Message ID: **193** Entry time: **Mon Dec 11 15:31:43 2023**

Experiment Date:	2023 12 11
Duration (Days):	5
User:	Anja Miokovic, Stjepko Fazinic, Iva Bozicevic Mihalic, Marija Tkalcevic
Accelerator:	Tandetron
Beam Line:	Old uProbe
Project:	Hi-REXS (HRZZ projekt)
Experiment Title:	RBS analysis of previously measured paint layers, HR PIXE of pure Na, HR PIXE of Al in standards
Beam:	2MeV H
Method:	HR PIXE, PIXE, RBS

Chamber positioned according to the black marks on the floor that were drawn in February.

PIXE: Coarse gain=1 k; Fine gain=3; Shaping time=2 us; covered with Al (1 mm thick) mask with hole of 2r=1.7 mm

RBS: Bias=+50 V; Coarse gain=200; Fine gain=4.85; Shaping time=1 us; covered with teflon mask (3 mm thick) with hole of 2r=3 mm

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 ºC, T_{back}= 23 ºC, Chiller at 18 ºC

Remark: **Maps in Spector are flipped both horizontally and vertically (rotated for 180 degrees)!**

11.12.2023.

Beam: 2 MeV H^+ TDT

Focus: Me 17.1 A, Ox 34.9 A (collimator slits very open, Ando's slits very closed for best focus)

Scanning: x=10.0, y=9.55

I~1.5 nA on the metal before the measurement

Sample 27608 on holder together with Cu mesh 400, quartz and scintillating paper.

Measuring only PIXE and RBS spectra today.

FILE	SAMPLE	COMMENT
2312074	Cu mesh 400	SS=5x0.1
2312075	Sample 27608	SS=0.7x1, finding similar position as the one we measured in February
2312076	Sample 27608 - ROI 1	high Ca; I~2.5 nA
2312077	Sample 27608 - ROI 2	high S+PbM, position corresponds to the position P1 from TechnArt poster; I~2-2.2 nA
2312078	Sample 27608 - ROI 3	Ti+BaL on green line, position corresponds to the position P3 from TechnArt poster
2312079	Sample 27608 - ROI 4	Ti+BaL on blue line, position corresponds to the position P2 from TechnArt poster
2312080	Al holder	I~2.2 nA; needed to calibrate RBS spectra

Sample 23511 was put instead of sample 27608.
It is not the same sample we measured in February, but it is from the same painting and should have the same composition.

FILE	SAMPLE	COMMENT
2312081	Sample 23511	SS=0.7x1
2312082	Sample 23511 - ROI 1	SS=0.7x1, selecting only part of map where the sample is, right part of sample - part with black pieces
2312083	Sample 23511 - ROI 2	SS= 0.35x1, zoomed in on central part of the sample; I~2.5 nA

2312084	Sample 23511 - ROI 3	from file 2312083 we selected position with high RBS count -> we believe it's black part
2312085	Sample 23511 - ROI 4	SS=0.35x1, left part of sample - part with orange line
2312086	Sample 23511 - ROI 5	from file 2312085 we selected position with high Hg count -> we believe it's orange part
2312087	Sample 23511 - ROI 6	from file 2312085 we selected position with high Pb count and low Hg count -> we are not sure to which color of the paint layer this corresponds

12.12.2023.

Beam: 2 MeV H^+ TDT

Focus: Me 17.4 A, Ox 34.8 A (collimator slits very open, Ando's slits very closed for best focus)

Scanning: x=10.0, y=9.55

Diffraction crystal: Beryl(1010)

I~4 nA on the metal before the measurement

Trying for the last time to get Na+Zn sample.
First we tried with nitrogen as inert gas, then with argon. It does not seem that we succeded, it oxidizises before we make vacuum in chamber.
We will scan it and collect PIXE and RBS spectra just to check.

FILE	SAMPLE	COMMENT
2312088	Na_2O+Zn	SS=1x1, I~4 nA
2312089	Na_2O+Zn - ROI 1	position from 2312088 map with no Zn -> we want to inspect RBS spectrum without Zn
2312090	Na_2O+Zn - ROI 2	position from 2312088 map with high Zn count -> inspecting change in RBS spectrum
2312091	Na_2O	SS=1x1, part of sample where there is no Zn, maybe above carbon tape
2312092	Na_2O - ROI 3	position from 2312091 map with max Na count -> it seems that it is thick layer of Na_2O

Sample 23521 was found.
Sample 23521 on holder together with Cu mesh 400, Pb chunk, quartz and scintillating paper.

I~ 3.5 nA on metal before measurement

Measuring only PIXE and RBS spectra.

FILE	SAMPLE	COMMENT
2312093	Cu mesh 400	SS=5x0.1
Andro's vertical slit closed for 10' each; I~2 nA on metal		
2312094	Cu mesh 400	SS=5x0.1
2312095	Sample 23521	same position as in February; I~1 nA, SS=5x0.1
2312096	Sample 23521 - ROI 1	position from 2312095 map on separated chunk of sample with high Hg and no Pb -> corresponds to the position P1 from TechnArt poster (red colour on the sample)
2312097	Sample 23521 - ROI 2	position from 2312095 map with high Pb and no Hg, and with not too high Ca -> corresponds to the position P2 from TechnArt poster (orange colour on the sample)
2312098	Sample 23521 - ROI 3	position from 2312095 map that corresponds to the position P3 from TechnArt poster (could be black colour on the sample)
2312099	Sample 23521 (new area)	SS=2.5x0.1, right part of the sample - part further away from separated part, we want to inspect thin black line on this part
2312100	Sample 23521 - ROI 4	position from 2312099 map that we hope is thin black line
2312101	Sample 23521 - ROI 5	position from 2312099 map that corresponds to red colour on sample
PIXE spectra of ROIs 2312100 and 2312101 seem very similar -> we are not sure if we can "hit" black part of the sample.		

We plan to measure black part on the sample 23511 because there it is much bigger than on the sample 23521.

13.12.2023.

Beam: 2 MeV H^+ TDT

Focus: Me 16.5 A, Ox 34.7 A

Scanning: x=10.0, y=9.55

Sample 23511 on holder together with Cu mesh 400, Pb chunk, quartz and scintillating paper.

I~1.2 nA on the metal before the measurement (after Andro's vertical slits were opened for 10' each)

FILE	DETECTOR	SAMPLE	t_{exp}/s	N_{frames}	COMMENT
2312102	SDD	Cu mesh 400	/	/	SS=5x0.1
2312103	SDD	Sample 23511	/	/	part of the sample where black region is, SS=7x0.1
2312104	SDD	- || -	/	/	part of the sample where black region is zoomed in a bit, SS=5x0.1
2312105	SDD, RBS	Sample 23511 - ROI 1	/	/	scanning only ROI from 2312104 map where sample is
2312106	CCD	Sample 23511 - ROI 2	10	1	position from 2312105 map with high Pb and no Hg -> we believe that is the most black part of the sample; PbMa line visible in Vista on ch~700
2312107	CCD	DARK	6	10	
2312108	SDD, RBS, CCD	Sample 23511 - ROI 2	6	63	from Matlab analysis we see that PbMb is at the right edge of the frame, stopped
Crystal moved a bit to the front. PbMa line on ch~640 in Vista.					
2312109	SDD, RBS, CCD	Sample 23511 - ROI 2	6	675	I~1.5 nA

Energy window used in Matlab analysis: [500, 700]

We want to inspect MIS lines of light metal in some compounds where its mass fraction is about 10%, to see if it is possible to do chemical speciation of metal of interest in such samples.

Decided to measure Al in 3 standards:
1. IAEA PT XRF 08 Natural soil -> w(Al)=15.2%
2. IAEA SL-1 Lake sediment -> w(Al)=8.9%
3. ISE 886 Riverclay -> w(Al)=4.5%

Samples: IAEA Natural soil (position 1), IAEA Lake sediment (position 2), ISE Riverclay (position 3)

Diffraction crystal: ADP(101) on 9.9 cm, peeking out of holder for 3 mm

I~5 nA on metal

FILE	DETECTOR	SAMPLE	t_{exp}/s	N_{frames}	COMMENT
2312110	SDD	IAEA Natural soil	/	/	SS=5x0.1 (~420x420 um^2), checking homogenity of the sample
2312111	SDD	- || -	/	/	SS=1x0.1 (~85x85 um^2), zoomed in from area scanned in 2312110
2312112	SDD	- || -	/	/	SS=0.2x0.1, again zoomed in (we are not sure if for scanned area this small dimensions reduce linerly) -> we conclude that the sample is homogeneous
2312113	CCD	IAEA Natural soil - ROI	2	1	position from 2312111 map, $AlKa_{1,2}$ line visible in Vista on ch~480
2312114	CCD	DARK	2	10	
2312115	SDD, RBS, CCD	IAEA Natural soil - ROI	2	900	I~4 nA
2312116	SDD	IAEA Lake sediment	/	/	SS=5x0.1 (~420x420 um^2), checking homogenity of the sample
2312117	SDD	- || -	/	/	SS=1x0.1 (~85x85 um^2), zoomed in from area scanned in 2312116 -> we conclude that the sample is homogeneous
2312118	CCD	DARK	4	10	
2312119	SDD, RBS, CCD	IAEA Lake sediment - ROI	4	900	position from 2312117 map, I~3-4 nA
2312120	SDD, RBS, CCD	- || -	4	300	

Energy window used in Matlab analysis: [320, 450]

14.12.2023.

Beam: 2 MeV H^+ TDT

Focus: Me 17.0 A, Ox 34.8 A

Samples: IAEA Natural soil (position 1), IAEA Lake sediment (position 2), ISE Riverclay (position 3)

Diffraction crystal: ADP(101) on 9.9 cm, peeking out of holder for 3 mm

I~2 nA on metal before measurement

FILE	DETECTOR	SAMPLE	t_{exp}/s	N_{frames}	COMMENT
2312121	SDD	ISE Riverclay	/	/	SS=5x0.1 (~420x420 um^2), checking homogenity of the sample -> it is heterogeneous!
2312122	SDD	ISE Riverclay	/	/	- || -; new position scanned by mistake - wanted to move for little but motors were not at speed=Very slow
2312123	SDD	ISE Riverclay	/	/	SS=1x0.1 (~85x85 um^2), zoomed in from area scanned in 2312122
2312124	CCD	ISE Riverclay - ROI	4	1	position from 2312123 map with low Si and high Al
2312125	CCD	DARK	3	10	
2312126	SDD, RBS, CCD	ISE Riverclay - ROI	3	720	I~4-3 nA (very unstable)
2312127	SDD, RBS, CCD	ISE Riverclay - ROI	3	1080	I~3 nA

Energy window used in Matlab analysis: [320, 450]

Beam: 3 MeV He^{2+} TDT

Focus: Me 20.5 A, Ox 42.9 A

Samples: IAEA Natural soil (position 1), IAEA Lake sediment (position 2), ISE Riverclay (position 3), NIST 620 Glass (position 4), quartz (position 5), Na_2SO_3 (position 6)

Diffraction crystal: Beryl(1010) on 10.5 cm, peeking out of holder for 3 mm

I~2.2 nA on metal before measurement

Measuring only HR PIXE spectra in this set of measurements. (using only CCD detector)

FILE	SAMPLE	t_{exp}/s	N_{frames}	COMMENT
2312128	Na_2SO_3	20	1	I~1 nA; $NaKa_{1,2}$ visible in Vista on ch~440, for this t_{exp} there is very little events in the frame
Andro's slits are opened to increase the current. -> on metal: I~6 nA, on Na_2SO_3: I~3.4 nA.				
2312129	Na_2SO_3	10	1	I~3.4 nA
2312130	DARK	8	10	
2312131	Na_2SO_3	8	360	I~3.4 nA -> around frame 105 Željko increased current to I~5.6 nA -> Andro's slits closed to reduce current to I~4-3.8 nA
2312132	Na_2SO_3	8	360	I~4-3.5 nA
2312133	Glass 620 (Na_2O)	20	1	I~4 nA, $NaKa_{1,2}$ not visible because of the high S background
2312134	DARK	10	10	
2312135	Glass 620 (Na_2O)	10	900	I~3.6 nA -> low number of events per frame (~75) so Andro's slits are opened to increase current -> I~5.4 nA

Energy window used in Matlab analysis: [200, 300]

15.12.2023.

Beam: 3 MeV He^{2+} TDT

Focus: Me 20.5 A, Ox 42.9 A

Samples: IAEA Natural soil (position 1), IAEA Lake sediment (position 2), ISE Riverclay (position 3), NIST 620 Glass (position 4), quartz (position 5), Na_2SO_3 (position 6)

Diffraction crystal: ADP(101) on 9.9 cm, peeking out of holder for 3 mm

I~4 nA on metal before measurement

Decided to measure Al in standard IAEA PT XRF 08 Natural soil (w(Al)=15.2%) also with He beam.

FILE	DETECTOR	SAMPLE	t_{exp}/s	N_{frames}	COMMENT
2312136	CCD	IAEA Natural soil	30	1	$AlKa_{1,2}$ visible in Vista on ch~640
2312137	CCD	IAEA Natural soil	10	1	I~1.5 nA, crystal moved to the front -> $AlKa_{1,2}$ visible in Vista on ch~490
2312138	CCD	IAEA Natural soil	10	1	I~3.2 nA
2312139	CCD	DARK	8	10	
2312140	CCD, SDD, RBS	IAEA Natural soil	8	480	I~3.2-3.5 nA; we forgot to stop Spector on time (beam was continous for some time and bias was removed from RBS detector)

Energy window used in Matlab analysis: [320, 450]

It was decided to use extra time to inspect TiKa region with our HR PIXE detector.

FILE	SAMPLE	t_{exp}/s	N_{frames}	COMMENT
2312141	Ti	5	1	$TiKa_{1,2}$ line visible in Vista on ch~415
2312142	DARK	5	10	
2312143	Ti	5	500	I~3.6 nA, beam was lost for some time
2312144	Ti	5	200	
2312145	TiO	8	360	I~3.7 nA
2312146	TiC	8	360	
2312147	Ti	5	400	2312147_0.raw was overwrited by mistake so it was deleted
2312148	TiO_2	8	360	

Energy window used in Matlab analysis: [1080, 1280]