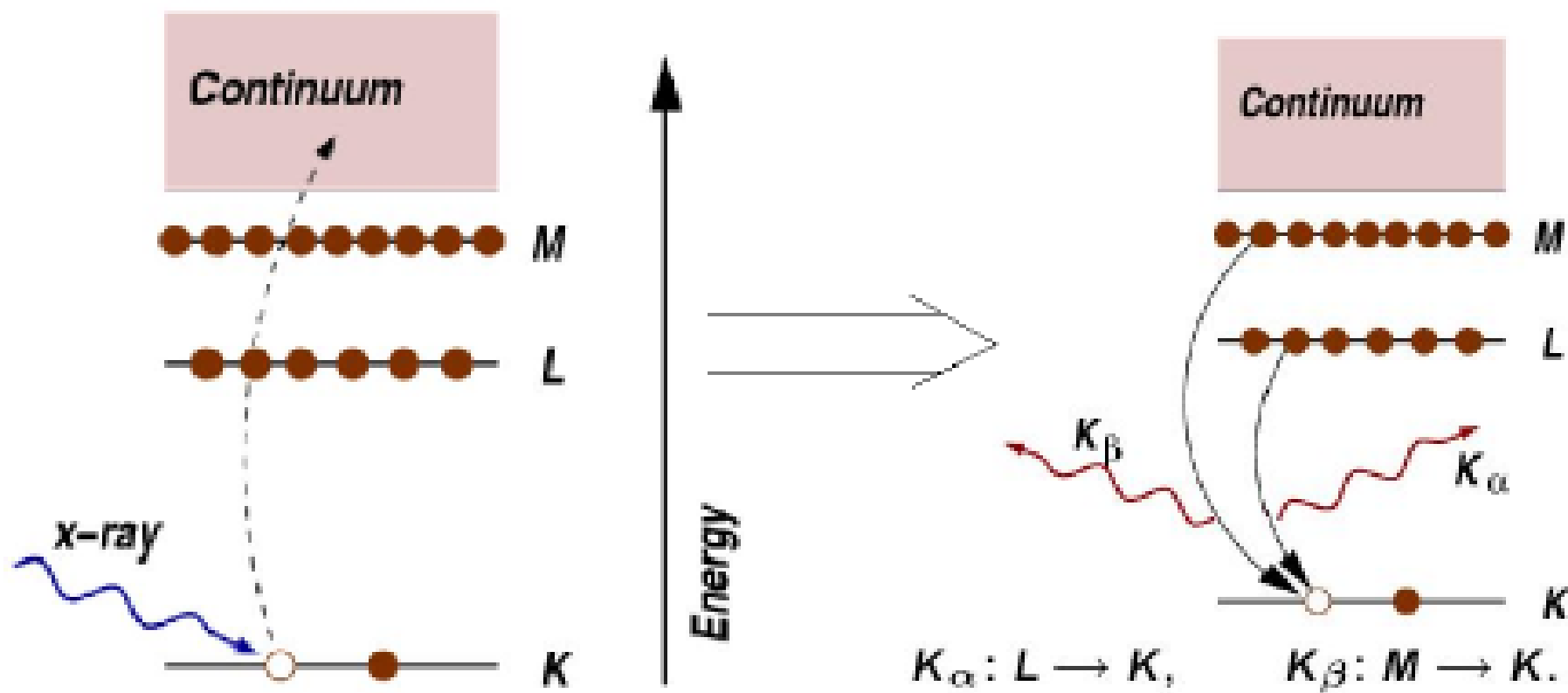


# X-ray fluorescence (XRF) analysis of protein crystals



# why XRF-spectra?

useful for

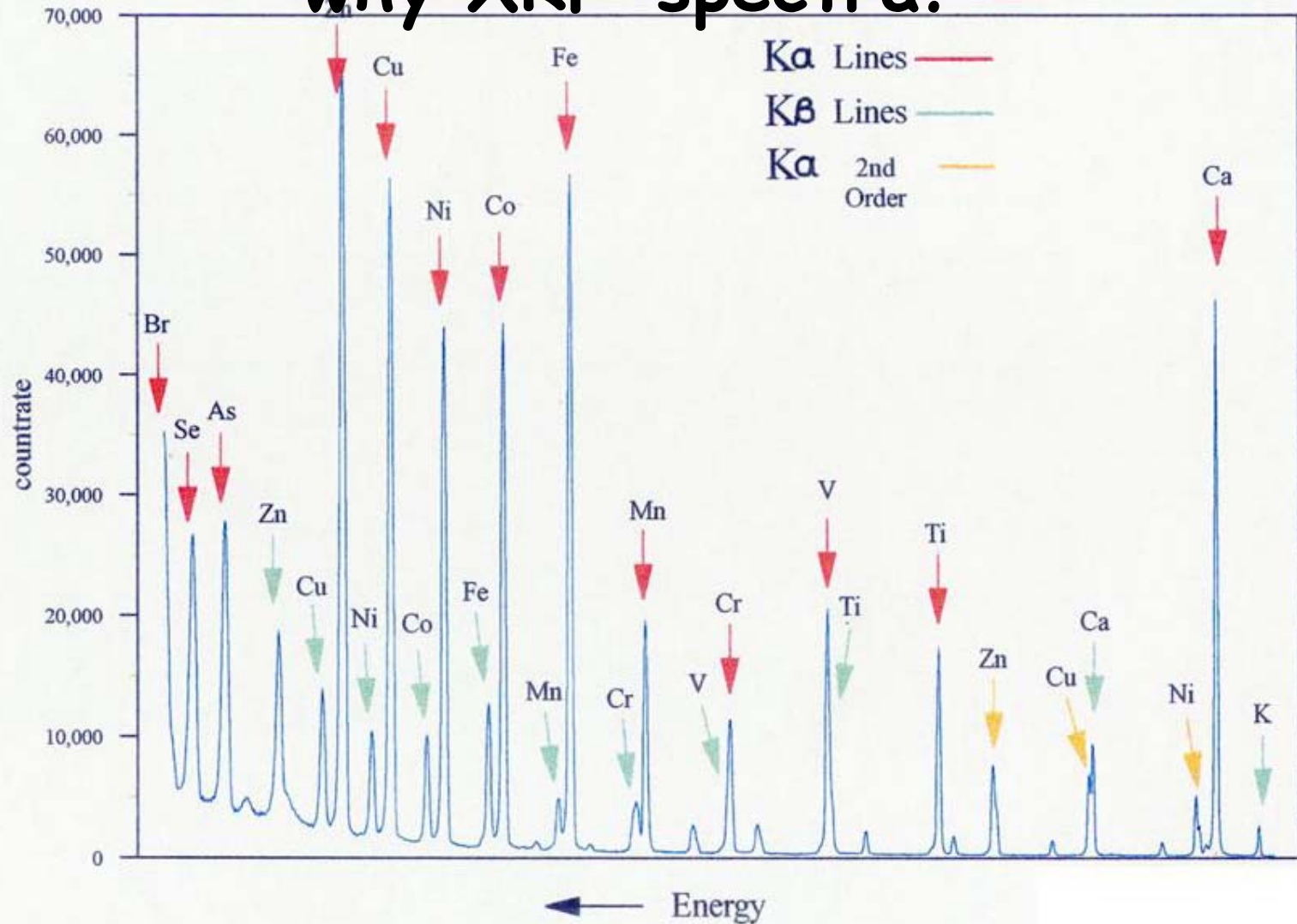
- Crystallisation
- New projects (substructure)
- Heavy atom presence
- 'Exclusive' binding

# why XRF-spectra?

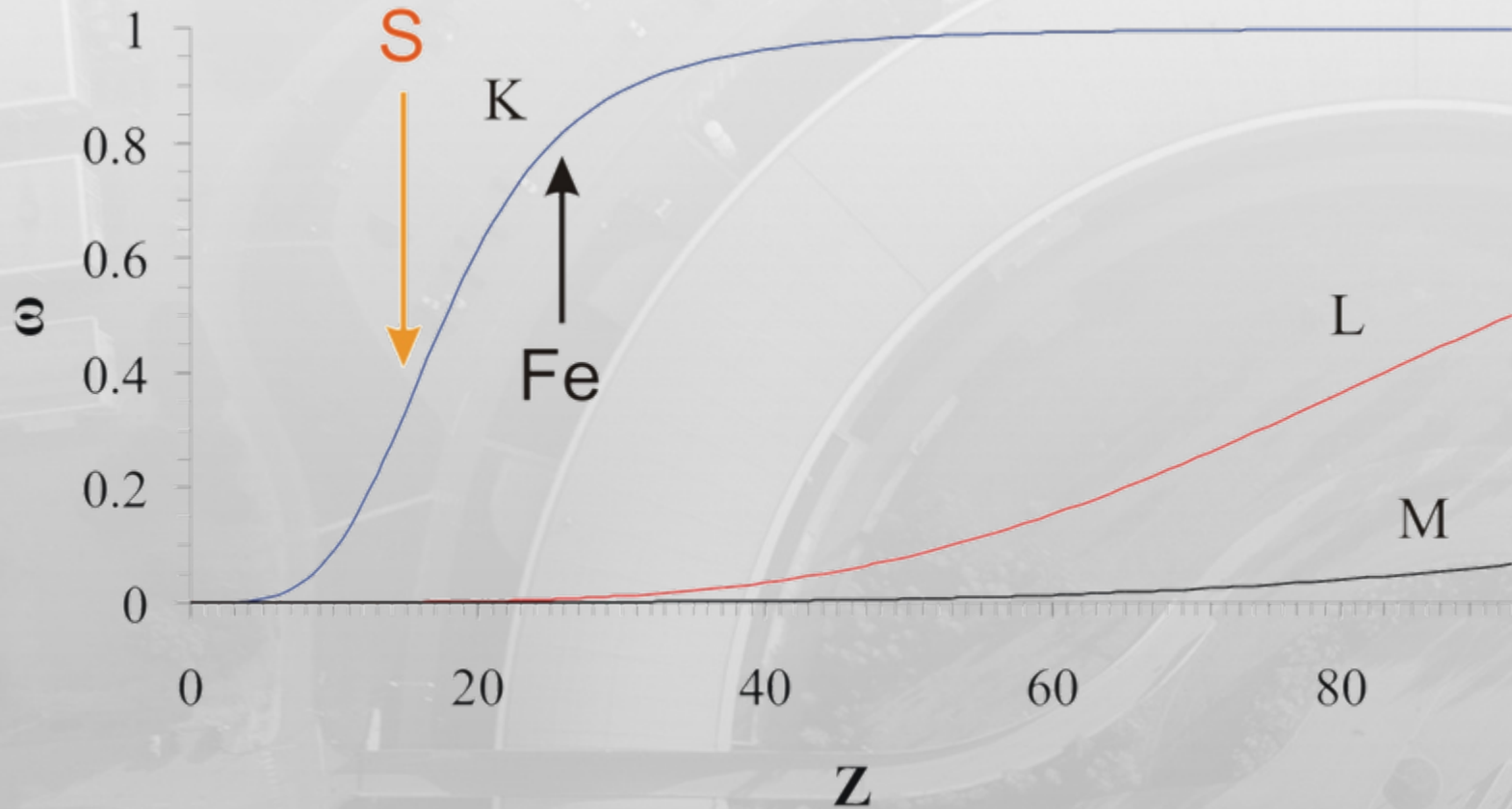
useful for

- Crystallisation
- New projects (substructure)
- Heavy atom presence
- 'Exclusive' binding
- Quick
- Non (little) "destructive"
- Higher sensitivity than absorption edge scans
- Applicable also on fixed wavelength beamlines

# why XRF-spectra?

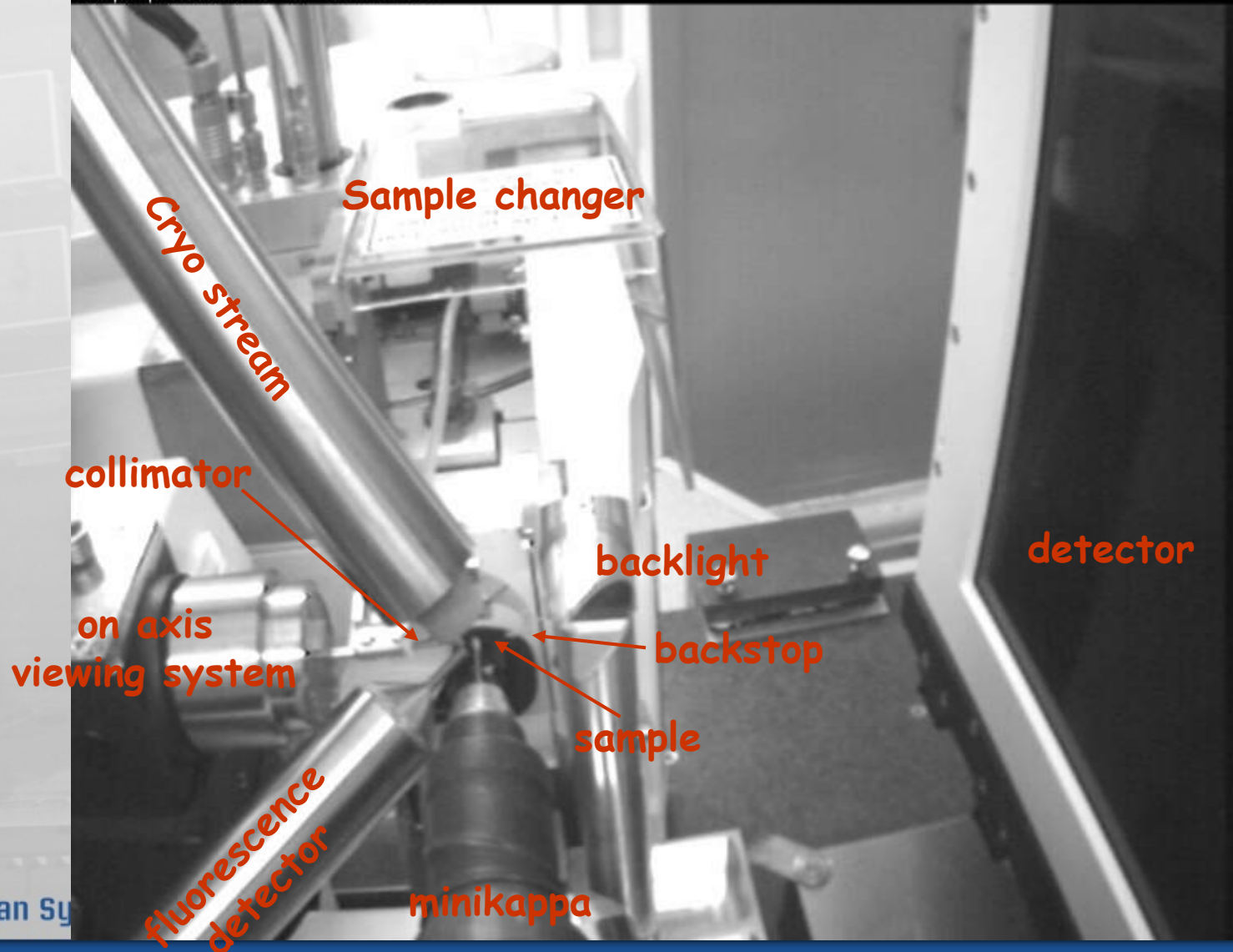


# why XRF-spectra?

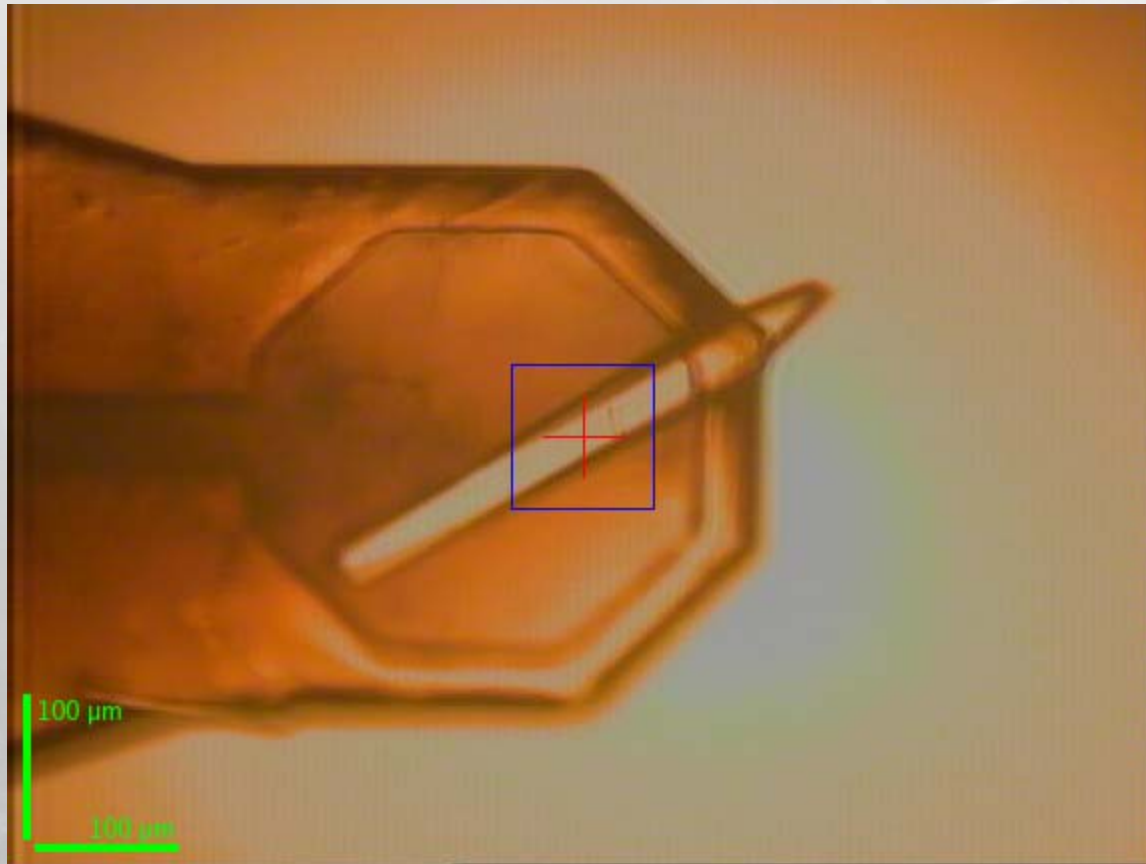


# general sample environment

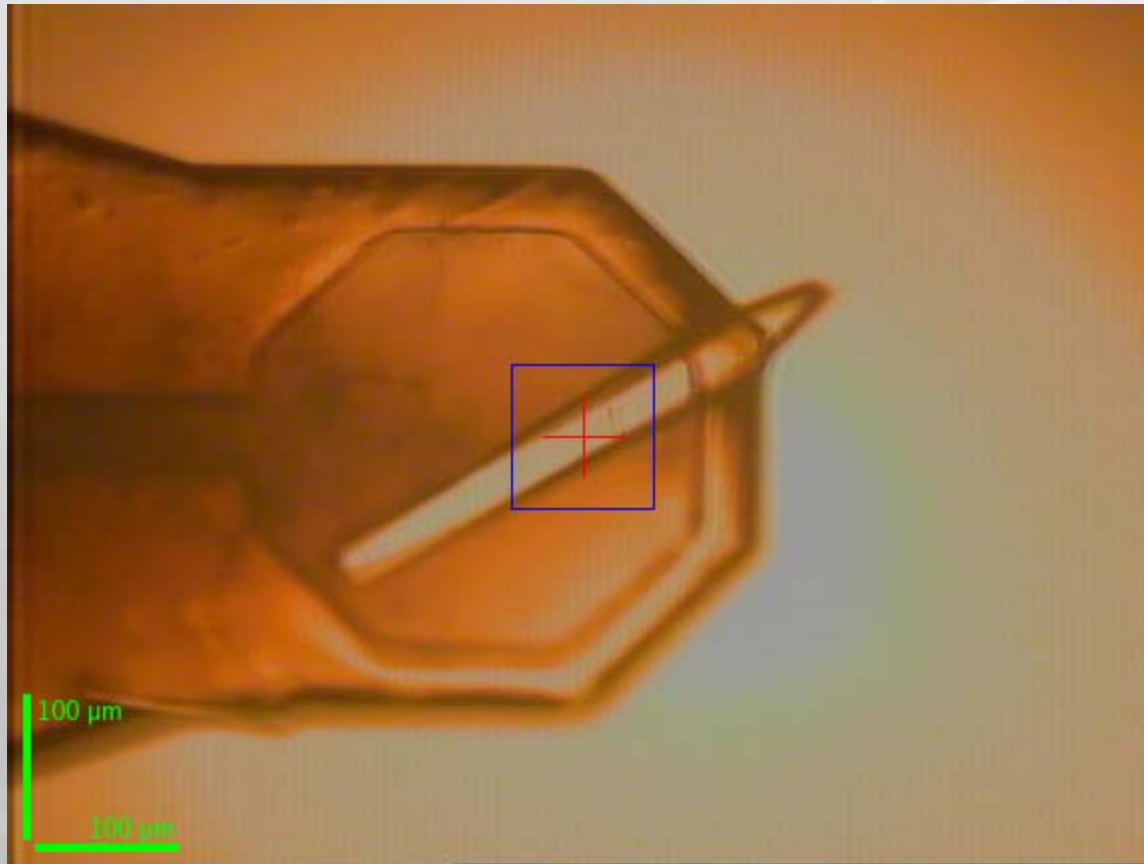
sample position 2007-11-26 17:18:54



# XRF - spectra



# XRF - spectra



also possible with

- 'bad' crystal
- protein solution

# XRF - spectra

mxCuBE

File Instrumentation Help

Hutch

Collect

Energy sca

Xfe spectrum

Image

☒ Xfespectrum Parameters
 

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

Browse

Xfe spectrum

Start spectrum

Xfe spectrum status

Function

Mca Hypermet

Background

No Background

GRAPH

TABLE

CONCENTRATIONS

DIAGNOSTICS

Fit

Zoom

Log

Y-axis

X-axis

Fit

Print

X: Y:

Fit of XXXXXXXXXX from Channel XXXXX to XXXX

☐ Short Tail
 ☐ Long Tail
 ☐ Step Tail
 ☒ Escape
 ☐ Pile-up
 ☐ Strip Back.

Configure

Tools

Energy

Current: 12.7003 keV 0.976 Å

Move to: keV

Counts

Channel

Fit Again!

Print

HTML Report

Matrix Spectrum

Peaks Spectrum

Information messages

Submit feedback

Chat (1)

spec (38)

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)

ESRF

Machine current

173.4 mA

uniformmultibunch

03:09

Cryo

100.0 K

51%

Dry: unknown

Superdry: unknown

Icing: unknown

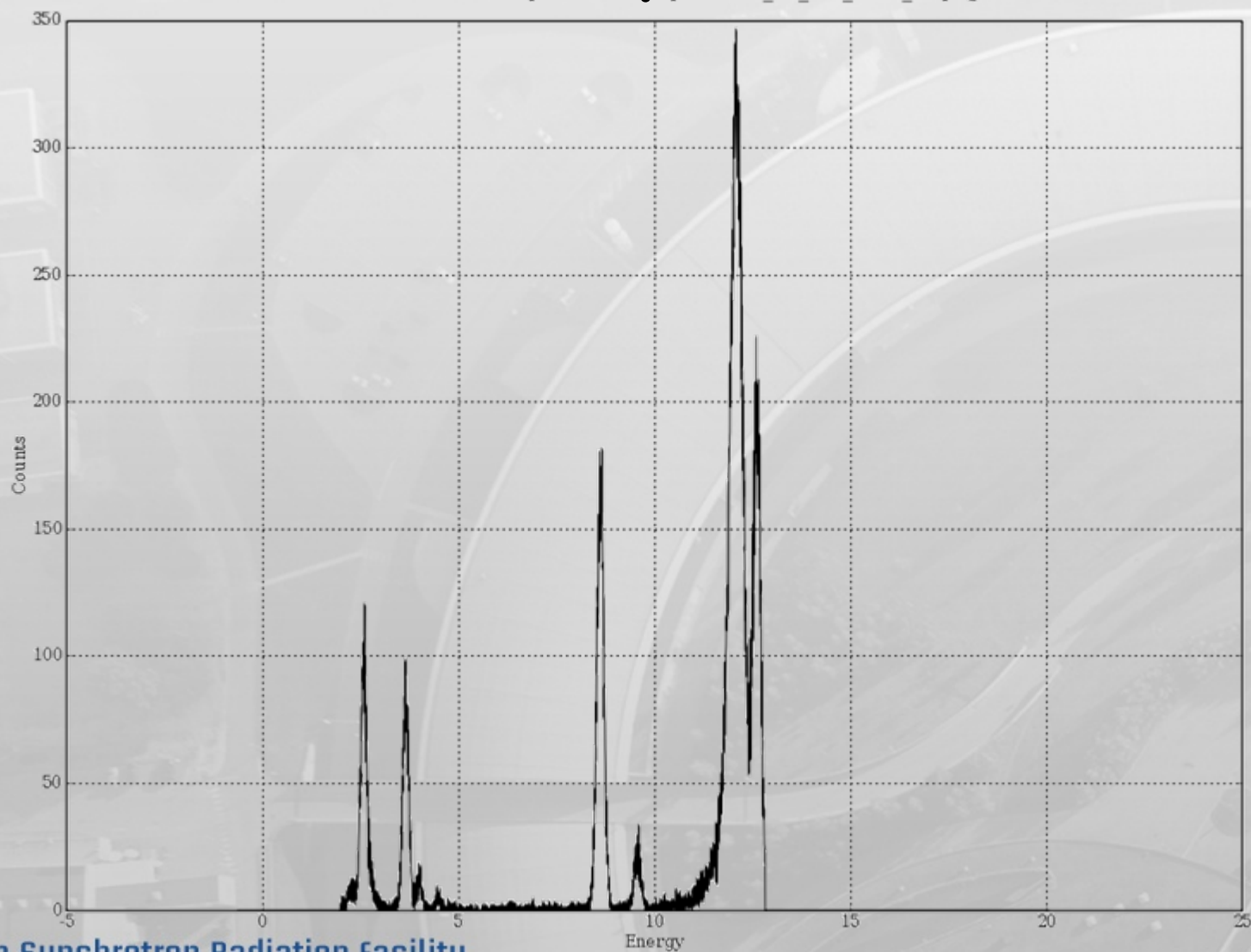
Collect stage

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

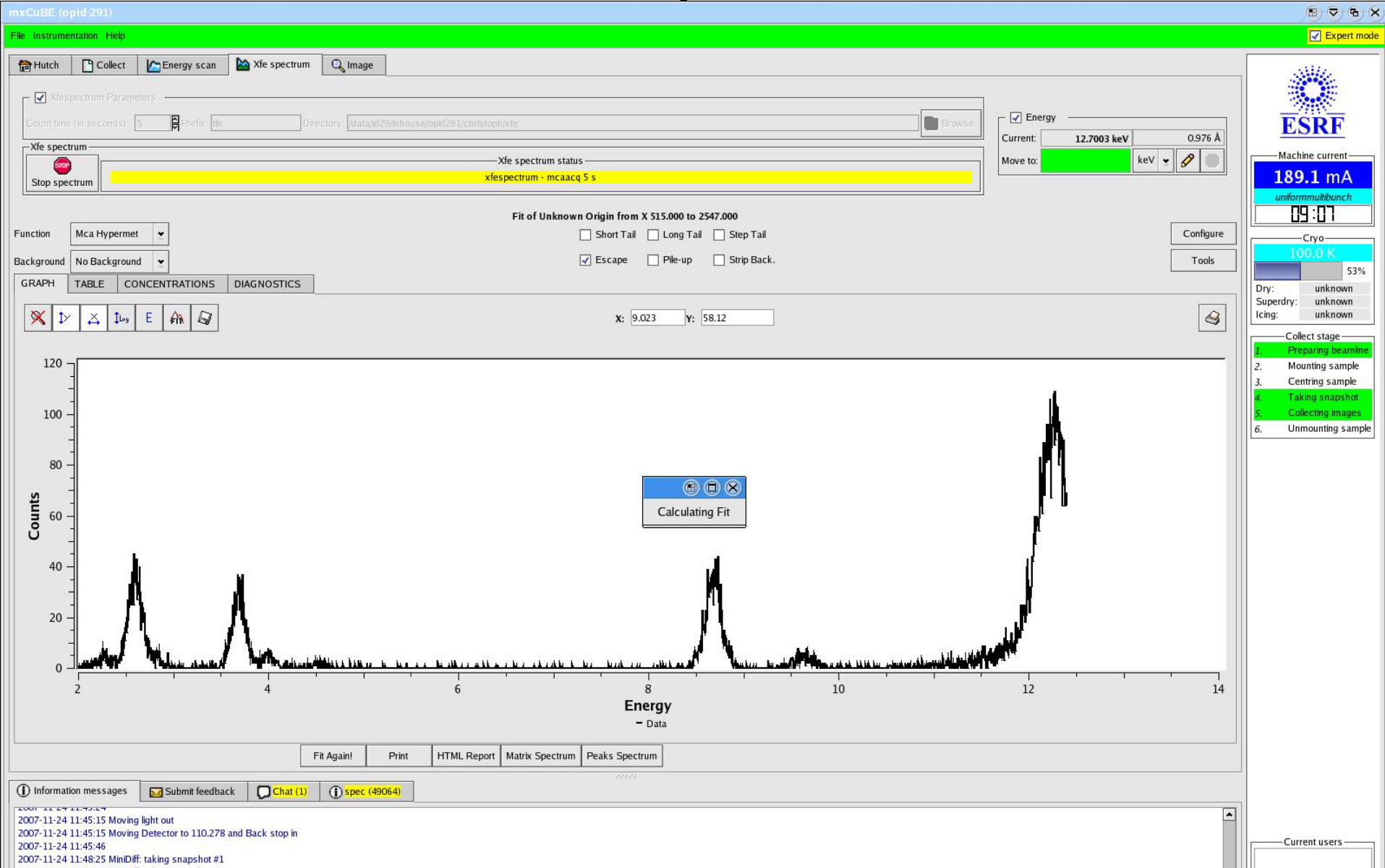
Current users

# XRF - spectra

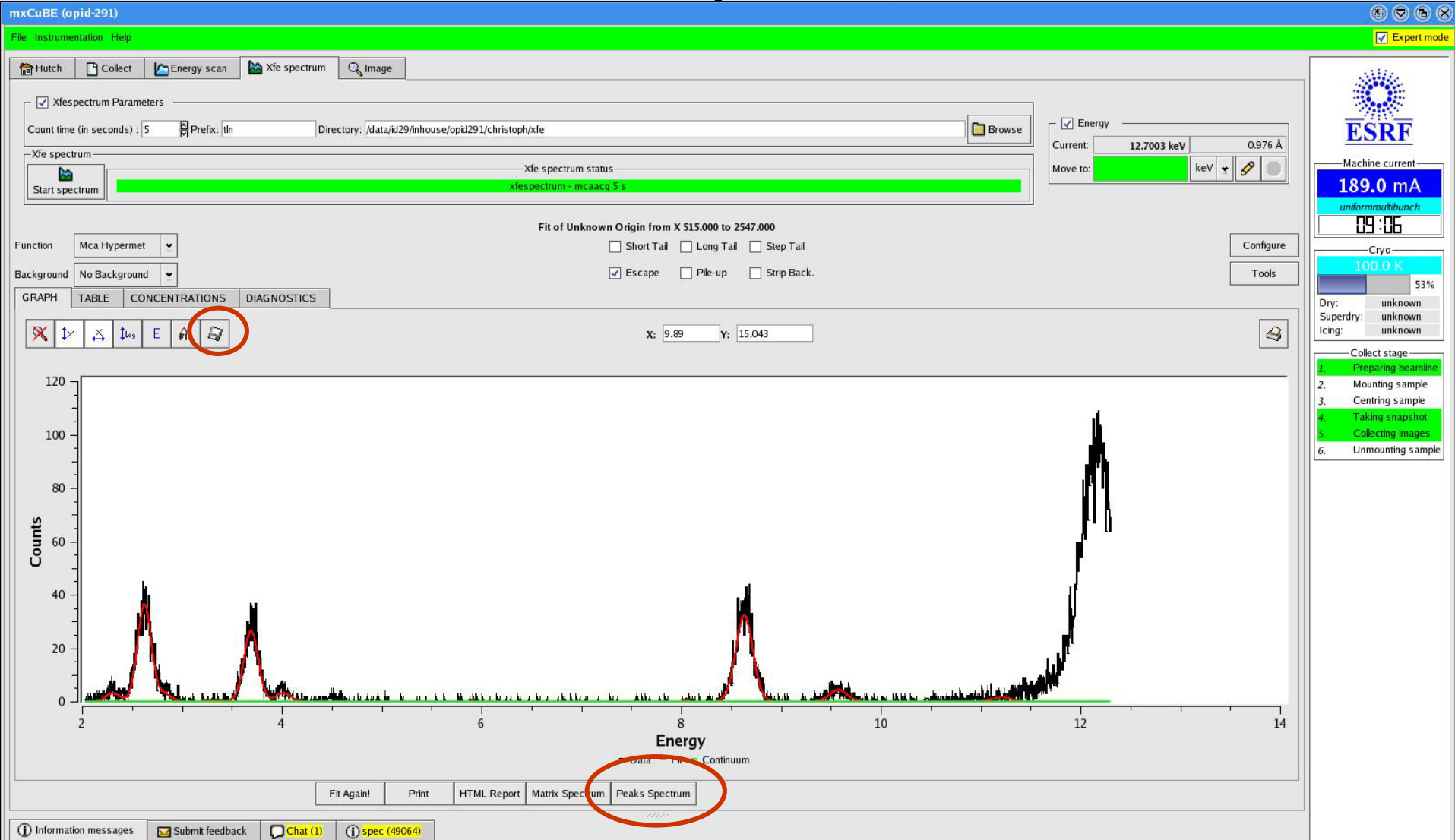
/data/id29/inhouse/opid291/christoph/xfe/tln\_30\_Oct\_2008\_03.png



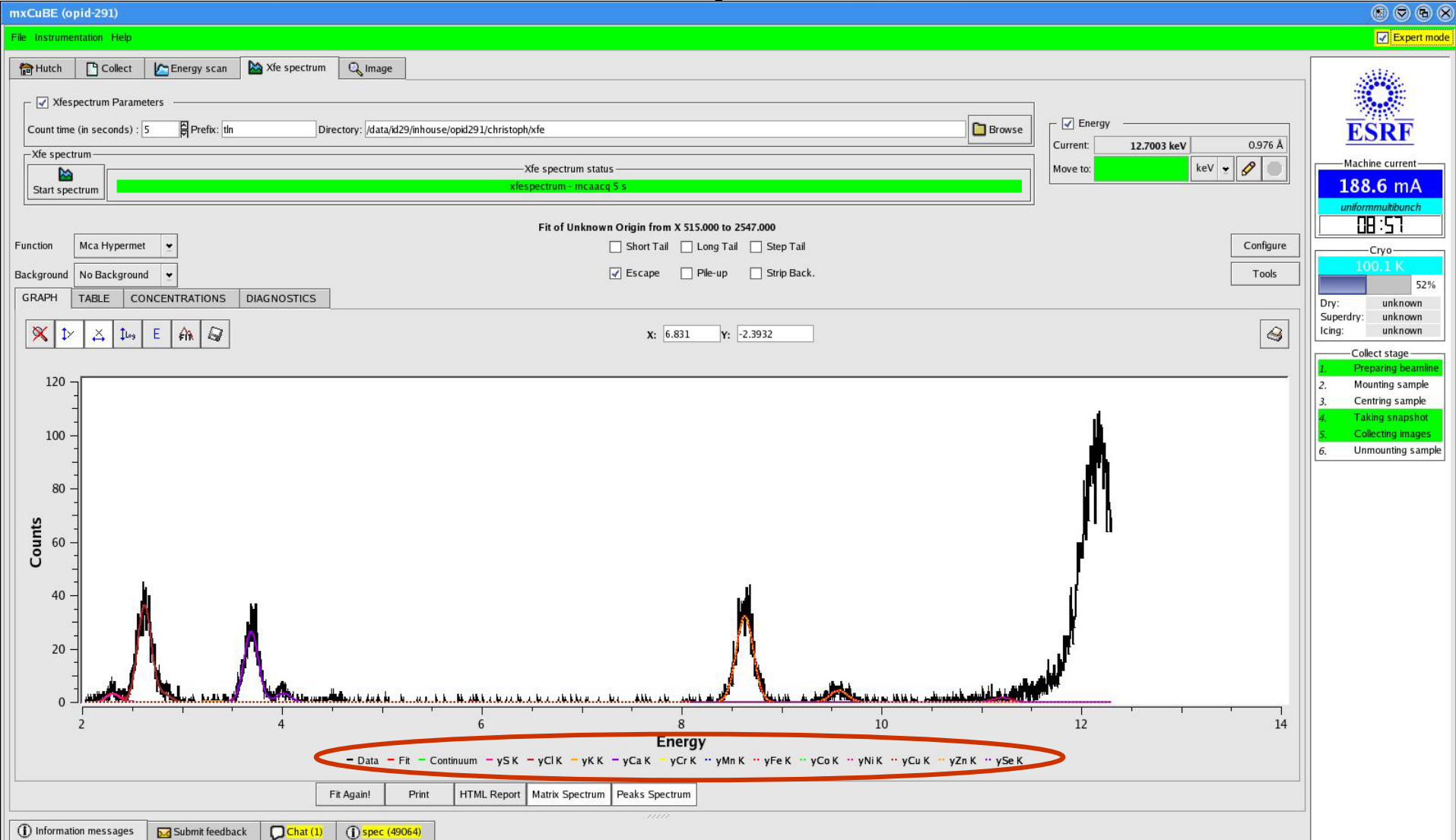
# XRF - spectra



# XRF - spectra



# XRF - spectra



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

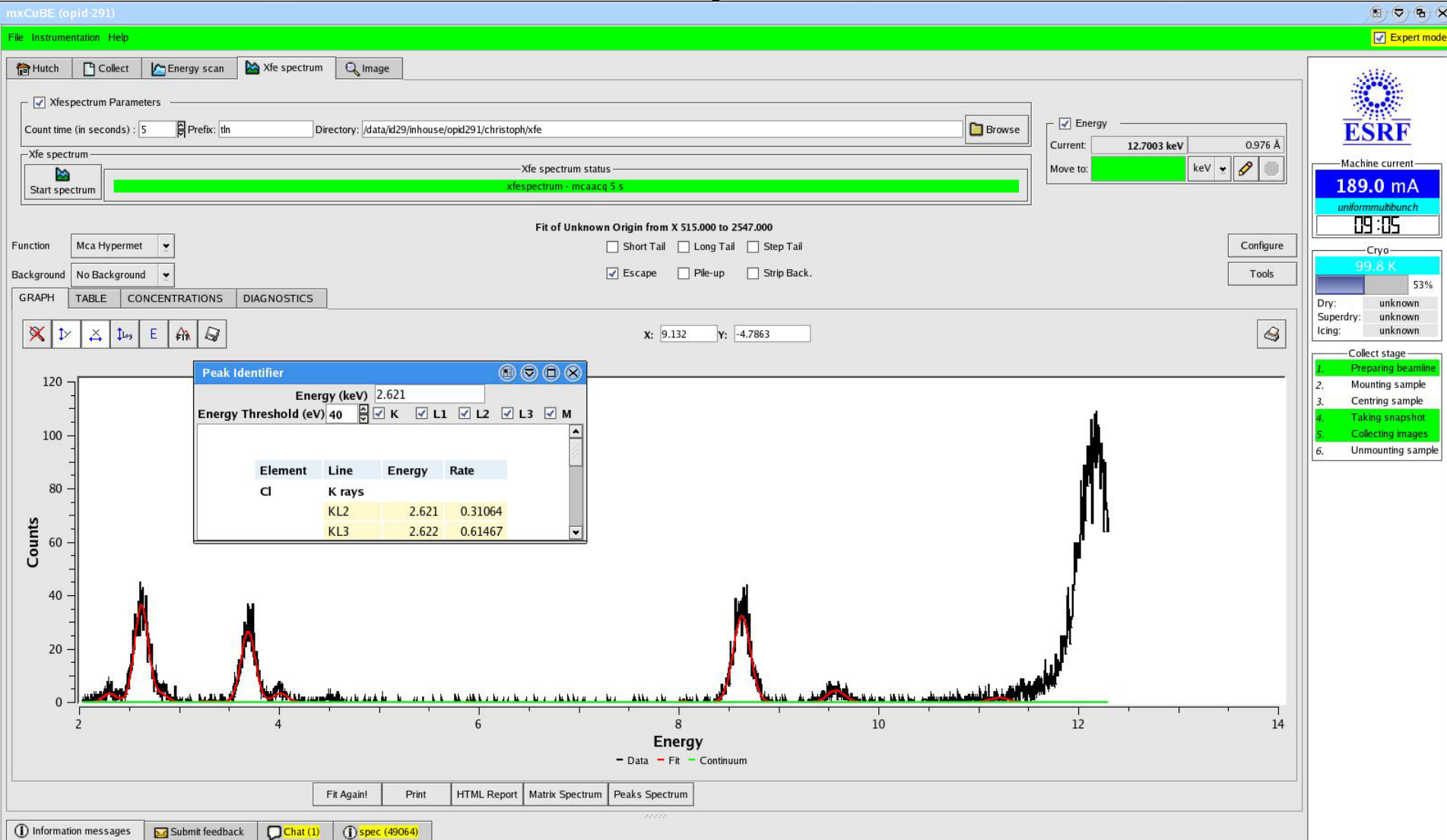
4. Taking snapshot

5. Collecting images

6. Unmounting sample

Current users

# XRF - spectra



Machine current

189.0 mA

uniformmultibunch

09:05

Cryo

99.8 K

53%

Dry:

unknown

Superdry:

unknown

Icing:

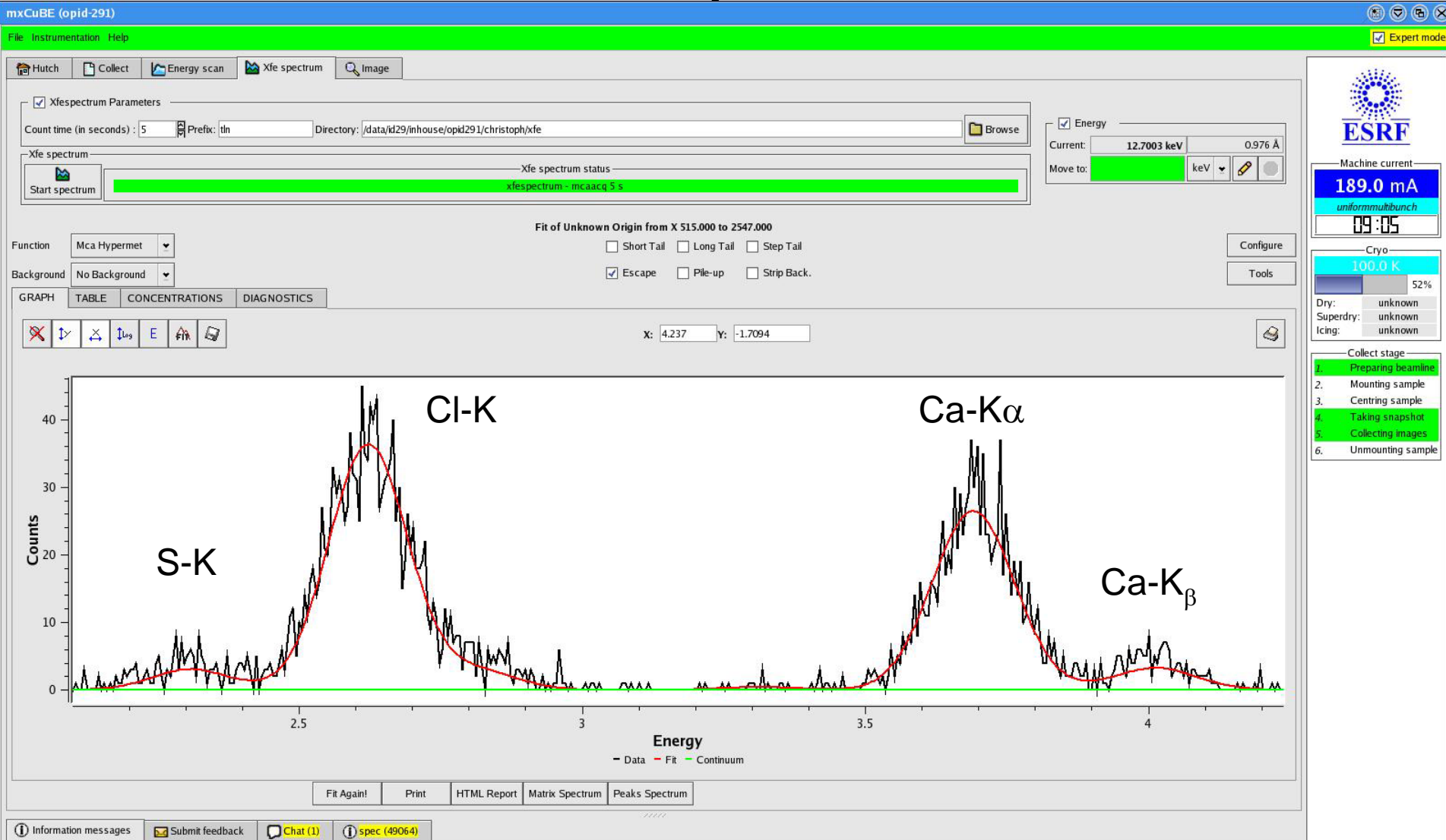
unknown

Collect stage

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

Current users

# XRF - spectra



# XRF - spectra



# XRF - spectra

mxCuBE (opid-291)

File Instrumentation Help

☒ Hutch 
 ☐ Collect 
 ☐ Energy scan 
 ☒ Xfe spectrum 
 ☐ Image

☒ Xfespectrum Parameters

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

☒ Energy

Current: 12.7003 keV 0.976 Å  
 Move to:  keV

Xfe spectrum status: xfespectrum - mcaacq 5 s

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

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

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



Machine current

188.9 mA

uniformmultibunch

09:04

Cryo

99.8 K

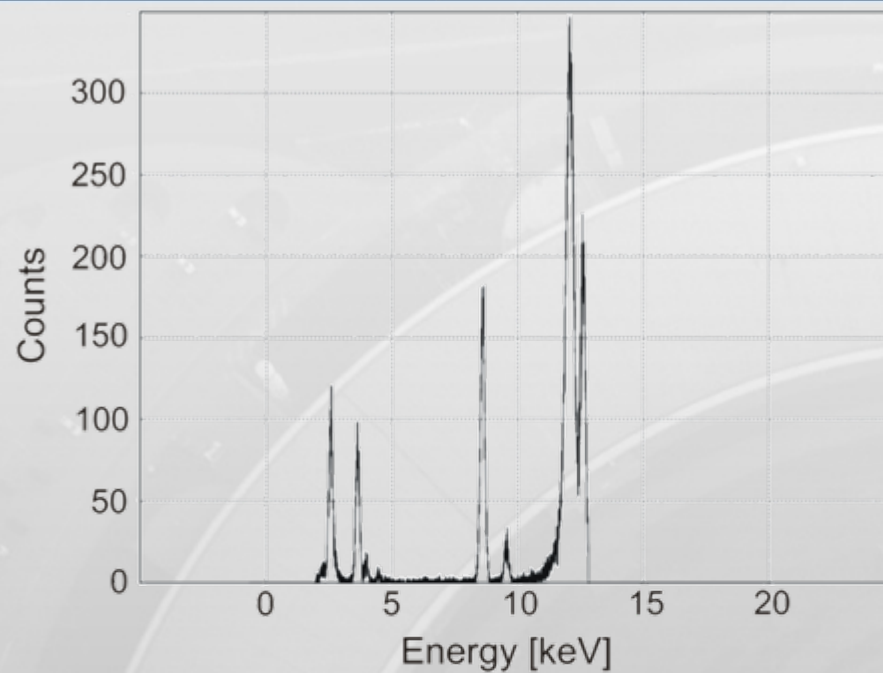
52%

Dry: unknown  
Superdry: unknown  
Icing: unknown

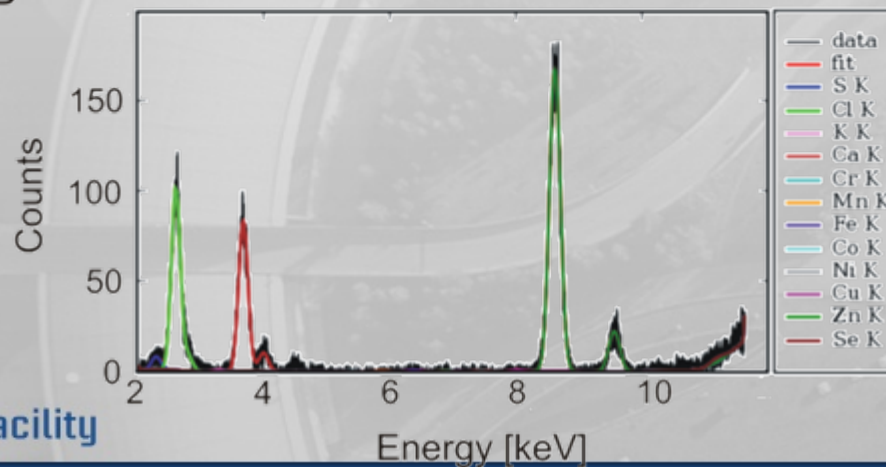
Collect stage

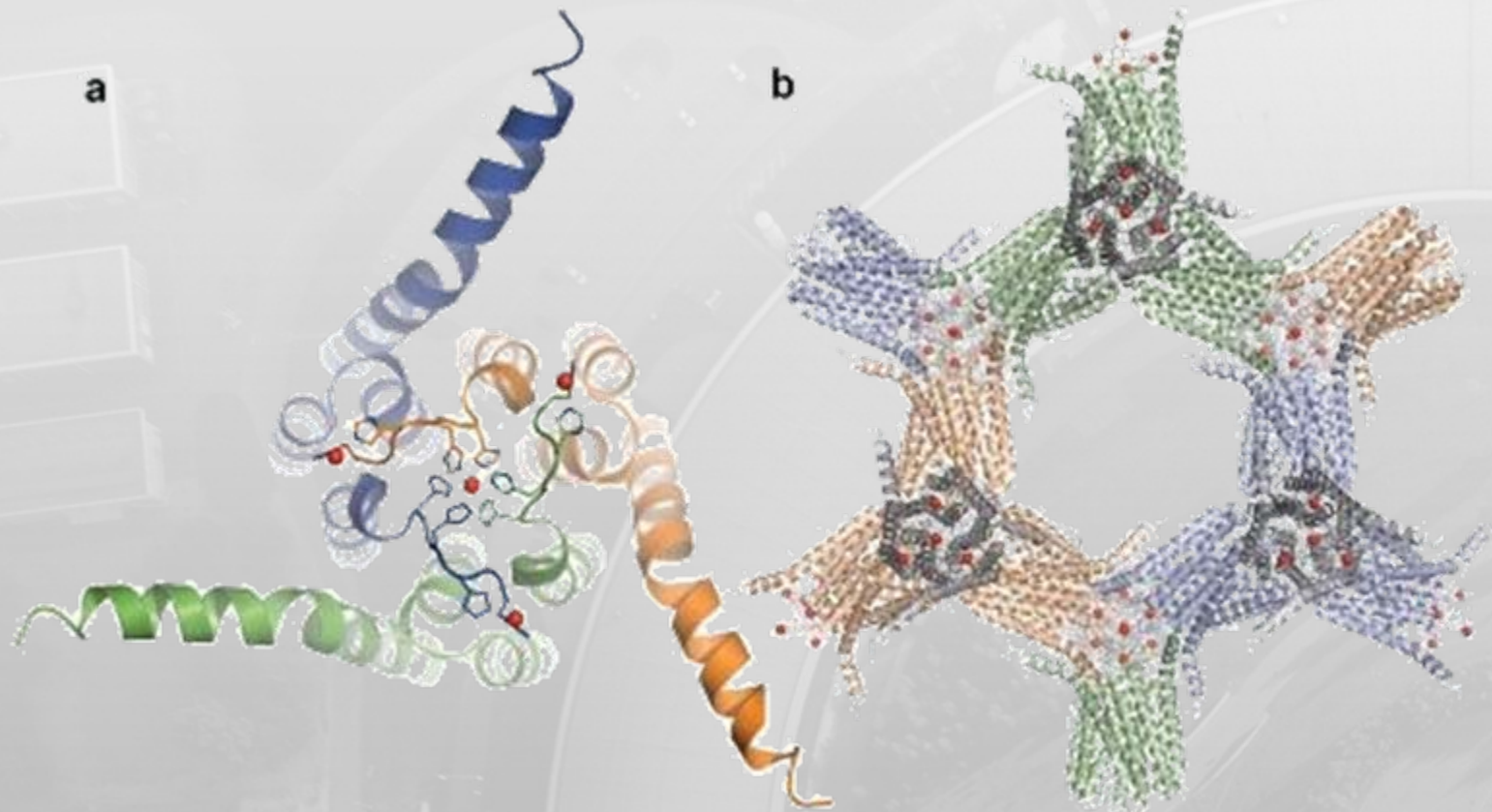
- Preparing beamline
- Mounting sample
- Centring sample
- Taking snapshot
- Collecting images
- Unmounting sample

Current users



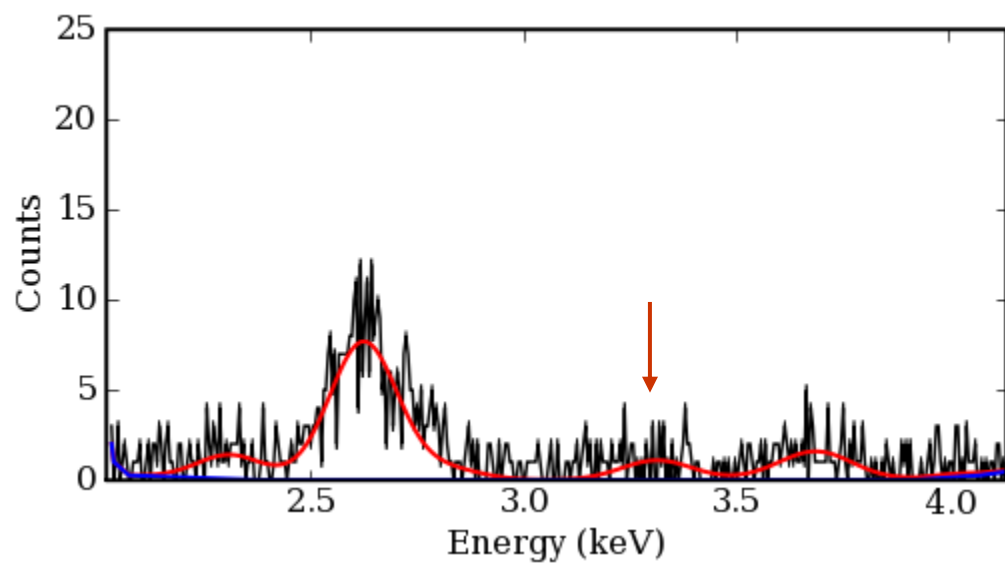
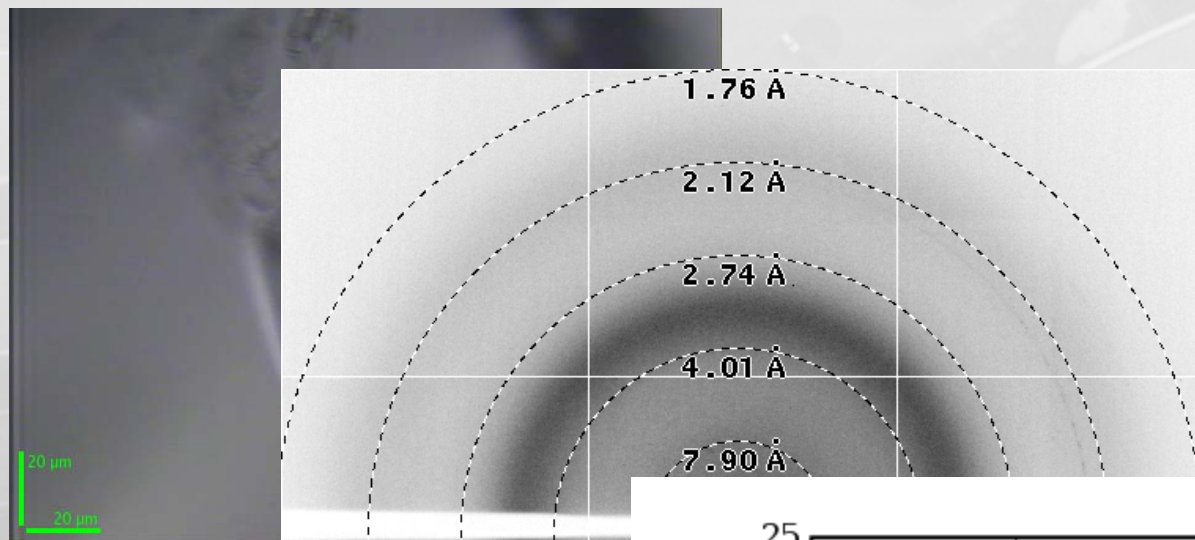
B



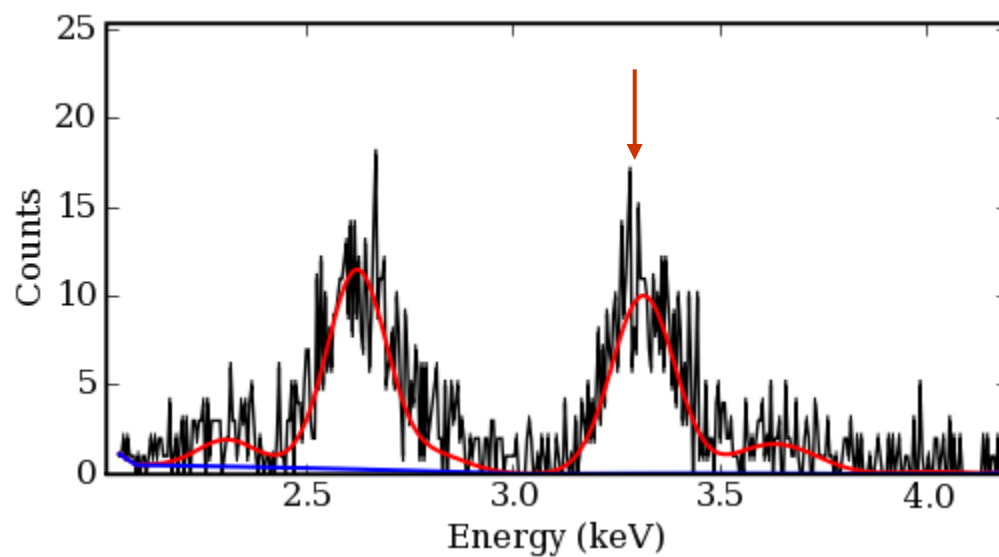
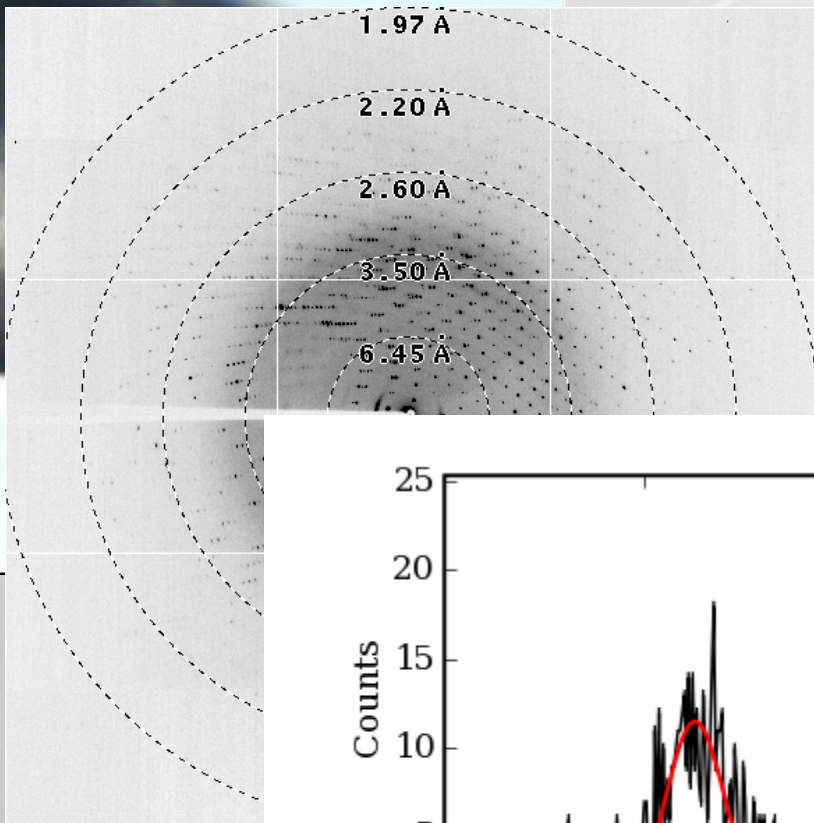
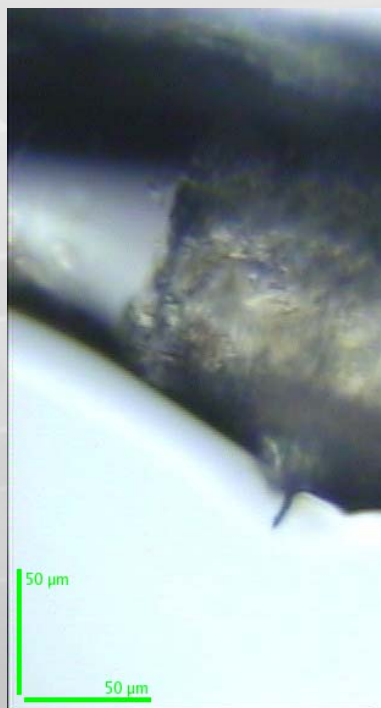


Daniel Martinez Molina *et al.*, (2007) Structural basis for synthesis of inflammatory mediators by human leukotriene C4 synthase. *Nature* 448, 613-616

# XRF - spectra



# XRF-spectra



# Conclusion

- Quick
- High sensitivity
- Useful with
  - I) crystallisation
  - II) new projects
  - III) rapid check for HA presence
- Completely integrated into mxCuBE
- Easy to use / user friendly

publication in March edition of JAC