

# SC - 5024

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## 1. Summarize of the exp:

Is described here the essential information needed to use the data. You may find the complete experimental report below which is a little messy since we met several pbm during our work.

We performed simultaneous SAXS-WAXS measurements for every sample from Group1, 2, 3 and 4. The temperature range asked has been done for all of them except the Group1 for which the maximum reachable temperature was around 147°C due to the broken resistor on one side of the sample holder. Our problem of heating had two consequences : firstly to not be able to reach 160°C and secondly that the temperature values are not exactly the ones asked and mentioned in the header of the files and summarized in the file Temperature\_Lyu (measured temperature). In order to estimate the real temperatures (Treal), we have measured temperature in the first sample position S1 and last one S11 and compare them with set temperature. Temperature of the samples starting from 6th position up to 11th need to be corrected. The table below contains real temperature of the sample at the position 11 at the known set temperature. All others positions can be interpolated between set temperature and the T11.

Measured temp. Real Temp.

30.0	28.5
40.0	38.5
50.0	47.6
60.0	56.4
70.0	66.2
80.0	75.6
90.0	84.5
100.0	93.4
110.0	102.9
120.0	112.3
130.0	122.3

Data reduction has been done right now without subtraction of the empty cell. The names of the files are like for the first part of the experiment, for example :

Group1/21Feb25D5\_3500\_S1.dat. D5 corresponds to SAXS and WOS corresponds to WAXS. Files are classified in several folders named Group1, 2 and 3. For this example, 3500 corresponds to the run number. S1 corresponds to the first sample of your priority list being in the group 1. I wrote in red in the exp. report (below), the names of the samples, the associated names (S1, S2...) and the position in the sample holder for each group.

We also had some transmission measurement problems. You will see some curves, the first temperatures of the group 1 are shifted compared to the others. It is possible to correct that by applying a multiplicative factor. I could do that if you can not but in a first attempt, I prefer to deliver the data like this in order for you to know. This transmission pbm also creates difficulties to substrat the empty cell. the empty cell having a large pick around  $0.19 \text{ \AA}^{-1}$ , it is possible to estimate the multiplicative factor to apply to the empty cell intensity to eliminate this peak. One more time, I prefer to not do it in a first attempt. The empty cell run numbers are written in green in the exp. report.

## Experimental report :

This notebook corresponds to the second part of the SC-5024 experiment started in dec 2020.

The setup used is made with the new WOS (17cm long) plus the removable kapton holder allowing to measure the transmission and the beastop holder. A 5 cm of diameter kapton window is used just after the WOS to close the vaccum SAXS part.

calibration at 8 KeV

it worked fine for the D5 but for the WOS it looks like the statistics in the wos were not enough.

Zr fluoresces at 17.5kev. It is used for measuring the flat field.

loopscan 60 15 ---> for the D5

Name of the calibrations :

8keV\_fast\_20210224

for both detectors

```

Current Positions (user, dial)
  mono      moveh      courb      omega      gap      khimono      gamma      courbb
  mono      moveh      courb      omega      gap      khimono      gamma      courbb
7.098312  15.324179  1.029000  -12.710018  15.181250  -1.282410  -0.037127  1.028643
7.098306   9.317167  1.029007  -7.760018  -21.387900  -1.888077  -1.084423  1.028643

  courbf    M1_alpha    M1_khi    M1_bdam    M1_bdav    M1_ty    M1_tz    M2_alpha
  courbf    alpha1     khi1     bdam1     bdav1     ty1     tz1     alpha2
1.028643   0.220000    0.000000  193.407563  193.407563  17.500000  0.564297  0.191541
1.028643   0.992399    1.070700  193.407563  193.407563  17.500000  0.564297  -1.227658

  M2_khi    M2_bdam    M2_bdav    M2_ty    M2_tz    fluo1     rien     diode
  khi2     bdam2     bdav2     ty2     tz2     fluo1     rien     diode
-0.005999  251.296516  308.503599  17.500000  -0.621602  0.000000  1.000000  9.500000
-1.166281  220.039616  319.246699  17.500000  -0.621602  0.000000  1.000000  9.500000

  Slit1Up  Slit1Down  Slit1Front  Slit1Back  VertGap1  VertOffs1  HoriGap1  HoriOffs1
  su1      sd1         sf1         sb1         vg1         vol         hg1         ho1
-2.460250  0.479750  -0.763531  -1.236469  1.980499  1.469999  2.000000  -0.236469
-2.460250  0.479750  -0.763531  -1.236469  1.980499  1.469999  2.000000  -0.236469

  zcam      ycam
  zcam      ycam
7.382500  -9.524500
-7.382500  -9.524500

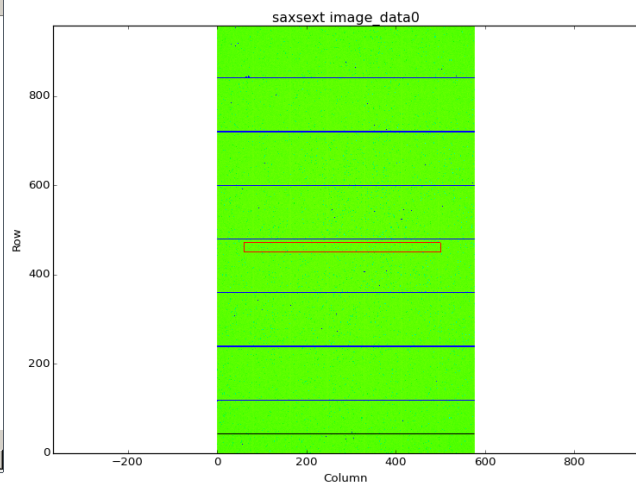
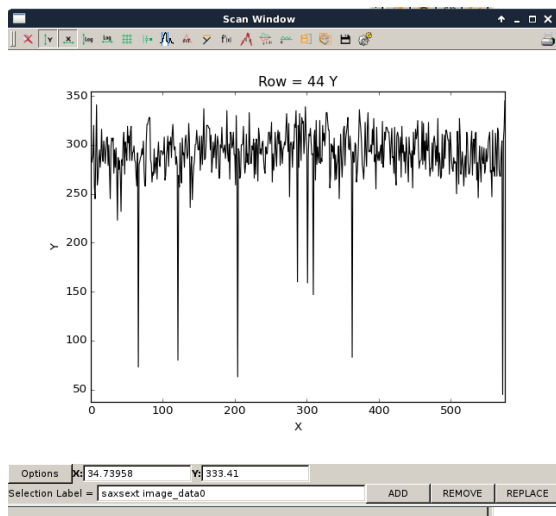
```

First images

Image Saved to File "/users/opd02/raw/21Feb24D5\_0360.edf"

Image Saved to File "/users/opd02/raw/21Feb24WOS\_0360.edf"

last images 419



New calibration for the WOS :

8keV\_fast\_20210224\_2 worse than 8keV\_fast\_20210224

We use 8keV\_fast\_20210224 for WOS

timescan for flat :

from /21Feb24WOS\_0561.edf to 1017




Measurements during the night (night2.mac):

for each sample, the first measurement is the one for measuring the transmission (kapton on), the second one is the real measure

1609-10	ZS41	1	60	T=30C
1611-12	ZS41Li	2	60	
	ZS11	3		missing
1613-14	ZS11Li	4	90	
1615-16	ZS21	5	60	
1617-18	ZS13	6	90	
1619-20	ZS12	7	60	
1621-22	ZS12Li	8	90	
1623-24	ZS1-07	9	60	
1625-26	ZS1-15	10	90	
1627-28	ZS31	11	60	

1657-76		1-11, no 3		T=40
1678-97		1-11, no 3		T =50
1699-1718		1-11, no 3		T=60
1720-1739		1-11, no 3		T=70
1741-60		1-11, no 3		T = 80
1762-81		1-11, no 3		T=90
1783-1802		1-11, no 3		T=100
1804-23		1-11, no 3		T=110
1825-44		1-11, no 3		T=120
1846-65		1-11, no 3		T=130
1867-86				T=130
1888-1907				T=125
1909-28				T=120
1930-49				T=115
1951-70				T=110
1972-91				T=105
1993-2012				T=100
2014-33				T=95
2035-54				T=90
2056-75				T=85
2077-96				T=80
2098-2117				T=75
2119-38				T=70
2140-59				T=65
2161-80				T=60
2182-2201				T=55
2203-22				T=50
2224-43				T=45
2245-64				T=40

2266-85				T=35
2287-2306				T=30
<p>Sample 3 has not been measured. It was missing in the program. I stopped the macro around 8am since it could not reach the last temperature 25C. it had started around 10pm last on thursday. it has been too quick.</p> <p>Temperature ramping rate was 50K/min which far to fast and could cause the problem with regulation.</p> <p>Moreover, positions of the sampleholder were not correct and not all samples has been measured.</p> <p style="text-align: center;"><b>Group 1</b></p>				
2405-2406	empty cap		only once, not in the script time = 90s transm 30s	T= 28
2407	C14	S2 in position 2		
08	C12	S3 in position 3		
09	C814	S4 in position 4		
10	C10	S5 in position 5		
11	C8	S6 in position 6		
12	C6	S7 in position 7		
13	IL	S8 in position 8		
		9		
14	SCN (from 4)	S10 in position 10		
<p>start script night2.mac: T min 30, Tmax 160. <b>Need to remove sample from pos 10 at 70C. Done!</b></p>				
2416-2423				T = 30
2424-2431				T = 40
2432-				T = 50
-2467(stop)				T = 60
<p>in the end of each temperature there is dscan gamma for 30 points.</p>				

2533-				T=60
-2596 (before dscan)				T = 70
2629- with closed shutter				T = 80
2783-				T = 80
				T = 90
				T = 100
				T = 110
				T = 120
				T = 130
-3104				T = 140
3107				T = 148
not possible				T = 160

I think the problem with temperature setpoint that the motion of nanodac is interrupted by other actions in the script, for example, moving sample table. Then the setpoint is taken from the actual temperature, which is higher due to overshooting.

```
#C----- temp ok
Sample 1: X=222      Z=33.8  NAME=Sample
Sample 2: X=208      Z=33.6  NAME=Sample
Sample 3: X=194      Z=33.5  NAME=Sample
Sample 4: X=180      Z=33.4  NAME=Sample
Sample 5: X=166      Z=33.3  NAME=Sample
Sample 6: X=152      Z=33.2  NAME=Sample
Sample 7: X=138      Z=33.1  NAME=Sample
Sample 8: X=124      Z=33      NAME=Sample
Sample 9: X=110      Z=32.9  NAME=Sample
Sample 10: X=96       Z=32.8  NAME=Sample
Sample 11: X=82       Z=32.8  NAME=Sample
Sample 11: X=82       Z=32.8  NAME=Sample
Motion aborted on motor 0, unit 2, "nanod".

Asked the motion to stop at the position 79.8102

sample=2 x=207.96 z=33.6
```

change PID from 4.7 - 95 - 20 to 5 - 95 - 20

### cooling

not possible				T = 155
not possible				150



				145
				140
				135
				130
				125
				120
				115
				110
				105
				100
				95
				90
				85
				80
				75
				70
				65
				60
				55
				50
				45
				40
				35
				30

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pbm of temperature regulation during the night. Marina do a new macro for the decrease in temperature, from 120C to 30C forst run of this macro =1419.

				120
				115
				110
				105
				100
				95
				90
				85
				80
				75
				70
4671-72 4683-84				65
4717-18 4729-30				60
4763-64 4775-76				55
4809-10 4820-21				45
4855-56 4867-68				40
4901-4902 4913-4914				35
4947-4948 ... 4959-4960				30

Saturday morning

I try a move in temp from 30 to 35 with PID (P=10). Since the red T increased slowly until 30 and stop, the orange one goes up to 36.3 with a quite large shift in time.

**Group 3**

5026-5027		transm 30 s meas 90 sec	empty cell	
5028		1	MXPD10	S1 position 2
5029		1	MXPD5	S2 position 3
5031		10	MXPD3	S3 position 4
5032		10	PD	S4 position 5
5033		10	SCN	S5 position 6

First temperature done manually at T=32C (macro group3-32C.mac)

5035-5036		5-90	MXPD10	
5037-5038		5-90	MXPD5	
5039-5040		5-90	MXPD3	
5041-5042		5-90	PD	
5043-5044		5-90	SCN	

for the higher temperature, macro : group3.mac

Increase :

5046-47 5054-55				T=40
5088-5089 5196-5097				50
5130-...				60
5172-...				70
5214-...				80

decrease

5228				75
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5242				70
5256				65
5270				60
5284				55
5298				50
5312				45
				40
				35
-5643				30

### Group 2

Group 2 30-130-30  
script group2.mac

5751-	ZS41	S1 position 1		30
	ZS41Li	S2 position 2		
	ZS11	S3 position 3		
	ZS11Li	S4 position 4		
	ZS21	S5 position 5		
	ZS13	S6 position 6		
	ZS12	S7 position 7		

	ZS12Li	S8 position 8		
	ZS1-07	S9 position 9		
	ZS1-15	S10 position 10		
	ZS31	S11 position 11		

5805-5857			changed T ramp to 1C/min	40
				50
				60
				70
				80
				90
				100
				110
				120
				130
				125
				120
				115
				110
				105
				100
				95
				90
				85

				80
				75
				70
				65
				60
				55
				50
				45
				40
				35
7392				30

Measure of references in order to determine the new WOS geometry.

7477	LaB6 0.7mm	90 s		
7478	Cr2O3 0.7mm	90s		
7479	LaB6 0.7mm	90 s		
7480	Cr2O3 0.7mm	90s		
7481	LaB6 1mm	90 s		less good too thick
7482	Cr2O3 1mm	90s		strange intensity of ring different from one module to another
7483	Cr2O3	90s		
7484	AgBe	90s		1.5 ou 2mm

7485	SrCl2 2M	90s		just to see for a flat but bump of
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				water on the wosna

one of the resistance of nanodac has been broken which did not let us go to the 160C. The thermocouple was between positions 5 and 6. So, positions 7-11 might not have the right temperature. probably there is a gradient of temperatures.