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Experiment Date:	2022 11 21
Duration (Days):	5
User:	Stjepko Fazinic, Iva Bozicevic Mihalic, Anja Miokovic
Accelerator:	Tandatron
Beam Line:	Old uProbe
Project:	Hi-REXS (HRZZ projekt)
Experiment Title:	Measuring HR spectra of Mg and Na + testing combination of PIXE mapping and HR PIXE
Beam:	2MeV H
Method:	HR PIXE, PIXE

SDD added to chamber.

Before measurement parallelity of crystal holder and chamber wall was checked. Also, one screw of crystal holder's motor was loose so it was tightened.

Beam deflection was connected to the horizontal deflector with -700 V.

GreatControl: X Binning = OFF, Y Binning = OFF, Readout Speed = 500 kHz, Gain = Max Sensitivity, Correct Bias = UNCHECKED,  $T_{ccd} = -70$  °C,  $T_{back} = 23$  °C, Chiller at 18 °C

### 21.11.2022.

Chamber positioned similar as it was in June. Shifted for ~5 mm, so that SDD is facing the beam spot.

Andro's slits were fully open so we closed them horizontally. At each side for 3 full turns.

Beam: 2 MeV H<sup>+</sup>

Focus: Me 15.6A, Ox 34.3A

Diffraction crystal: Beryl(1010) at 11.2 cm, with blue dot facing the main door

Current on metal before measurement ~ 2 nA

Samples: Mg (position 1), Mg+Ge (position 2), MgO+Ge (position 3)

FILE	t <sub>exp</sub> /s	Nframes	SAMPLE	COMMENTS
2211197	5	1	Mg	MgKa line is visible only at the righthmost part of the frame, most of the frame is inaccessible
2211198	5	1	Mg	moved to the other point on sample just to make sure that holder is not making shadow -> same thing, problem is in the geometry of the setup
2211199	5	1	Mg	geometry fixed: direction of putting crystal in chamber changed (and marked so that blue spot is facing main door) and crystal is placed in holder so that ~4 mm of it is peeking -> altogether crystal is ~5 mm closer to the beam spot; with Beryl at 11.2+ cm line is again at the rightmost part of the frame
2211200	5	1	Mg	Beryl at 11.75 cm and line is almost at the leftmost part of the frame; now only small left part of the frame is inaccessible so we are satisfied
2211201	5	1	Mg	Beryl at 11.4 cm, position of crystal we want to use for measuring Mg+Ge
2211202	5	10	DARK	
2211203	5	500	Mg+Ge	same position, I~2.5 nA
2211204	5	500	Mg+Ge	-    -, I~2-2.25 nA
2211205	5	250	Mg+Ge	-    -, I~2.25-2.5 nA

### 22.12.2022.

Beam: 2 MeV H<sup>+</sup>

Focus: Me 15.6A, Ox 34.3A (not changed from yesterday)

Diffraction crystal: Beryl(1010) at 11.4 cm, with blue dot facing the main door (same position as yesterday)

Current on metal before measurement ~ 2 nA

Samples: Mg (position 1), Mg+Ge (position 2), MgO+Ge (position 3)

FILE	t <sub>exp</sub> /s	Nframes	SAMPLE	COMMENTS
2211206	10	1	MgO+Ge	I~1.5 nA, position on the sample which is shining with very low intensity
2211207	8	10	DARK	
2211208	8	50	MgO+Ge	same position, I~2-1.5 nA; spectrum checked -> MgKa:GeLa=3:1, we want higher Ge
2211209	8	50	MgO+Ge	new position which shines with even lower intensity, I~1.75 nA; spectrum checked -> MgKa:GeLa=2:3, maybe we are good here
2211210	8	50	MgO+Ge	same position; spectrum from files 209+210: MgKa:GeLa=1:2, we are not satisfied -> it seems that after some time Ge count increases, maybe MgO is only on surface and we burn it?
2211211	6	1	MgO+Ge	new position which shines more than previous
2211212	6	10	DARK	
2211213	6	450	MgO+Ge	same position, I~1.7 nA; spectrum checked for first 70 frames -> MgKa:GeLa=3:1, but it seems we can't do better
2211214	4	10	DARK	current increased, so we reduced t <sub>exp</sub>
2211215	4	600	MgO+Ge	same position, I~2.75 nA

We analysed spectra from MgO+Ge and Mg+Ge.

Analysis of Mg+Ge spectrum gives E(GeLb) closer to the one gotten in June than in October. -> Conclusion: our geometry is similar to the one in June, we are satisfied

Energy shift of the MgKa12 line is almost nonexistent ~0.02 eV. :( Is our resolution not good enough to see shifts of MgKa12 line well?

Since we are not satisfied with results gotten from Mg compounds +Ge we move to Na samples.

Samples: NaCl (position 1), NaCl+Zn (position 2), NaSO<sub>4</sub> (position 3), NaBr (position 4)

From GUI Crystal code: To see NaKa12 line crystal should be moved for ~9 mm. -> Initial position of Beryl: 10.5 cm

FILE	t <sub>exp</sub> /s	N <sub>frames</sub>	SAMPLE	COMMENTS
2211216	6	50	NaCl	I~1.25 nA; when looking at frame in Vista we are not sure if we see line -> Matlab code analysis -> it's a line!
2211217	20	1	NaCl	Beryl at 10.55 cm, line is at channel ~550
2211218	6	10	DARK	
2211219	6	450	NaCl	same position, I~1.2 nA; with this shorter t <sub>exp</sub> we do not recognize line in Vista; energy window taken for Matlab analysis = [200,300]
2211220	6	450	NaCl	same position, I~1 nA
2211221	6	450	NaCl	-    -
2211222	6	75	NaCl	-    -

### 23.11.2022.

Beam: 2 MeV H<sup>+</sup>

Focus: Me 15.7A, Ox 34.4A

Diffraction crystal: Beryl(1010) at 10.55 cm, with blue dot facing the main door (same position as yesterday)

Current on metal before measurement ~ 2 nA (initially it was ~5 nA so the colimator slits were closed)

Samples: NaCl (position 1), NaCl+Zn (position 2), NaSO<sub>4</sub> (position 3), NaBr (position 4)

FILE	t <sub>exp</sub> /s	N <sub>frames</sub>	SAMPLE	COMMENTS
2211223	10	1	NaBr	line found in Vista at the same position as yesterday (channel ~550)
2211224	10	10	DARK	
2211225	10	300	NaBr	same position, I~1.3 nA
2211226	10	300	NaBr	same position, I is varying a lot: 0.8-1.4 nA, from plot I-t we see periodic oscillations, but also current decreases over time
2211227	10	300	NaBr	same position, to increase the current we opened colimator slits before continuing the measurement, I is varying: 1.2-1.7 nA
2211228	10	300	NaBr	same position, I~1.6-2.1 nA
2211229	10	300	NaBr	same position, I~1.8-2.3 nA
2211230	10	300	NaBr	-    -
2211231	10	300	NaBr	same position, I~1.3-1.8 nA

### 24.11.2022. Testing PIXE mapping + HR PIXE

Beam: 2 MeV H<sup>+</sup>

Focus: Me 16.3A, Ox 34.7A (final)

Scanning: x=10.0, y=9.55

Diffraction crystal: PET(002) at 11.4 cm (completely pushed in the folder)

Current on metal before measurement ~ 1 nA

Different holder in the chamber -> Samples: Cu mesh 400, Domagoj's sample 27608, Pb (chunk)

FILE	DETECTOR	SAMPLE	t <sub>exp</sub> /s	N <sub>frames</sub>	COMMENT
2211232	SDD	Cu grid 400	/	/	we are not satisfied with resolution, refocusing the beam
2211233	SDD	Cu grid 400	/	/	SS=0.5x1 (area ~0.5x0.5 mm <sup>2</sup> ), Fisher slits closed a bit -> I~0.3 nA on metal
2211234	SDD	Cu grid 400	/	/	-  -, checking after lunch -> still good
2211235	SDD	Cu grid 400	/	/	SS=5x0.1 (area ~0.4x0.4 mm <sup>2</sup> )
2211236	SDD	Sample 27608	/	/	SS=1x1, ROI maps added -> maps are upside down (flipped both horizontally and vertically) relative to the real situation

Before measuring HR PIXE of Pb we completely opened the Fisher slits to increase the current.

When measuring with both detectors at the same time we get 3 files (.raw from GreatControl, .dat+.lst from Spector) with the same name but in different folders - one connected with data from CCD and one with data collected with Spector.

FILE	DETECTOR	SAMPLE	t <sub>exp</sub> /s	N <sub>frames</sub>	COMMENT
2211237	CCD+SDD	Pb	60	1	I~0,6 nA, not seeing line in Vista
2211238	CCD	Pb	10	20	checking if we see anything in spectrum(energy window=[100,500]) ->no
2211239	CCD	Pb	30	1	moved closer to the sample with crystal, line visible at channel no~880
2211240	CCD	Pb	60	1	moved even closer with crystal, line visible at channel no~800
2211241	CCD	Pb	60	1	moving crystal closer and closer to check if the whole frame is available -> there is narrow unavaible region at the left part of the frame
2211242	CCD	Pb	60	1	PET at 11.7 cm -> position we want to work with, PbMa line at ch~600 and PbMb line at ch~850
2211243	CCD	Pb	10	150	same position, I~0.6-0.2 nA; from Matlab analysis -> energy window = [550,700]
2211244	CCD	Pb	10	150	-    -
2211245	SDD	Sample 27608	/	/	SS=1x1, SDD rate~3,5 kHz; the detector is pulled out of the chamber so that its housing is 10 cm away from the screw
2211246	CCD+SDD	Sample 27608 - ROI with high S+Pb counts	60	1	green line of the sample, in Vista we see only SKa12 line
2211247	CCD	-    -	10	1	we still see the line in Vista
2211248	CCD	DARK	10	10	
2211249	CCD	Sample 27608 - ROI with high S+Pb counts	10	30	spectrum analysed -> we see SKa3+SKa4 and also SKb

We want to make line scan that passes through different regions of sample.

Rectangle ROI that will be scanned can be defined with coordinates of its 4 vertices. Coordinates need to be written in .log file.

Files with coordinates are named: SCANPIXE\_no\_.log (where \_no\_ is specific file number) and one can find them on Old microprobe PC at path: C:\Spector\_v2\SCAN\ .

To define ROI to be scanned in Spector one needs to open Scan - History and then export ROI that corresponds to wanted .log file.

Beware: .log files must have numbers in ascending order and no number can be skipped -> only then Spector is able to load all files.

PROCESS: 1. Export .log file with wanted ROI.

2. Start Spector to record file with the same name.
3. Start GreatControl(CCD) to record files with the same name.
4. When CCD is done, stop Spector.

We decided to scan regions of  $3 \times 3 \text{ px}^2$  (we believe that is approximately the size of our beam spot) across the line. Starting pixel of each ROI is upper left vertex.

During whole time of experiment we measure current over time and we will export file containing  $I(t)$  after finishing with the experiment. To connect recorded values with particular ROIs we also keep track of approximate start time of ROI measurement.

FILE	DETECTOR	SAMPLE	$t_{\text{exp/s}}$	$N_{\text{frames}}$	ROI AREA + START PIXEL	START TIME	COMMENT
2211509	CCD+SDD	Sample 27608 - ROI	10	25	$3 \times 3 \text{ px}^2$ , (36,102)	17:06	
2211510	CCD+SDD	-    -	10	25	$3 \times 3 \text{ px}^2$ , (36,99)	17:18	
2211511	CCD+SDD	-    -	10	25	$3 \times 3 \text{ px}^2$ , (36,96)	17:24	Spector was accidentally stopped after frame 23
2211512	CCD+SDD	-    -	10	25	$3 \times 3 \text{ px}^2$ , (36,93)	17:29	
2211513	CCD+SDD	-    -	10	25	$3 \times 3 \text{ px}^2$ , (36,90)	17:37	
2211514	CCD+SDD	-    -	10	25	$3 \times 3 \text{ px}^2$ , (36,87)	17:43	
2211515	CCD+SDD	-    -	10	25	$3 \times 3 \text{ px}^2$ , (36,84)	17:50	
2211516	CCD+SDD	-    -	10	25	$3 \times 3 \text{ px}^2$ , (36,81)	17:59	
2211517	CCD+SDD	-    -	10	25	$3 \times 3 \text{ px}^2$ , (36,78)	18:05	
2211518	CCD+SDD	-    -	10	25	$3 \times 3 \text{ px}^2$ , (36,75)	18:10	
2211519	CCD+SDD	-    -	10	25	$3 \times 3 \text{ px}^2$ , (36,72)	18:22	current optimized before this measurement, $I \sim 1.3-1.5 \text{ nA}$ , SDD rate $\sim 3.3 \text{ kHz}$
2211520	CCD+SDD	-    -	10	25	$3 \times 3 \text{ px}^2$ , (36,69)	18:28	
2211521	CCD+SDD	-    -	10	25	$3 \times 3 \text{ px}^2$ , (36,66)	18:35	
2211522	CCD+SDD	-    -	10	25	$3 \times 3 \text{ px}^2$ , (36,63)	18:40	
2211523	CCD+SDD	-    -	10	25	$3 \times 3 \text{ px}^2$ , (36,60)	18:49	
2211524	CCD+SDD	-    -	10	25	$3 \times 3 \text{ px}^2$ , (36,57)	18:53	
2211525	CCD+SDD	-    -	10	25	$3 \times 3 \text{ px}^2$ , (36,54)	18:59	
2211526	CCD+SDD	-    -	10	25	$3 \times 3 \text{ px}^2$ , (36,51)	19:05	
2211527	CCD+SDD	-    -	10	25	$3 \times 3 \text{ px}^2$ , (36,48)	19:10	
2211528	CCD+SDD	-    -	10	25	$3 \times 3 \text{ px}^2$ , (36,45)	19:16	
2211529	CCD+SDD	-    -	10	25	$3 \times 3 \text{ px}^2$ , (36,42)	19:21	
2211530	CCD+SDD	-    -	10	25	$3 \times 3 \text{ px}^2$ , (36,39)	19:28	
2211531	CCD+SDD	-    -	10	25	$3 \times 3 \text{ px}^2$ , (36,36)	19:33	
2211532	CCD+SDD	-    -	10	25	$3 \times 3 \text{ px}^2$ , (36,33)	19:38	

File with  $I(t)$  recording saved as: I\_export\_24.11 in CCD\Data folder.

## 25.11.2022.

Beam: 2 MeV  $\text{H}^+$

Focus: Me 16.5A, Ox 34.6A

Scanning:  $x=10.0$ ,  $y=9.55$

Diffraction crystal: PET(002) at 11.4 cm

Current on metal before measurement  $\sim 0.7 \text{ nA}$  with colimator's slits closed a bit.

Same samples as yesterday: Cu mesh 400, Domagoj's sample 27608, Pb (chunk)

FILE	DETECTOR	SAMPLE	$t_{\text{exp/s}}$	$N_{\text{frames}}$	ROI AREA + START PIXEL	START TIME	COMMENT
2211533	SDD	Cu grid 400	/	/	/	/	SS=5x0.1; we are not satisfied
2211534	SDD	Cu grid 400	/	/	/	/	SS=5x0.1, Fisher slits closed a bit -> $I \sim 0.25$ on metal -> we are now satisfied with the resolution
2211535	SDD	Sample 27608	/	/	/	/	SS=1x1, Fisher slits opened before scanning the sample -> $I \sim 1.3 \text{ nA}$ on metal
2211536	CCD+SDD	Sample 27608 - ROI	8	120	$3 \times 3 \text{ px}^2$ , (23,75)	11:16	green line of the sample; $I \sim 1-1.2 \text{ nA}$ , SDD rate $\sim 3.2 \text{ kHz}$
2211537	CCD	DARK	8	10			
2211538	CCD+SDD	Sample 27608 - ROI	8	300	$3 \times 3 \text{ px}^2$ , (24,89)	11:43	black part of the sample; $I \sim 0.9-1.2 \text{ nA}$ , SDD rate $\sim 3.5 \text{ kHz}$ ; current fell during measurement
2211539	CCD+SDD	Sample 27608 - ROI	8	120	-    -	13:15	same ROI, $I \sim 0.9-1.4 \text{ nA}$
2211540	SDD	Sample 27608	/	/	/	/	SS=1x1, new map of the whole sample scanned, just to make sure that we are still on the same position

2211541	CCD+SDD	Sample 27608 - ROI	8	120	3x3 px <sup>2</sup> , (34,104)	13:45	blue line of the sample; I~0.9-1.3 nA, SDD rate~4.5 kHz
2211542	CCD+SDD	Sample 27608 - ROI	8	120	-    -	14:07	same ROI; I~0.8-1.3 nA, SDD rate~4.5 kHz
2211543	CCD+SDD	Sample 27608 - ROI	8	120	3x3 px <sup>2</sup> , (11,31)	14:33	orange line from the edge of the sample; I~1-1.4 nA, SDD rate~2.5 kHz
2211544	CCD+SDD	Sample 27608 - ROI	8	120	-    -	14:55	-    -
2211545	CCD+SDD	Sample 27608 - ROI	8	120	3x3 px <sup>2</sup> , (19,62)	15:34	orange line next to the green; I~0.9-1.3 nA, SDD rate~4.2 kHz

After recording file 2211538 I(t) on Faraday cup 3 was recorded. -> Recording is saved as file: I-t\_FARday\_cup3 in CCD\Data folder.

File with I(t) recording of the whole experiment saved as: I\_export\_25.11 in CCD\Data folder.

Attachment 1: [Mudronja's\\_sample\\_microscope\\_image\\_scanned\\_area.jpg](#) 4.122 MB Uploaded 11/28/2022 3:07:50 PM | [Hide](#) | [Hide all](#)

