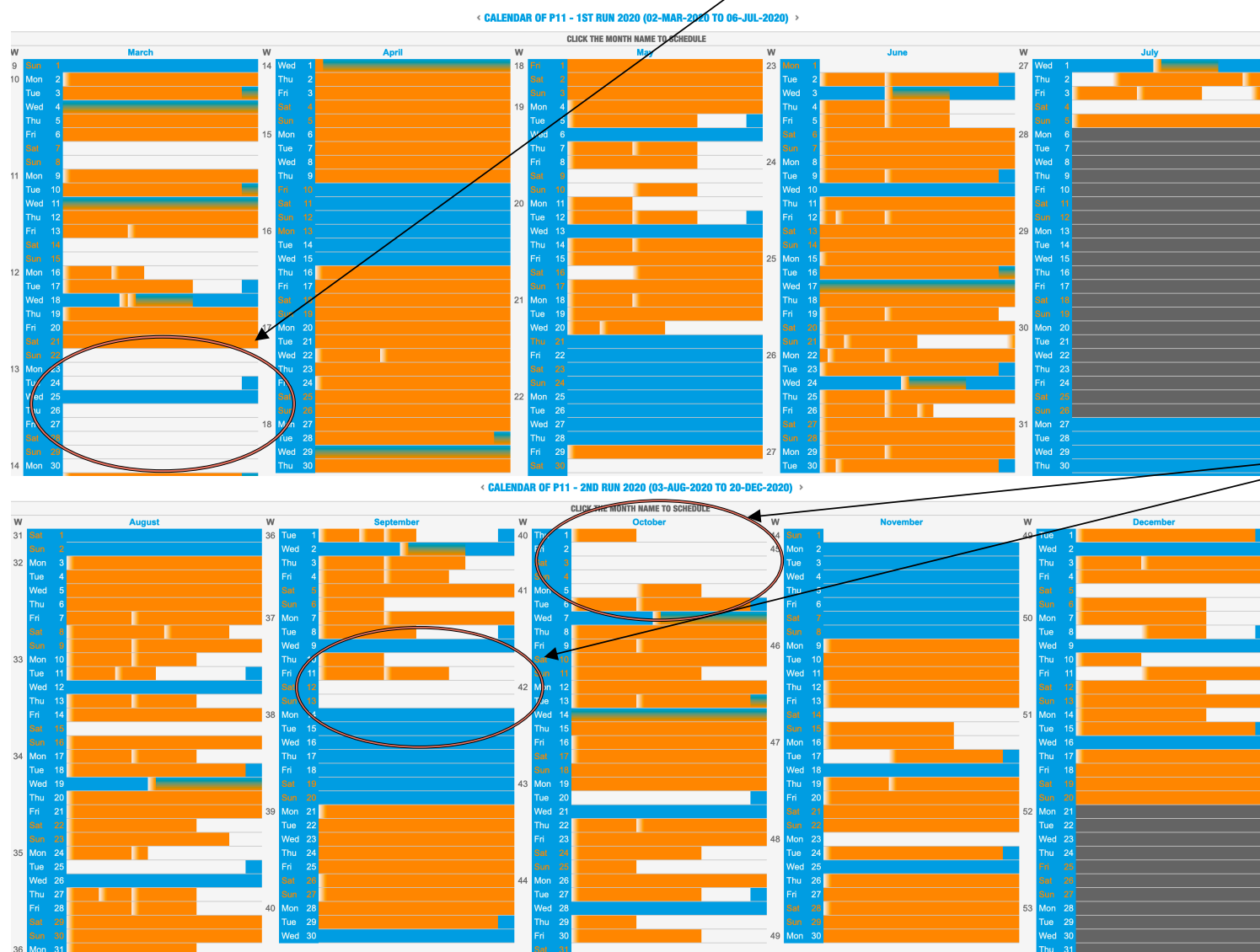


```
pompidor -- Sessions x Connection x +
max-display.desy.de:3443[connect#09a47bd77c54b83b588bf3de4c1c1]
Applications Terminal
File Edit View Terminal Tabs Help
[pompidor@max-display002/asap3/petra3/gpfs/pl1/2021/data/11010919% cd processed
ls
ls
index.html
insulin_sample5
insulin_sample5_001
insulin_sample5/insulin_sample5_001
insulin_sample5/insulin_sample5_001% ls
ls
manual
insulin_sample5/insulin_sample5_001% cd manual
insulin_sample5/insulin_sample5_001% ls
ABS.cbf  DECAT.cbf  GXPARM.XDS  SPOT.XDS
ABSOMP.cbf  DEFTFX.LP  OX-CORRECTIONS.cbf  X-CORRECTIONS.cbf
BKGINIT.cbf  DX-CORRECTIONS.cbf  IDXREF.LP  XDS_ASCII.HKL
BKGPIX.cbf  DX-CORRECTIONS.cbf  INIT.LP  XDS.INP
BLANK.cbf  FRAME.cbf  INTEGRATE.HKL  XPARM.XDS
COLSPOT.LP  GAIN.cbf  INTEGRATE.LP  XYCORR.LP
CORRECT.LP  OX-CORRECTIONS.cbf  MODPIX.cbf  Y-CORRECTIONS.cbf
[pompidor@max-display002/asap3/petra3/gpfs/pl1/2021/data/11010919/processed/insulin_sample5/insulin_sample5_001/manual%]
```



2020 at P11

Machine down due to the lock-down!




Pandemic vs user operations 2020

Suspended user operations mid-March - April 2020: only Fast-track open

Masks & distancing: May-October 2020

Enhanced remote at P11 since October 2020
Covid-tests since November 2020



**IMPORTANT INFORMATION ON COVID-19
FOR WORKERS AND USERS AT P11 BEAMLINE**

Dear User/DESY worker,

Due to the current COVID-19 restrictions, the following **safety procedures are mandatory for experiments and technical work** at P11 Beamline/DESY Campus:


1. The **maximum** number of **participants coming at DESY campus** per experiment is **3 persons**.
2. The **maximum** number of **participants on-site at the beamline** (control hut and experimental hut) is **2 persons**, preferably **only 1 if possible**.
3. Two persons in a **ventilated room** can work **without masks** only if they keep a **steady distance of 2 meters**.
4. If 2 persons must work in a **non-ventilated room for less than 15 minutes** at a distance **below 2 meters**, a **simple mouth/nose protection** must be worn (so-called surgical masks). Self-sewn masks are also permitted.
5. If 2 persons must work in a **non-ventilated room for more than 15 minutes** at a distance **below 2 meters**, **FFP3 masks must be worn**, and provisionally also FFP2 masks.
6. **Gloves** should be always used if possible.
7. Please **disinfect your hands** and any shared equipment (keyboards etc) after use.

In case of doubts, please ask a P11 Team member.

Thank you for your collaboration,

Your P11 Team

Deutsches Elektronen-Synchrotron DESY
Ein Forschungszentrum der Helmholtz-Gemeinschaft



DESY

Running slip for Covid-19 prevention (Two-sided form)

From 26. October 2020 required to participate in beamtimes at PETRA III and FLASH.

Before going to the experiment, approach the Photon Science Users Office to fill in this form and get signatures and instructions!

Surname: _____

Name: _____

Date of birth: _____

Institute / Group: _____

1) For employee on the Bahrenfeld Campus:

Hereby I confirm that I am employed on the Bahrenfeld Campus and observe the current "General safety rules at DESY for handling the corona virus".
A molecular biological test is not required.

Date _____ Signature (employee) _____

2) For Guests on the Bahrenfeld Campus:

Onsite from: _____ to: _____ at Beamline: _____

Current molecular biological test from _____ has been submitted and is negative. The DESY medical service must be consulted: ☐ yes ☐ no

Date _____ Signature (user office) _____

Photon Science Users Office: Building 47C / L106
Office Thomas Dietrich: Building 25B / 235 (only from 2:00 to 4:00 pm)

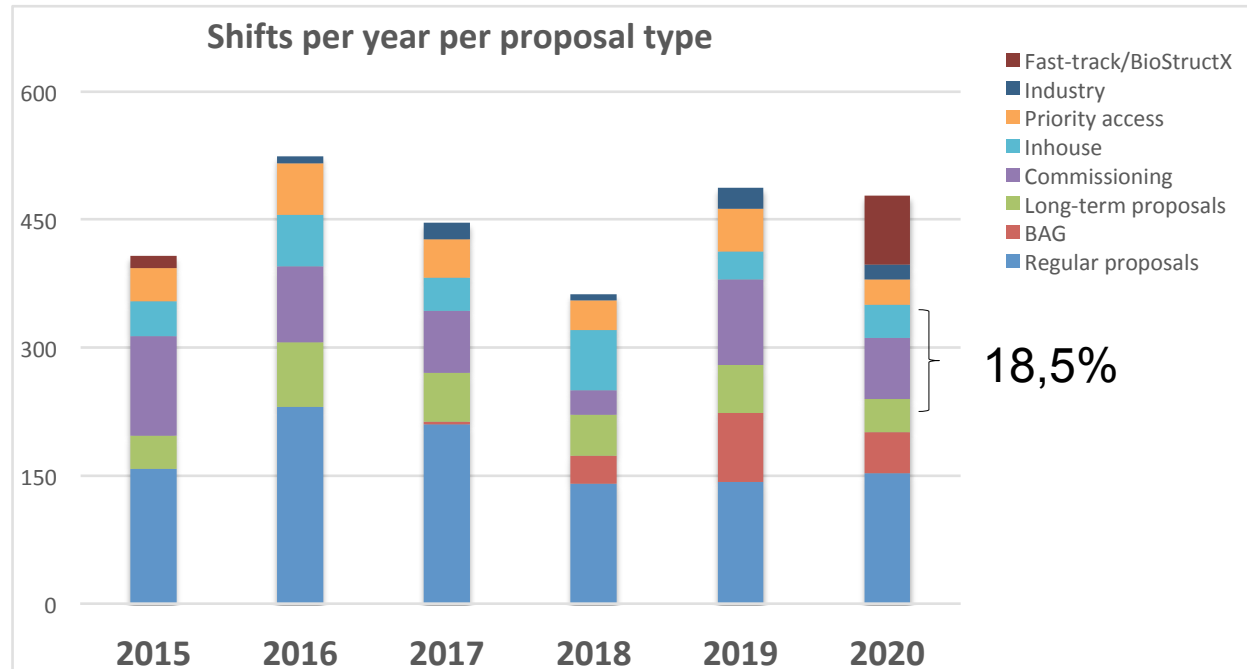
Deutsches Elektronen-Synchrotron DESY
Notkestraße 85
22607 Hamburg
www.desy.de

Standort Zeuthen
Platanenallee 6
15738 Zeuthen

Direktorium
Prof. Dr. H. Dösch
(Vorsitzender)
C. Harnagis
(Stv. Vorsitzender)
Prof. Dr. W. Leemans
Prof. Dr. J. Mnich
Prof. Dr. C. Stegmann
Prof. Dr. E. Weckert
Dr. A. Wiltner, CTO
(Bevollmächtigter des
Direktoriums für Innovation)

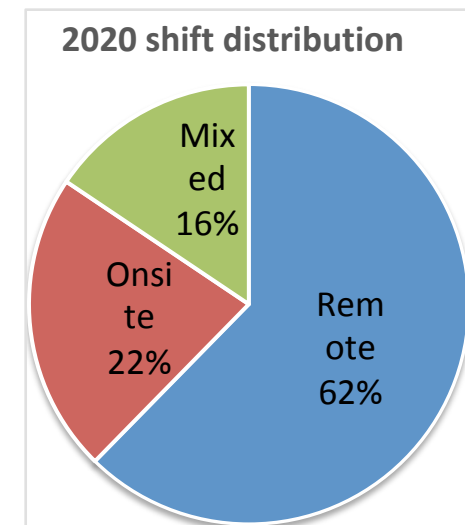
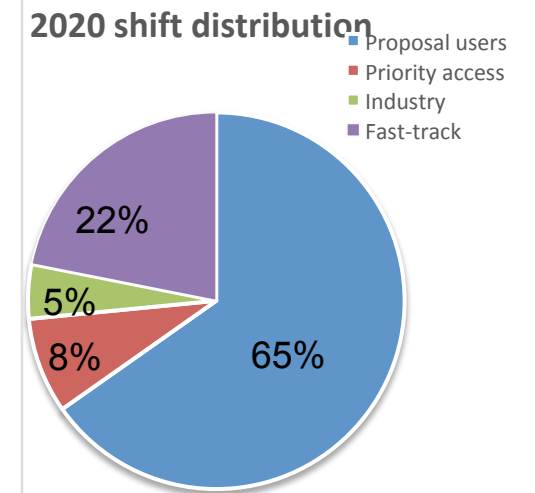
User statistics

All shifts



Numbers extracted from shifts actually carried out
 Inhouse and commissioning are by P11 staff only
 Serial Synchrotron Crystallography with tape-drive 24 shifts, with
 chips 18 shifts
 Some empty days due to repairs and short-notice cancellation
 11 shifts given out from INH for 'friendly users' i.e. users with no
 valid proposal => need for rolling access scheme

Excluding inhouse/ commissioning



SARS-CoV2 -related research arising from P11

Also outside of the fast-track procedure

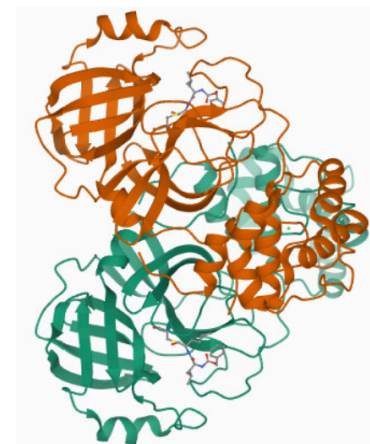
37 structures deposited from P11 (Main Protease)

- 30 from the fast-track screening campaign by Alke Meents

- 3 through other fast-track projects

- 2 through priority access

- 2 through regular proposals



Publications:

“Crystal structure of SARS-CoV-2 main protease provides a basis for design of improved α -ketoamide inhibitor” (Zhang et al., 2020, Science, 368, 409-412)

“Catalytic cleavage of HEAT and subsequent covalent binding of the tetralone moiety by the SARS-CoV-2 main protease” (Günther et al., 2020)

“SARS-CoV-2 MPro inhibitors and activity base probes for patient-sample imaging” (Rut et al., Nat. Chem. Biol., 2020)

“Repurposing the HCV NS3–4A protease drug boceprevir as COVID-19 therapeutics” (Oerlemans et al., RSC Medicinal Chemistry 2021)

Thank you

Johanna Hakanpää

Sofiane Saouane

Eva Crosas

Spyros Chatziefthymiou

Jan Meyer

Bernhard Kistner

Jakob Urbschat

Jan-Peter Kurz

