

X-ray fluorescence emission analysis of protein crystals

why Xfe-spectra?

- useful for new projects (substructure)
- quick check for HA presence
- easy check for 'exclusive' binding

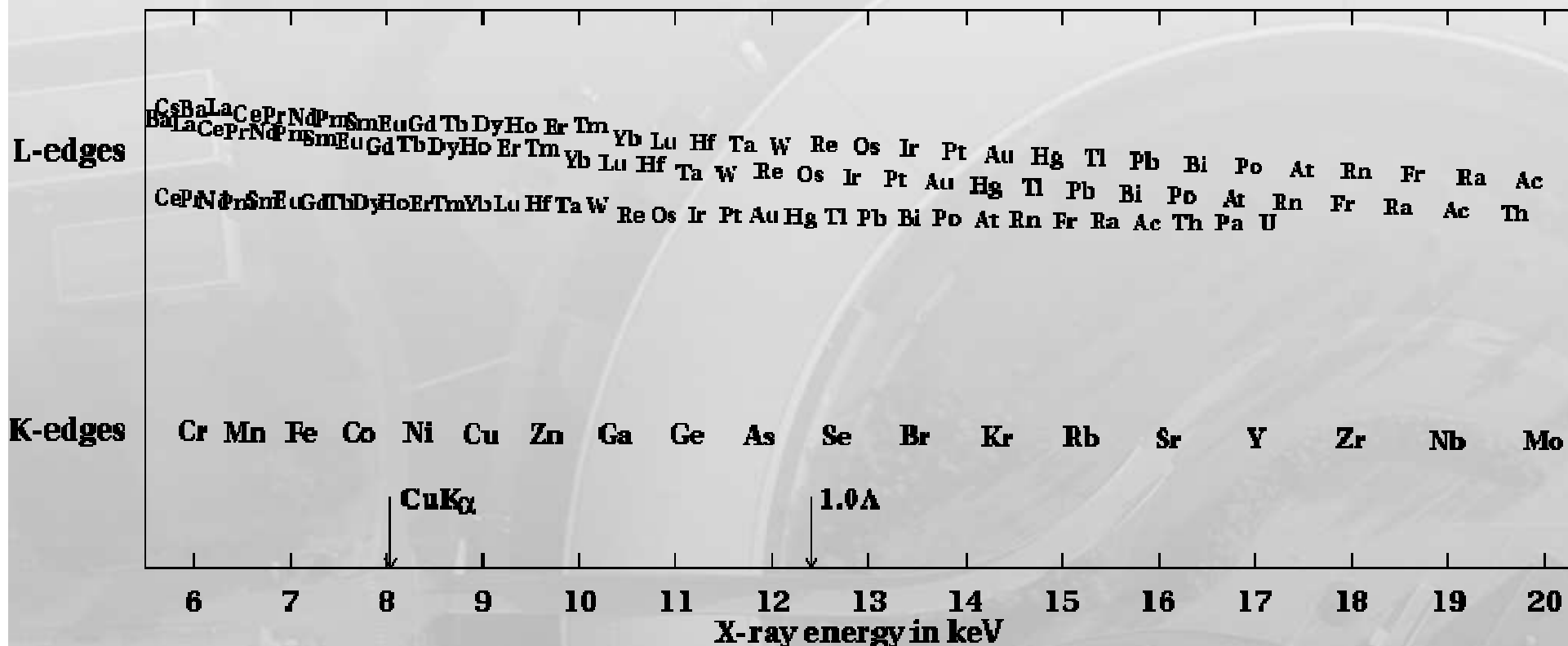
why Xfe-spectra?

- useful for new projects (substructure)
- quick check for HA presence
- easy check for 'exclusive' binding

- quick
- non (little) "destructive"
- higher sensitivity than absorption edge scans
- applicable also on fixed wavelength beamlines

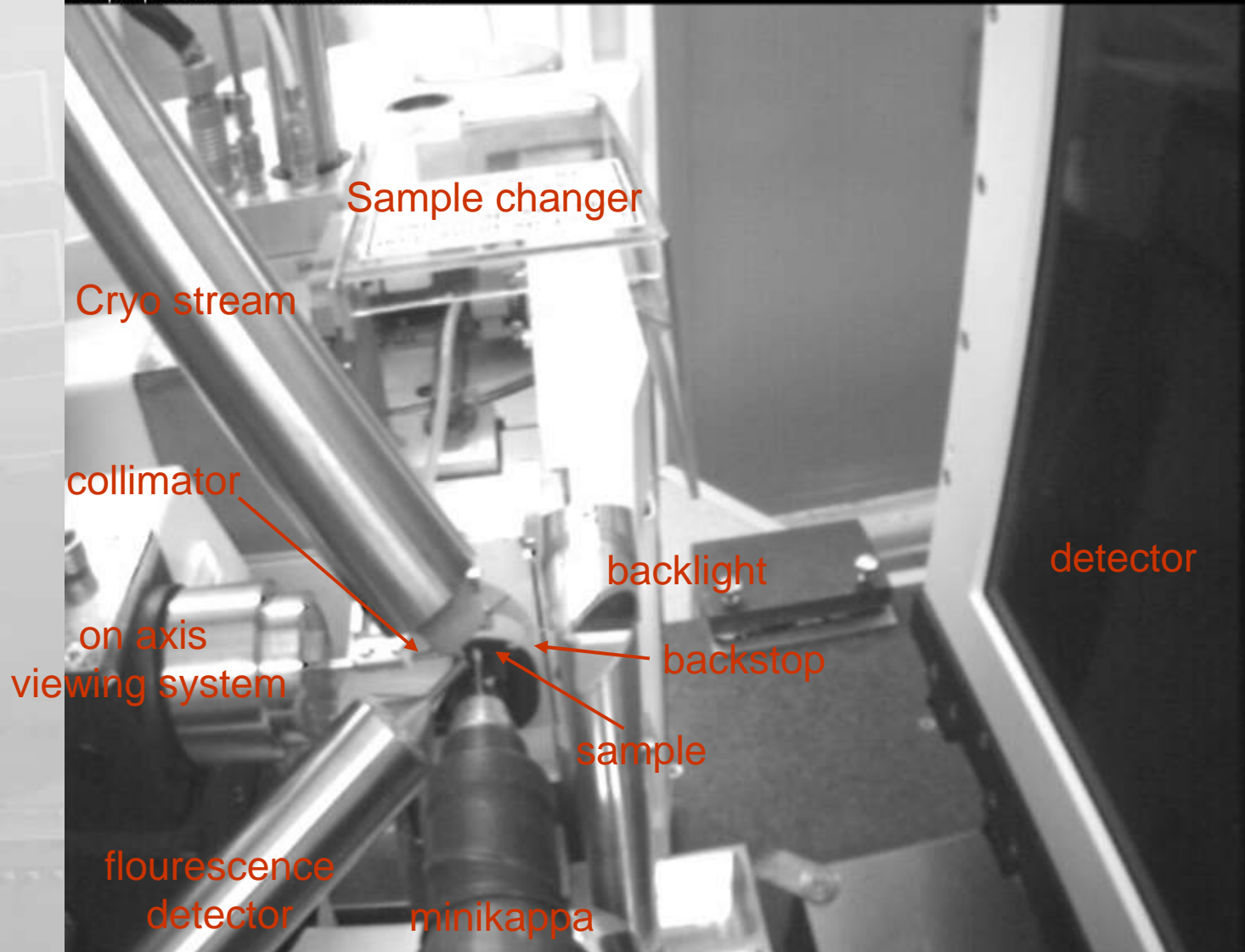
why Xfe-spectra?

Absorption edges useful for anomalous scattering experiments

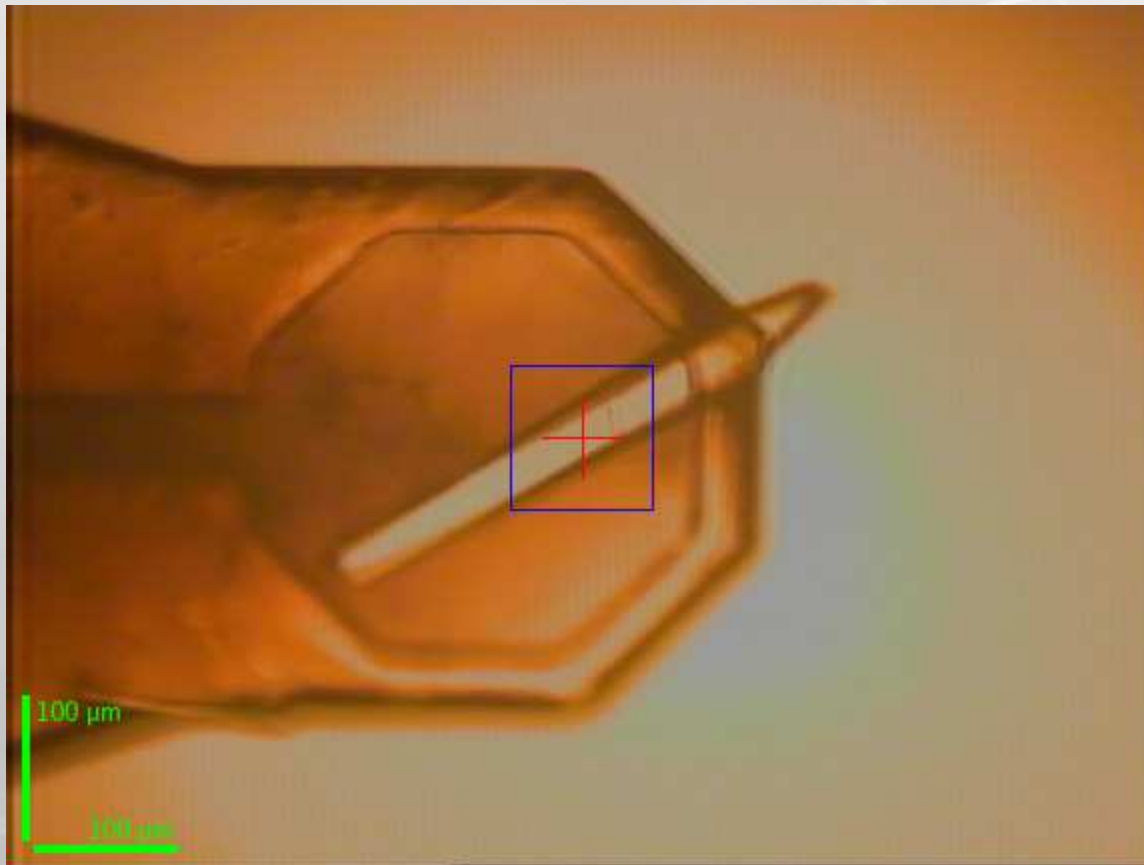


general sample environment

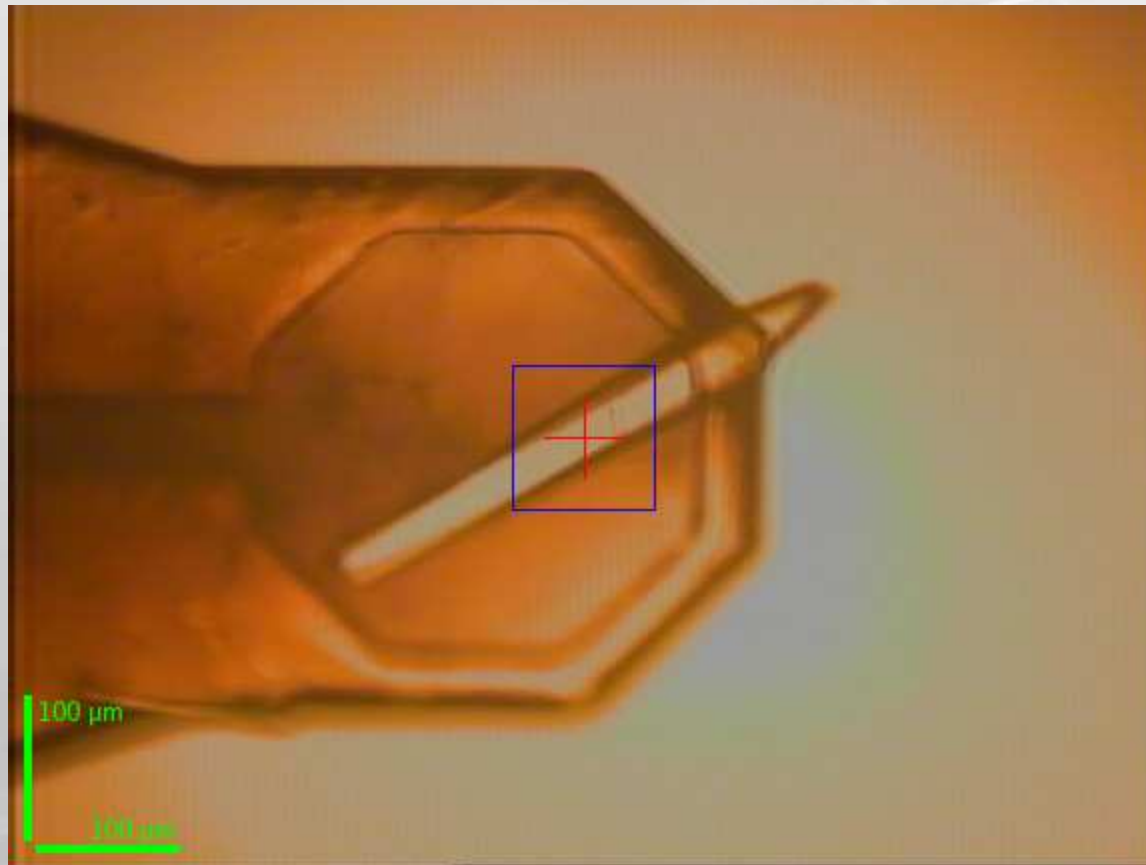
sample position 2007-11-26 17:18:54



Xfe-spectra



Xfe-spectra



**also possible with - 'bad' crystal
- protein solution**

Xfe-spectra

mxCuBE

File Instrumentation Help

Hutch Collect Energy sca **Xfe spectrum** Image

Xfespectrum Parameters

Count time (in seconds): 1 Prefix: Directory:

Xfe spectrum Xfe spectrum status

Function: Mca Hypermet

Background: No Background

Energy

Current: 12.7003 keV 0.976 Å

Move to: keV

Short Tail Long Tail Step Tail

Escape Pile-up Strip Back

GRAPH TABLE CONCENTRATIONS DIAGNOSTICS

Fit of XXXXXXXXXXXX from Channel XXXXX to XXXX

Counts

Channel

Fit Again! Print HTML Report Matrix Spectrum Peaks Spectrum

Information messages Chat (1)

2007-11-26 17:50:33 DataCollect: you should specify the fatal data collection return channel spec channel

2007-11-26 17:50:33 Transmission factor 0.740554%

2007-11-26 17:50:34 DataCollect: waiting for images...

2007-11-26 17:50:34 LdapLogin: connecting to LDAP server ldap.esrf.fr:389

2007-11-26 17:50:35 SampleChangerBrick3: state changed (STANDBY)

Machine current: 173.4 mA uniformmultibunch 03:09

Cryo: 100.0 K 51%

Dry: unknown

Superdry: unknown

Icing: unknown

Collect stage

1. Preparing beamline
2. Mounting sample
3. Centring sample
4. Taking snapshot
5. Collecting images
6. Unmounting sample

Current users

Xfe-spectra

mxCuBE (opid 291)

File Instrumentation Help



Expert mode

Hutch Collect Energy scan Xfe spectrum Image

Xfespectrum Parameters

Count time (in seconds): 5 Prefix: in Directory: /data/kd29/inhouse/opid291/christoph/xfe

Xfe spectrum

Xfe spectrum status

xfespectrum - mcaacq 5 s

Energy

Current: 12.7003 keV 0.976 Å

Move to: keV



Machine current

189.1 mA

uniformmultibunch

09:07

Cryo

100.0 K

53%

Dry: unknown

Superdry: unknown

Icing: unknown

Collect stage

1. Preparing beamline
2. Mounting sample
3. Centring sample
4. Taking snapshot
5. Collecting images
6. Unmounting sample

Function Mca Hypermet

Background No Background

Fit of Unknown Origin from X 515.000 to 2547.000

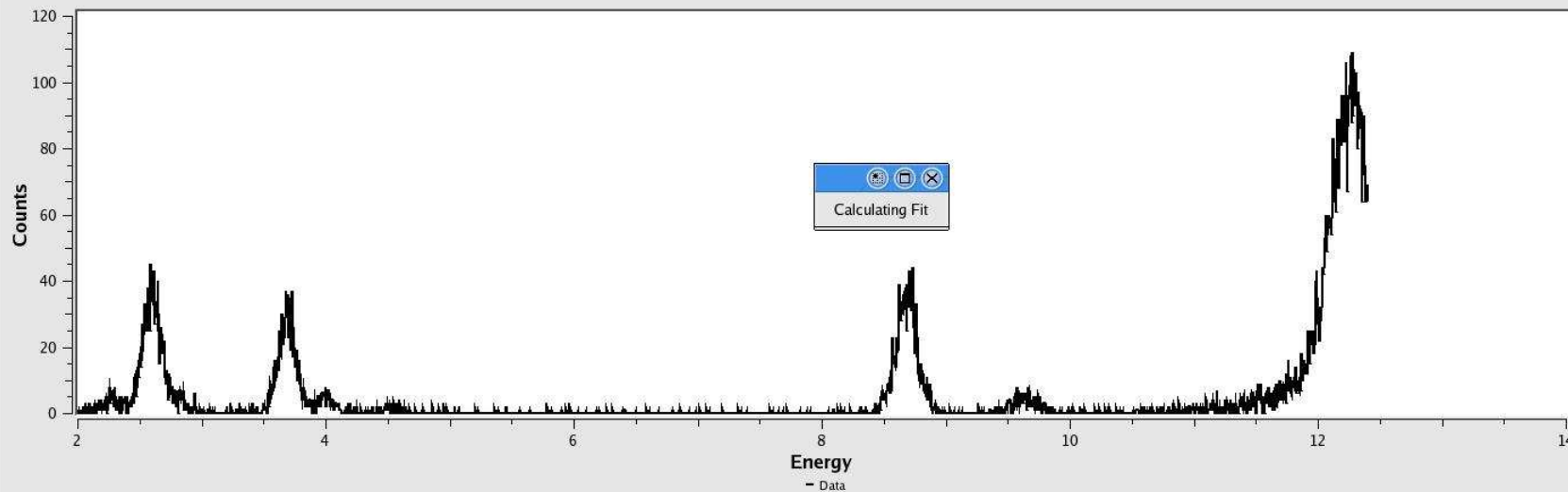
Short Tail Long Tail Step Tail

Escape Pile-up Strip Back

GRAPH TABLE CONCENTRATIONS DIAGNOSTICS



x: 9.023 y: 58.12



Information messages

2007-11-24 11:45:15 Moving light out
2007-11-24 11:45:15 Moving Detector to 110.278 and Back stop in
2007-11-24 11:45:46
2007-11-24 11:48:25 MiniDiff: taking snapshot #1
2007-11-24 11:48:30 MiniDiff: taking snapshot #2

Current users

Xfe-spectra

mxCuBE (opid-291)

File Instrumentation Help



Expert mode

Hutch Collect Energy scan Xfe spectrum Image

 Xfespectrum Parameters

Count time (in seconds): 5 Prefix: th Directory: /data/id29/inhouse/opid291/christoph/xfe

Browse

Xfe spectrum

Xfe spectrum status

Start spectrum

xfespectrum - mcaacq 5 s

 Energy

Current: 12.7003 keV 0.976 Å

Move to: keV

Function Mca Hypermet

Background No Background

Fit of Unknown Origin from X 515.000 to 2547.000

 Short Tail Long Tail Step Tail Escape Pile-up Strip Back

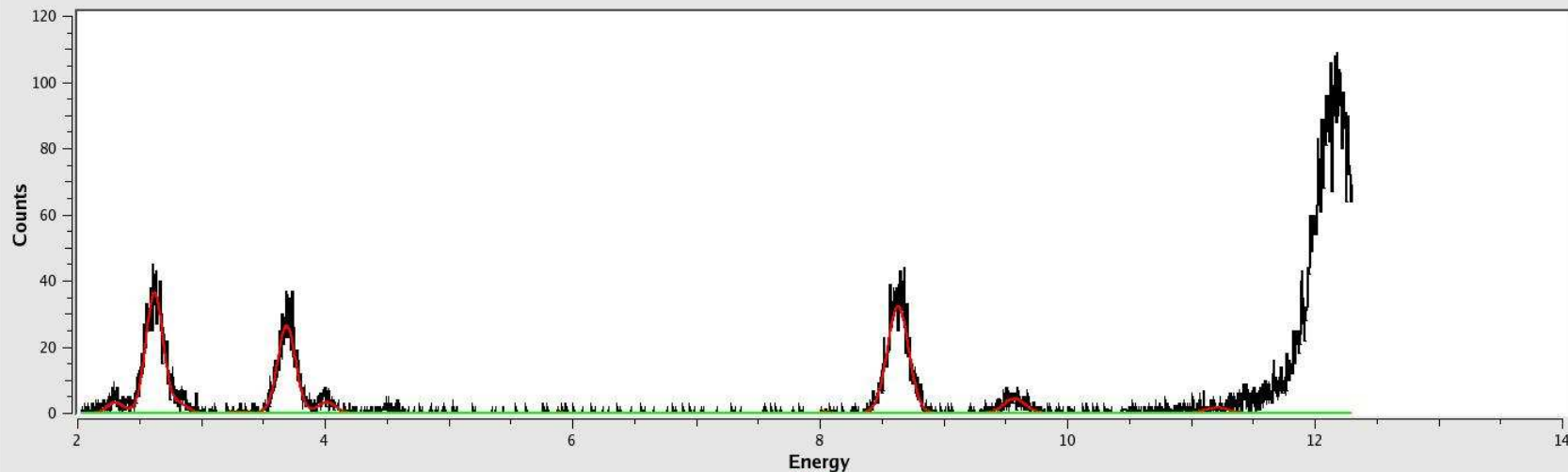
Configure

Tools

GRAPH TABLE CONCENTRATIONS DIAGNOSTICS



X: 9.89 Y: 15.043



Fit Again! Print HTML Report Matrix Spectrum Peaks Spectrum

Information messages

Submit feedback

Chat (1)

spec (49064)

2007-11-24 11:45:15 Moving light out
2007-11-24 11:45:15 Moving Detector to 110.278 and Back stop in
2007-11-24 11:45:46
2007-11-24 11:48:25 MiniDiff: taking snapshot #1
2007-11-24 11:48:30 MiniDiff: taking snapshot #2



Machine current

189.0 mA

uniformmultibunch

09:06

Cryo

100.0 K

53%

Dry: unknown

Superdry: unknown

Icing: unknown

Collect stage

1. Preparing beamline
2. Mounting sample
3. Centring sample
4. Taking snapshot
5. Collecting images
6. Unmounting sample

Current users

Xfe-spectra

mxCuBE (opid-291)

File Instrumentation Help



Expert mode

Hutch Collect Energy scan Xfe spectrum Image

 Xfespectrum Parameters

Count time (in seconds): 5 Prefix: th Directory: /data/id29/inhouse/opid291/christoph/xfe

Browse

Xfe spectrum

Xfe spectrum status

Start spectrum

xfespectrum - mcaacq 5 s

 Energy

Current: 12.7003 keV 0.976 Å

Move to: keV

Function Mca Hypermet

Background No Background

Fit of Unknown Origin from X 515.000 to 2547.000

 Short Tail Long Tail Step Tail Escape Pile-up Strip Back

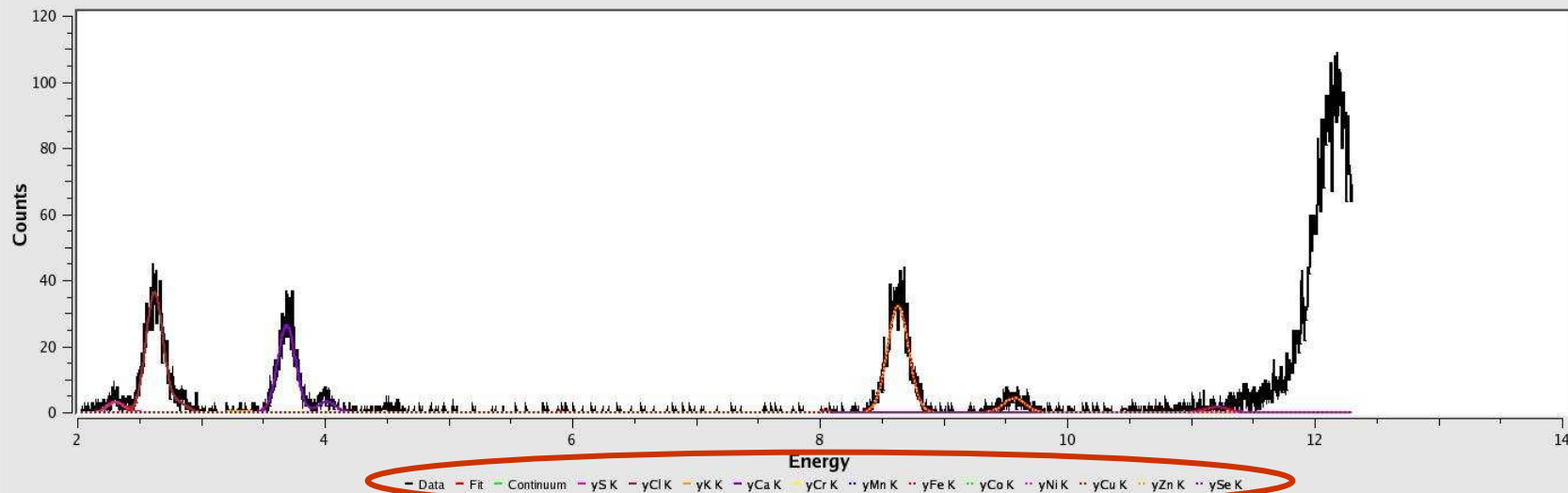
Configure

Tools

GRAPH TABLE CONCENTRATIONS DIAGNOSTICS



x: 6.831 y: -2.3932



Fit Again! Print HTML Report Matrix Spectrum Peaks Spectrum

Information messages

Submit feedback

Chat (1)

spec (49064)

2007-11-24 11:45:15 Moving light out
2007-11-24 11:45:15 Moving Detector to 110.278 and Back stop in
2007-11-24 11:45:46
2007-11-24 11:48:25 MiniDiff: taking snapshot #1
2007-11-24 11:48:30 MiniDiff: taking snapshot #2



Machine current

188.6 mA

uniformmultibunch

08:57

Cryo

100.1 K

52%

Dry: unknown

Superdry: unknown

Icing: unknown

Collect stage

1. Preparing beamline
2. Mounting sample
3. Centring sample
4. Taking snapshot
5. Collecting images
6. Unmounting sample

Current users

Xfe-spectra

mxCuBE (opid 291) Expert mode

File Instrumentation Help

Hutch Collect Energy scan Xfe spectrum Image

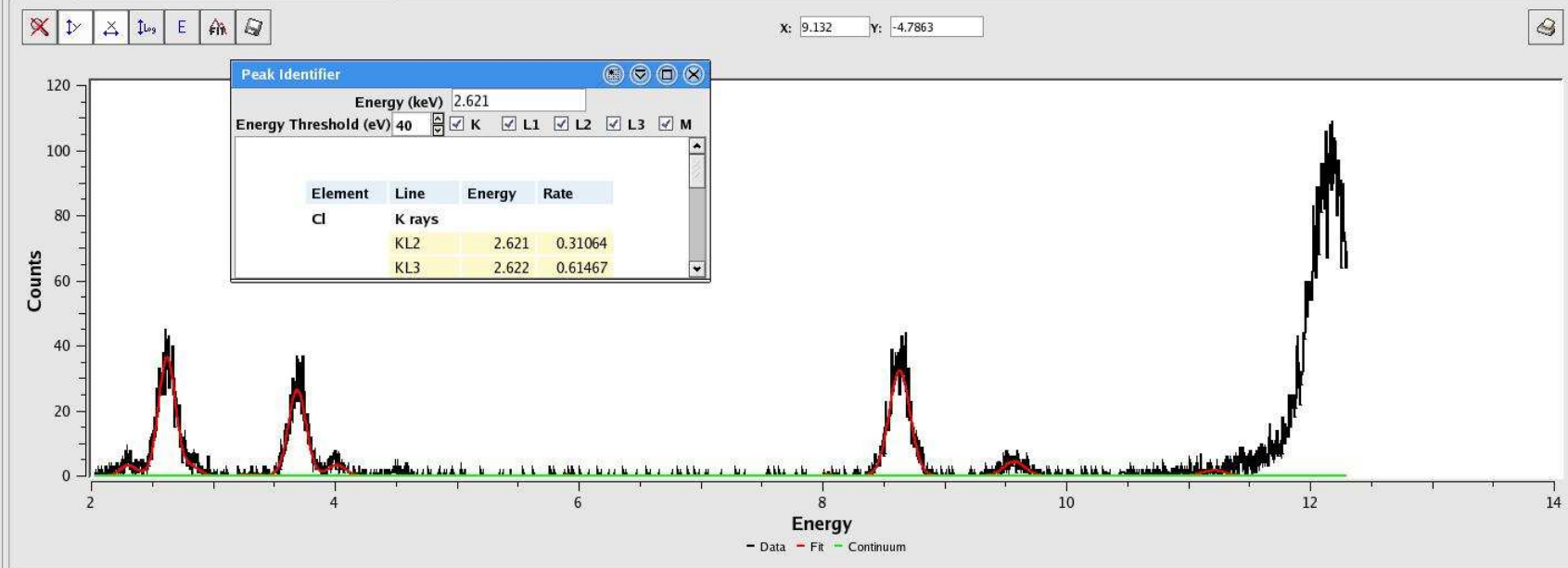
Xfespectrum Parameters
 Count time (in seconds): 5 Prefix: th Directory: /data/id29/inhouse/opid291/christoph/xfe Browse

Xfe spectrum Xfe spectrum status
xfespectrum - mcaacq 5 s

Energy
 Current: 12.7003 keV 0.976 Å
 Move to: keV

Function: Mca Hypermet
 Background: No Background
 Fit of Unknown Origin from X 515.000 to 2547.000
 Short Tail Long Tail Step Tail
 Escape Pile-up Strip Back

GRAPH TABLE CONCENTRATIONS DIAGNOSTICS



Machine current
189.0 mA
 uniformmultibunch
 09:05

Cryo
 99.8 K
 53%

Dry: unknown
 Superdry: unknown
 Icing: unknown

Collect stage

- Preparing beamline
- Mounting sample
- Centring sample
- Taking snapshot
- Collecting images
- Unmounting sample

Information messages Submit feedback Chat (1) spec (49064)

2007-11-24 11:45:15 Moving light out
 2007-11-24 11:45:15 Moving Detector to 110.278 and Back stop in
 2007-11-24 11:45:46
 2007-11-24 11:48:25 MiniDiff: taking snapshot #1
 2007-11-24 11:49:30 MiniDiff: taking snapshot #2

Current users

Xfe-spectra

mxCuBE (opid-291)

File Instrumentation Help



Expert mode

Hutch Collect Energy scan Xfe spectrum Image

 Xfespectrum Parameters

Count time (in seconds): 5 Prefix: th Directory: /data/id29/inhouse/opid291/christoph/xfe

Browse

Xfe spectrum

Xfe spectrum status

Start spectrum

xfespectrum - mcaacq 5 s

 Energy

Current: 12.7003 keV 0.976 Å

Move to: keV

Function Mca Hypermet

Background No Background

Fit of Unknown Origin from X 515.000 to 2547.000

 Short Tail Long Tail Step Tail Escape Pile-up Strip Back

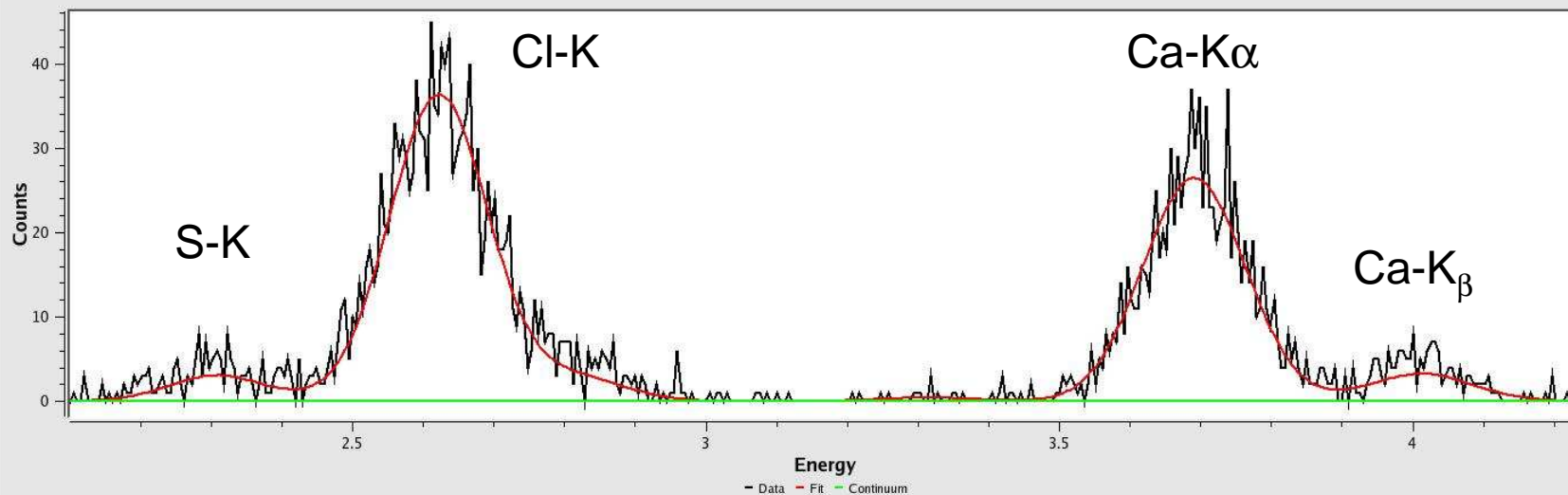
Configure

Tools

GRAPH TABLE CONCENTRATIONS DIAGNOSTICS



x: 4.237 y: -1.7094



Fit Again! Print HTML Report Matrix Spectrum Peaks Spectrum

Information messages Submit feedback Chat (1) spec (49064)

2007-11-24 11:45:15 Moving light out
2007-11-24 11:45:15 Moving Detector to 110.278 and Back stop in
2007-11-24 11:45:46
2007-11-24 11:48:25 MiniDiff: taking snapshot #1
2007-11-24 11:48:30 MiniDiff: taking snapshot #2



Machine current

189.0 mA

uniformmultibunch

09:05

Cryo

100.0 K

52%

Dry: unknown

Superdry: unknown

Icing: unknown

Collect stage

1. Preparing beamline
2. Mounting sample
3. Centring sample
4. Taking snapshot
5. Collecting images
6. Unmounting sample

Current users

Xfe-spectra

mxCuBE (opid 291)

File Instrumentation Help



Expert mode

Hutch Collect Energy scan Xfe spectrum Image

 Xfespectrum Parameters

Count time (in seconds): 5 Prefix: th Directory: /data/id29/inhouse/opid291/christoph/xfe

Browse

Xfe spectrum

Xfe spectrum status

Start spectrum

xfespectrum - mcaacq 5 s

 Energy

Current: 12.7003 keV 0.976 Å

Move to: keV

Function Mca Hypermet

Background No Background

Fit of Unknown Origin from X 515.000 to 2547.000

 Short Tail Long Tail Step Tail Escape Pile-up Strip Back

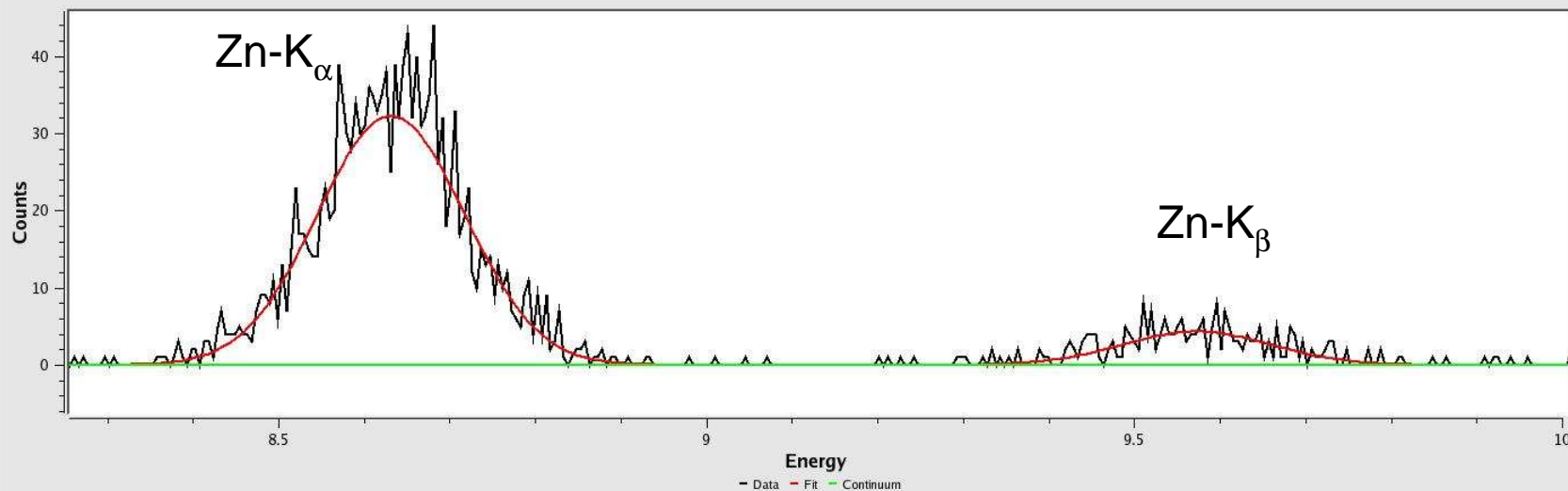
Configure

Tools

GRAPH TABLE CONCENTRATIONS DIAGNOSTICS



x: 10 y: -6.1538



Fit Again! Print HTML Report Matrix Spectrum Peaks Spectrum

Information messages

Submit feedback

Chat (1)

spec (49064)

2007-11-24 11:45:15 Moving light out
2007-11-24 11:45:15 Moving Detector to 110.278 and Back stop in
2007-11-24 11:45:46
2007-11-24 11:48:25 MiniDiff: taking snapshot #1
2007-11-24 11:48:38 MiniDiff: taking snapshot #2



Machine current

189.0 mA

uniformmultibunch

09:05

Cryo

100.2 K

52%

Dry: unknown

Superdry: unknown

Icing: unknown

Collect stage

1. Preparing beamline
2. Mounting sample
3. Centring sample
4. Taking snapshot
5. Collecting images
6. Unmounting sample

Current users

Xfe-spectra

mxCuBE (opid-291)

File Instrumentation Help

Expert mode

Hutch Collect Energy scan Xfe spectrum Image

Xfespectrum Parameters

Count time (in seconds) : 5 Prefix: tln Directory: /data/id29/inhouse/opid291/christoph/xfe

Energy 12.7003 keV 0.976 Å
Move to: keV

Xfe spectrum status: xfespectrum - mcaacq 5 s

Function Mca Hypermet

Fit of Unknown Origin from X 515.000 to 2547.000
 Short Tail Long Tail Step Tail
 Escape Pile-up Strip Back

Background No Background

GRAP TABLE CONCENTRATIONS DIAGNOSTICS

Element	Group	Fit Area	Sigma	Energy	Ratio	FWHM	Chi square
1 S	K	1.0955e+02	1.14e+01				
2	KL3	1.0318e+02	1.07e+01	2.307	0.94185	0.161	1.01
3	KM3	6.3702e+00	6.61e-01	2.464	0.05815	0.162	1.08
4	KL3 SLKM3esc	4.4284e-02	4.60e-03	0.471	0.00043	0.147	0.00
5	KL3 SLKL3esc	1.5305e+00	1.59e-01	0.568	0.01483	0.148	0.00
6	KM3 SLKM3esc	2.6189e-03	2.72e-04	0.628	0.00041	0.148	0.00
7	KM3 SLKL3esc	9.0134e-02	9.36e-03	0.724	0.01415	0.149	0.00
8 Cl	K	1.3468e+03	3.70e+01				
9	KL3	1.2462e+03	3.43e+01	2.622	0.92531	0.163	1.16
10	KM3	1.0059e+02	2.77e+00	2.816	0.07469	0.164	1.08
11	KL3 SLKM3esc	4.8833e-01	1.34e-02	0.786	0.00039	0.150	0.00
12	KL3 SLKL3esc	1.6731e+01	4.60e-01	0.882	0.01343	0.150	0.00
13	KM3 SLKM3esc	3.6913e-02	1.01e-03	0.980	0.00037	0.151	0.00
14	KM3 SLKL3esc	1.2573e+00	3.46e-02	1.076	0.01250	0.152	0.00
15 K	K	1.7483e+01	7.97e+00				
16	KL3	1.5671e+01	7.14e+00	3.313	0.89635	0.168	0.29
17	KM3	1.8122e+00	8.26e-01	3.590	0.10365	0.170	0.78
18	KL3 SLKM3esc	4.8636e-03	2.22e-03	1.477	0.00031	0.155	0.00
19	KL3 SLKL3esc	1.6341e-01	7.45e-02	1.573	0.01043	0.156	0.00
20	KM3 SLKM3esc	5.1050e-04	2.33e-04	1.754	0.00028	0.157	0.00
21	KM3 SLKL3esc	1.7030e-02	7.76e-03	1.850	0.00940	0.158	0.32
22 Ca	K	1.0679e+03	3.30e+01				
23	KL3	9.4866e+02	2.93e+01	3.690	0.88837	0.170	0.86

Fit Again! Print HTML Report Matrix Spectrum Peaks Spectrum

Information messages Submit feedback Chat (1) spec (49064)

2007-11-24 11:45:15 Moving light out
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2007-11-24 11:45:46
2007-11-24 11:48:25 MiniDiff: taking snapshot #1
2007-11-24 11:48:28 MiniDiff: taking snapshot #2



Machine current
188.9 mA
uniformmultibunch
09:04

Cryo
99.8 K 52%
Dry: unknown
Superdry: unknown
Icing: unknown

- Collect stage
- Preparing beamline
 - Mounting sample
 - Centring sample
 - Taking snapshot
 - Collecting images
 - Unmounting sample

Current users

Conclusion

- quick
- high sensitivity
- useful with I) new projects
II) rapid check for HA presence (when “back-soaked”)
- completely integrated into beamline software
- easy to use / user friendly