LogBook

2024_07_22

Sample: #1 Cu-6Sm

Loading: 2.7 mg of sample in the capillary

Sample: 60%wt CuO, 40%wt 6Sm (calcinated at 500°C, 4%at. Zr, 6%at Sm, 90%at Ce)

Temperature calibration: To reach a sample temp. “T_sam”, you need to set a temperature “T_sp”

T_sp = (T_sam - 7.27) / 0.772

For T_sam= 200 °C; set T_sp= 249.7

For T_sam= 220 °C; set T_sp= 275.7

For T_sam= 240 °C; set T_sp= 301.6

For T_sam= 260 °C; set T_sp= 327.5

For T_sam= 280 °C; set T_sp= 353.4

For T_sam= 300 °C; set T_sp= 379.3

For T_sam= 320 °C; set T_sp= 405.2

For T_sam= 340 °C; set T_sp= 431.2

For T_sam= 360 °C; set T_sp= 457.1

For T_sam= 380 °C; set T_sp= 483.0

For T_sam= 400 °C; set T_sp= 508.9

For T_sam= 550 °C; set T_sp= 703.3

For T_sam= 600 °C; set T_sp= 768.1

ROI1: Ce L_3

ROI2: Sm L_3

ROI3: Cu K_α (this is irrelevant)

ROI4*: Zr* K_α

***ROI5**: Fe K_α*

Plot PFY (cps): ROIX/mcaLt (Lifetime of MCA detector)

Plot Transmission: Diode1/AS_IC (average of the Ionch) for Cu K-edge mostly

Plot Ref: ionch1

Probable errors:

- When you stop scan with CTRL+C, afterwards do “monomot3”, “mv monoh 25”, “fshopen” and go to the energy again “mv monoE edge”

-the ionization chamber (“ionch1”) was frozen and hat to be restarted with “Keithley_reset_6485”

- try “fshopen” if there is no signal

- if “ionch1” signal is too low (for example, at E>20keV), beamtstop check of the “eiger” function will not work. Thus, you do “unuse_beamtstop_check”

Good scans: Export these files

1 – Pristine RT (“001_Cu-6Sm_Pristine_RT”)

Temperature: 28°C

Flow: set-point at 5 mL/min Ar (after 3am, 0 mL/min)

Pressure: atmospheric pressure

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Comments
1	Eiger	18keV	RT	1	20 sec
2	Ref	Mo K-edge	RT	1	Mo reference (filter3 pos 14)
3-5	EXAFS	Zr K-edge	RT	1	Mode 4, ROI 4 (BEAMSTOP IN)
6-9	EXAFS	Zr K-edge	RT	1	Mode 4, ROI 4 (mz=-1.5, LOW SIGNAL)
10-12	EXAFS	Zr K-edge	RT	1	Mode 4, ROI 4 (mz=0, NO SIGNAL)
14	Eiger	18keV	RT	1	mz=0 (NO SIGNAL, not in the sample?)
15	Eiger	18keV	RT	1	mz=-1.5,10 sec (in the sample)
16	Ref	Cu K-edge	RT	1	
17	Ref	Cu K-edge	RT	1	filter3 16
18-21	NEXAFS	Cu K-edge	RT	1	Low intensity
22-23	NEXAFS	Cu K-edge	RT	1	Low intensity (to see the quality)
24	alignment	cr2ropi	RT	1	At Cu K-edge
28	Ref	Cu K-edge	RT	1	With new values of cr2ropi
29-30	Ref	Cu K-edge	RT	1	With new values of cr2roll
End of shift (3:00 am)					
Start of shift (7:39 am, 24/07/2024))					
32	Eiger	18keV	RT	1	10sec

33	Ref	Cu K-edge	RT	1	
34-35	NEXAFS	Cu K-edge	RT	1	Fluor. probably saturated (No filt.)
36	NEXAFS	Cu K-edge	RT	1	Filter Cu 0.034 mm
40	Ref	Fe K-edge	RT	1	
41-42	NEXAFS	Ce L_3-edge	RT	1	
43-44	NEXAFS	Ce L_3-edge	RT	1	After changing fluor. detector
45	Ref	Cu K-edge	RT	1	
46-47	NEXAFS	Cu K-edge	RT	1	Good transmission, fluor. signal is saturating
48-49	NEXAFS	Cu K-edge	RT	1	1 sec acq. (Fluor. signal is saturating)
50-51	NEXAFS	Cu K-edge	RT	1	Al 0.06 mm (Fluor. signal is saturating)
52-53	NEXAFS	Cu K-edge	RT	1	Mo 0.01 mm (Fluor. signal is saturating)
54-55	NEXAFS	Cu K-edge	RT	1	Al 0.2 mm Filter3=9 (Fluor. signal is good)
***Cu K-edge measurement*: Transmission is good w/o filters, Fluor. is good with Al 0.2 mm filter**					
77	Ref	Fe K-edge	RT	1	After realignment (Ivo magic)
78	NEXAFS	Ce L_3-edge	RT	1	Mode 2, 2 seconds per point / ABORT
80-85	NEXAFS	Ce L_3-edge	RT	1	Mode 2, 2 seconds per point
86-87	alignment	Sm L_3-edge	RT	1	
88-93	NEXAFS	Sm L_3-edge	RT	1	Mode 2, 2 seconds per point
94	Ref	Cu K-edge	RT	1	Abosorbes too much, not useful
95	Ref	Cu K-edge	RT	1	Filter3 = 22 (Cu 0.01 mm) NEW!
97-98	Ref Cu(0)	Cu K-edge	RT	1	Filter3 = 22 (Cu 0.01 mm), good reference
99-110	EXAFS	Cu K-edge	RT	1	Use Mode 4, Transmission (diode1)
111	Ref	Mo K-edge	RT	1	Energy calibration at 20keV
112-117	XANES	Zr K-edge	RT	1	Mode 2, 2 sec/point. Trans + Fluor.
118-120	alignment		RT	1	
122	Eiger	18keV	RT	1	10 sec
124	Eiger	18keV	RT	1	2 sec
125	Eiger	18keV	RT	1	60 sec
128	Ref	Cu K-edge	RT	1	Filter3 = 22 (Cu 0.01 mm)
End of measurement 18:05 (24/07/2024)					

2 – Activation (“002_Cu-6Sm_Activation_ramp”)

Temperature: 28°C – 400°C (T_sp= 508.9°C) with a ramp of 10°C/minute

Flow: 4 mL/min H2, 5 mL/min Ar (measured: 4.60 mL/min H2, 4.75 mL/min Ar)

8 minutes delay between injection and MS signal

Pressure: atmospheric pressure

Pressure “backpressure_contr2” is set at 0.3 bars (before it was 0.6 bars) so the gases in the massflow are constant

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments
2-3	alignment		RT	1		
4	Ref	Cu K-edge	RT	1		Filter3 = 22
5	NEXAFS	Cu K-edge	29°C	1	0.76	Mode 5, **start H2**

						flow, beamstop IN
6	NEXAFS	Cu K-edge	29°C	1	0.77	Mode 5
7	NEXAFS	Cu K-edge	29°C	1	0.77	Mode 5
8	NEXAFS	Cu K-edge	29°C	1	0.77	Mode 5
9	NEXAFS	Cu K-edge	29°C	1	0.77	Mode 5
10	NEXAFS	Cu K-edge	29°C	1	0.77	Mode 5, **start heating 10°C/min**
11	NEXAFS	Cu K-edge	53°C	1	0.78	Mode 5
12	NEXAFS	Cu K-edge	74°C	1	0.78	Mode 5
13	NEXAFS	Cu K-edge	105°C	1	0.78	Mode 5
14	NEXAFS	Cu K-edge	131°C	1	0.78	Mode 5
15	NEXAFS	Cu K-edge	170°C	1	0.78	Mode 5
16	NEXAFS	Cu K-edge	193°C	1	0.79	Mode 5
17	NEXAFS	Cu K-edge	222°C	1	0.79	Mode 5
18	NEXAFS	Cu K-edge	250°C	1	0.79	Mode 5, **starts reduction!**
19	NEXAFS	Cu K-edge	281°C	1	0.79	Mode 5
20	NEXAFS	Cu K-edge	314°C	1	19:04-19:07	Mode 5 (**ends at 327.5°C**,starts dwell)

3 – Activation stable (“003_Cu-6Sm_Activation_dwell”)

Temperature: 260°C (T_sp= 327.5°C) starts dwell at 19:07

Flow: 4 mL/min H2, 5 mL/min Ar (measured: 4.60 mL/min H2, 4.75 mL/min Ar)

8 minutes delay between injection and MS signal

Pressure: atmospheric pressure

Pressure “backpressure_contr2” is set at 0.3 bars (before it was 0.6 bars) so the gases in the massflow are constant

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments
1	eiger	18 keV	327.5°C	1	19;13	60 sec
3	Ref	Cu K-edge	327.5°C	1	19;22	Filter3 = 22
4-7	EXAFS	Cu K-edge	327.5°C	1	19;25	Mode 4
8	Ref	Fe K-edge	327.5°C	1	19;57	Filter3 = 19
9-14	NEXAFS	Ce L_3-edge	327.5°C	1	20;00	Mode 2
15	Ref	Cu K-edge	327.5°C	1	21;12	Filter3 = 22
16						WRONG
17-20	EXAFS	Cu K-edge	327.5°C	1	21;15	Mode 4
21	eiger	18 keV	327.5°C	1	21;50	60 sec

4 – Activation cooldown (“004_Cu-6Sm_Activation_CD”)

Temperature: From 260 to 200°C (T_sp= 249.7°C) starts at 22:14. Ends at 22:22.

Flow: 9 mL/min Ar (measured: X mL/min Ar)

10°C/min de rampa de enfriamiento

Pressure: atmospheric pressure

Protocol: eigerloopscan 60 10 (60 scans a 10 segundos por scan)

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments
1	Ref	Cu K-edge	327.5°C	1		Filter3 = 22, Wrong
					22;08	Ar flow to 9 ml/min
2-43	eigerloops	18 keV	327.5-249.7°C	1	22;13	10°C/min

5 – Reaction T1=200°C (“005_Cu-6Sm_Reaction_T1”)

NOTA: problema con las presiones. El MFC del CO2 no aguanta más de 4.06 bar de presión. Hasta que se arregle, se va a probar la reacción de metanación (P atmosférica-200-400°C de 50 en 50°C)

Temperature: 200°C (T_sp= 249.7°C) empieza a las 00:25

Flow: 4.69 mL/min H2*, 5 mL/min Ar, 1 mL CO2 (measured: 5.4 mL/min H2, 4.75 mL/min Ar, 1.35 mL/min CO2)

*Extrapolando

Pressure: 30 bar. Subida de P (10 bar/min) en 50 mL/min Ar (22:26-22:54). Después, cambio a flujos de reacción. FINALMENTE Patmosférica.

Pressure “backpressure_contr2” is set at 0.3 bars (before it was 0.6 bars) so the gases in the massflow are constant

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments
1	eiger	18 keV	249.7°C	1	00;30	60 sec
2	Ref	Cu K-edge	249.7°C	1	00;33	Filter3 = 22
3-6	EXAFS	Cu K-edge	249.7°C	1	00;41	Mode 4
7	Ref	Fe K-edge	249.7°C	1	1;16	Filter3 = 19
8-13	NEXAFS	Ce L_3-edge	249.7°C	1	1;31	Mode 2
14	Ref	Cu K-edge	249.7°C	1	2;15	Filter3 = 22
15-18	EXAFS	Cu K-edge	249.7°C	1	2;24	Mode 4
19	eiger	18 keV	249.7°C	1	3;02	60 sec

6 – Heating from 200 to 250°C (“006_Cu-6Sm_Ramp_T1T2”)

Temperature: From 200 to 250°C (T_sp= 249.7°C to 312.1°C) starts at 3:16. Ends at 3:22.

Flow: 4.69 mL/min H2*, 5 mL/min Ar, 1 mL CO2 (measured: 5.4 mL/min H2, 4.75 mL/min Ar, 1.35 mL/min CO2)

*Extrapolando

10°C/min de rampa de calentamiento

Pressure: atmospheric pressure

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments
1	Ref	Cu K-edge	249.7°C	1	3;05	Filter3 = 22
2-4	NEXAFS	Cu K-edge	249.7 to 312.1	1	3;16	10°C/min; inicio heating
5-6						WRONG

7 – Reaction T2=250°C (“007_Cu-6Sm_Reaction_T2”)

Temperature: 250°C (T_sp= 312.1°C) empieza a las 3:22

Flow: 4.69 mL/min H2*, 5 mL/min Ar, 1 mL CO2 (measured: 5.4 mL/min H2, 4.75 mL/min Ar, 1.35 mL/min CO2)

*Extrapolando

Pressure: P atmosférica

Pressure “backpressure_contr2” is set at 0.3 bars (before it was 0.6 bars) so the gases in the massflow are constant

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments
1	eiger	18 keV	312.1°C	1	3;33	60 sec
2	Ref	Cu K-edge	312.1°C	1	3;36	Filter3 = 22
3-6	EXAFS	Cu K-edge	312.1°C	1	3;48	Mode 4
7	Ref	Fe K-edge	312.1°C	1	4;21	Filter3 = 19
8-13	NEXAFS	Ce L_3-edge	312.1°C	1	4;28	Mode 2
14	Ref	Cu K-edge	312.1°C	1	5;15	Filter3 = 22
15-18	EXAFS	Cu K-edge	312.1°C	1	5;23	Mode 4. Beam caído (scan 18, 5;47)
19	eiger	18 keV	312.1°C	1	6;08	60 sec

8 – Heating from 250 to 300°C (“008_Cu-6Sm_Ramp_T2T3”)

Temperature: From 250 to 300°C (T_sp= 312.1°C to 379.3°C) starts at 6:24. Ends at 6:30.

Flow: 4.69 mL/min H2*, 5 mL/min Ar, 1 mL CO2 (measured: 5.4 mL/min H2, 4.75 mL/min Ar, 1.35 mL/min CO2)

*Extrapolando

10°C/min de rampa de calentamiento

Pressure: atmospheric pressure

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments
1	Ref	Cu K-edge	312.1°C	1	6;15	Filter3 = 22
2-4	NEXAFS	Cu K-edge	312.1 to 379.3	1	6;24	10°C/min; inicio heating

9 – Reaction T3=300°C (“009_Cu-6Sm_Reaction_T3”)

Temperature: 300°C (T_sp= 379.3°C) empieza a las 6;31

Flow: 4.69 mL/min H2*, 5 mL/min Ar, 1 mL CO2 (measured: 5.4 mL/min H2, 4.75 mL/min Ar, 1.35 mL/min CO2)

*Extrapolando

Pressure: P atmosférica

Pressure “backpressure_contr2” is set at 0.3 bars (before it was 0.6 bars) so the gases in the massflow are constant

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments
1	eiger	18 keV	379.3°C	1	6;40	60 sec
2	Ref	Cu K-edge	379.3°C	1	6;43	Filter3 = 22 ??
3	Ref	Cu K-edge	379.3°C	1	6;55	Filter3 = 22 ??
4-5	EXAFS	Cu K-edge	379.3°C	1	7;03	Mode 4 ??
6	Ref	Cu K-edge	379.3°C	1	7;08	Filter3 = 22
7-10	EXAFS	Cu K-edge	379.3°C	1	7;12	Mode 4
11	Ref	Fe K-edge	379.3°C	1	7;41	Filter3 = 19
12-15	NEXAFS	Ce L_3-edge	379.3°C	1	7;50	Mode 2
16-17	NEXAFS	Ce L_3-edge	379.3°C	1	8; 32	Mode 2
18	Ref	Cu K-edge	379.3°C	1	8;46	Filter3 = 22
19-22	EXAFS	Cu K-edge	379.3°C	1	8;54	Mode 4
23	eiger	18 keV	379.3°C	1	9;30	60 sec

10 – Heating from 300 to 350°C (“010_Cu-6Sm_Ramp_T3T4”)

Temperature: From 300 to 350°C (T_sp= 379.3°C to 444.4°C) starts at 9:4X. Ends at 9:4X.

Flow: 4.69 mL/min H2*, 5 mL/min Ar, 1 mL CO2 (measured: 5.4 mL/min H2, 4.75 mL/min Ar, 1.35 mL/min CO2)

*Extrapolando

10°C/min de rampa de calentamiento

Pressure: atmospheric pressure

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments
1	Ref	Cu K-edge	379.3°C	1	9;39	Filter3 = 22
2-4	NEXAFS	Cu K-edge	379.3 to 444.4	1	9;42	10°C/min ramp, mode 5
5-6	NEXAFS	Cu K-edge	444.4°C	1	9.54	**Change H_2 source** (small→big bottle) 16,0 h in MS

11 – Reaction T4=350°C (“011_Cu-6Sm_Reaction_T4”)

Temperature: 350C (T_sp= 444.4°C) empieza a las

Flow: 4.69 mL/min H2*, 5 mL/min Ar, 1 mL CO2 (measured: 5.4 mL/min H2, 4.75 mL/min Ar, 1.35 mL/min CO2)

*Extrapolando

Pressure: P atmosférica

Pressure “backpressure_contr2” is set at 0.3 bars (before it was 0.6 bars) so the gases in the massflow are constant

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments
1	eiger	18 keV	444.4°C	1	10;03	60 sec
2	Ref	Cu K-edge	444.4°C	1	10;10	Filter3 = 22, **BAD**!
3-5	EXAFS	Cu K-edge	444.4°C	1	10;14	Mode 4, ABORT
6-9	EXAFS	Cu K-edge	444.4°C	1	10;26	Mode 4, beamdump in last scan (point 122)
10	Ref	Cu K-edge	444.4°C	1	11;35	After injection, **BAD**!
11-14	EXAFS	Cu K-edge	444.4°C	1	11;38	
15	Ref	Fe K-edge	444.4°C	1	12;08	Filter3 = 19, **BAD**!
16-21	NEXAFS	Ce L_3-edge	444.4°C	1	12;14	Mode 2, ABORT 21
22-23	NEXAFS	Ce L_3-edge	444.4°C	1	13;01	Mode 2
24	Ref	Cu K-edge	444.4°C	1	13:15	Filter3 = 22, **BAD**!
27	Ref	Cu K-edge	444.4°C	1	13;40	Filter3 = 22
28-31	EXAFS	Cu K-edge	444.4°C	1	13;44	Mode 4
32	eiger	18 keV	444.4°C	1	14;23	60 sec

- **BAD** means that the ionization chamber (“ionch1”) was frozen and hat to be restarted with **“Keithley_reset_6485”**

12 – Heating from 350 to 400°C (“012_Cu-6Sm_Ramp_T4T5”)

Temperature: From 350 to 400°C (T_sp= 444.4°C to 508.9°C) starts at 14:XX. Ends at 15:XX.

Flow: 4.69 mL/min H2*, 5 mL/min Ar, 1 mL CO2 (measured: 5.4 mL/min H2, 4.75 mL/min Ar, 1.35 mL/min CO2)

*Extrapolando

10°C/min de rampa de calentamiento

Pressure: atmospheric pressure

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments
1	Ref	Cu K-edge	444.4°C	1	14;45	Filter3 = 22
2-5	NEXAFS	Cu K-edge	444.4 to 508.9	1	14;57	10°C/min ramp, mode 5
				1		

13 – Reaction T4=400°C (“013_Cu-6Sm_Reaction_T5”)

Temperature: 400C (T_sp= 508.9°C) empieza a las 15;08h

Flow: 4.69 mL/min H2*, 5 mL/min Ar, 1 mL CO2 (measured: 5.4 mL/min H2, 4.75 mL/min Ar, 1.35 mL/min CO2)

*Extrapolando

Pressure: P atmosférica

Pressure “backpressure_contr2” is set at 0.3 bars (before it was 0.6 bars) so the gases in the massflow are constant

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments
1	eiger	18 keV	508.9°C	1	15;13	60 sec
2	Ref	Cu K-edge	508.9°C	1	15;19	Filter3 = 22,
3-6	EXAFS	Cu K-edge	508.9°C	1	15;24	Mode 4
7	Ref	Fe K-edge	508.9°C	1	15;54	Filter3 = 19
8-13	NEXAFS	Ce L_3-edge	508.9°C	1	16;02	Mode 2
14-15	NEXAFS	Ce L_3-edge				Extra, don’t use
16	Ref	Cu K-edge	508.9°C	1	16;50	Filter3 = 22
17-20	EXAFS	Cu K-edge	508.9°C	1	16;53	Mode 4

21	eiger	18 keV	508.9°C	1	17;29	60 sec

14 – Cool down from 400 to 20°C (“014_Cu-6Sm_CD_T5RT”)

Temperature: From 400 to 20°C (T_sp= 25°C to 25°C) starts at 17:37. Ends at 19:29h.

Flow: 5 mL/min Ar (measured: 4.75 mL/min Ar)

10°C/min de rampa de calentamiento

Pressure: atmospheric pressure

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments
S1: 0-260	eiger	18keV	32	1	17;36	10 sec each scan; 17;37 starts cooldown

15 – Post Mortem at RT (“015_Cu-6Sm_PM_RT”)

Temperature: RT starts at 17:37. Ends at 00:28.

Flow: 5 mL/min Ar (measured: 4.75 mL/min Ar) IMPORTANT in scan 3 there is no more Ar flow. Dirk was working with the gases so we have our reactor in Ar but no flow.

Pressure: atmospheric pressure

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments
1	eiger	18 keV	30	1	18;34	60 sec
2	Ref	Mo k edge	30	1	18;37	Filter3 = 14
3-8	NEXAFS	Zr k edge	30	1	18;42	3 XANES, Mode 2
9	Ref	Cu	30	1	19;21	Filter3 = 22
9	Ref	Cu	30	1		Filter3 = 22
10-12	EXAFS	Cu K-edge				Measurement Stoped by mistake
13-24	EXAFS	Cu K-edge	30	1	19;31	3 EXAFS, Mode 4
25	Ref	Fe K-edge	30	1	21;00	Filter3 = 19
26-27	NEXAFS	Ce L_3-edge	30	1	21;06	3 NEXAFS, Mode 2
28-29						Checking ropi and roll counts are low for Ce
30-31	NEXAFS	Ce L_3-edge	30	1	21;26	3 NEXAFS, Mode 2,

						ERROR Lox counts again
32-37	NEXAFS	Ce L_3-edge, Fe ref				Several tests
45-50	NEXAFS	Ce L_3-edge	30	1	23;11	3 NEXAFS, Mode 2
51	Ref	Fe K-edge	30	1	23;55	Filter3 = 19
52-55	NEXAFS	Sm L_3-edge	30	1	00;01	3 NEXAFS, Mode 2
			End 25-7-2024 00:28			

2024_07_25

Sample: #2 CuFe-6Sm

Loading: 2-3 mg of sample in the capillary

Sample: 60%wt CuO, ????? CuFe???40%wt 6Sm (calcinated at 500°C, 4%at. Zr, 6%at Sm, 90%at Ce)

1 – Pristine RT (“001_CuFe-6Sm_Pristine_RT”)

Temperature: 28°C

Flow: set-point at 5 mL/min Ar (after 3am, 0 mL/min)

roi: 1: Ce; 2: Sm; 3: Cu; 4:Zr, 5:Fe

mz = - 1.68

Pressure: atmospheric pressure

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments
1	eiger	18 keV	30	1	00;55	60 sec
2	Ref	Cu	30	1	1;03	Filter3 = 22
3-5	EXAFS	Cu K-edge	30	1	1;06	Mode 4. Alinear
6	ropi		30	1	1;11	Para Cu
7	roll		30	1	1;15	Para Cu
8	Ref	Fe	30	1	1;17	Filter3 = 19, BAD
9	ropi		30	1	1;22	For Fe
10	roll		30	1	1;27	For Fe
11	Ref	Fe K-edge	30	1	1;29	Filter = 19
12-15	EXAFS	Fe k-edge	30	1	1;33	Mode 4, roi 5
16	Ref	Cu	30	1	2;08	Filter3 = 22
17-20	EXAFS	Cu K-edge	30	1	2;13	Mode 4
21	Ref	Fe K-edge	30	1	2;42	Filter3 = 19
22-27	NEXAFS	Ce L_3-edge	30	1	2;48	Mode 2
28	Ref	Fe K-edge	30	1	3;41	Filter3 = 19

2 – Activation (“002_CuFe-6Sm_Activation_ramp”)

Temperature: 28°C – 260°C (T_sp= 327.5°C) with a ramp of 10°C/minute. Starts at 3:52. Ends at 4:21

Flow: 4 mL/min H2, 5 mL/min Ar (measured: 4.60 mL/min H2, 4.75 mL/min Ar)

8 minutes delay between injection and MS signal

Pressure: atmospheric pressure

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments
1-10	NEXAFS	Fe K-edge	28 to 327.5°C	1	3;52	10°C/min ramp, mode 5

3 – Activation stable (“003_CuFe-6Sm_Activation_dwell”)

Temperature: 260°C (T_sp= 327.5°C) starts dwell at 4:22

Flow: 4 mL/min H2, 5 mL/min Ar (measured: 4.60 mL/min H2, 4.75 mL/min Ar)

8 minutes delay between injection and MS signal

Pressure: atmospheric pressure

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments
1	Ref	Cu	327.5	1	4;25	Filter3 = 22
2-5	EXAFS	Cu K-edge	327.5	1	4;28	Mode 4
6	Ref	Fe	327.5	1	4;58	Filter3 = 19
7-14	NEXAFS	Fe k-edge	327.5	1	5;05	Mode 2. scan18 (beam off 5;45-6;05)
15	Ref	Fe	327.5	1	6;09	Filter3 = 19. BAD
16	ropi		327.5	1	6;12	Para Cu. BAD
17	Ref	Fe	327.5	1	6;17	Filter3 = 19
18-19	NEXAFS	Fe k-edge	327.5	1	6;20	Mode 2. BAD
20-21	NEXAFS	Ce L_3-edge	327.5	1	6;35	Mode 2
22	ropi		327.5	1	6;45	Para Ce
23	roll		327.5	1	6;47	Para Ce
24-25	NEXAFS	Ce L_3-edge	327.5	1	6;49	Mode 2. Pocas cuentas
26	eiger	18 keV	327.5	1		60 sec
27	eiger	18 keV	327.5	1	7;16	60 sec; check evol.
28	eiger	18 keV	327.5	1	7;24	60 sec; check evol.

4 – Activation cooldown (“004_CuFe-6Sm_Activation_CD”)

Temperature: From 260 to 200°C (T_sp= 249.7°C) starts at . Ends at .

Flow: 9 mL/min Ar (measured: X mL/min Ar)

10°C/min de rampa de enfriamiento

Pressure: atmospheric pressure

Protocol: eigerloopscan 60 10 (60 scans a 10 segundos por scan)

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments
1-	eigerloops	18 keV	327.5-249.7°C	1	7;28	10°C/min

5 – Reaction T1=200°C (“005_CuFe-6Sm_Reaction_T1”)

Temperature: 200°C (T_sp= 249.7°C) empieza a las

Flow: 4.69 mL/min H2*, 5 mL/min Ar, 1 mL CO2 (measured: 5.4 mL/min H2, 4.75 mL/min Ar, 1.35 mL/min CO2)

*Extrapolando

Pressure: P atmosférica

Pressure “backpressure_contr2” is set at 0.3 bars (before it was 0.6 bars) so the gases in the massflow are constant

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments
1	eiger	18 keV	249.7	1	7;41	60 sec
2	Ref	Cu	249.7	1	7;45	Filter3 = 22
3-6	EXAFS	Cu	249.7	1	7;56	Mode 4
7	Ref	Fe	249.7	1	8;27	Filter3 = 19
8-13	NEXAFS	Fe K-edge	249.7	1	8;30	Mode 2
14-19	NEXAFS	Ce L_3-edge	249.7	1	9;11	Mode 2

6 – Heating from 200 to 250°C (“006_CuFe-6Sm_Ramp_T1T2”)

Temperature: From 200 to 250°C (T_sp= 249.7°C to 312.1°C) starts at 10;00:16. Ends at

Flow: 4.69 mL/min H2*, 5 mL/min Ar, 1 mL CO2 (measured: 5.4 mL/min H2, 4.75 mL/min Ar, 1.35 mL/min CO2)

*Extrapolando

10°C/min de rampa de calentamiento

Pressure: atmospheric pressure

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments
1	Ref	Cu K-edge	249.7°C	1	9;57	Filter3 = 22
2	Ref	Fe K-edge	249.7°C	1	10;03	Filter3 = 19
3-5	NEXAFS	Fe K-edge	249.7 to 312.1	1	10;08	10°C/min; inicio heating

7 – Reaction T2=250°C (“007_CuFe-6Sm_Reaction_T2”)

Temperature: 250°C (T_sp= 312.1°C) empieza a las 10;20

Flow: 4.69 mL/min H2*, 5 mL/min Ar, 1 mL CO2 (measured: 5.4 mL/min H2, 4.75 mL/min Ar, 1.35 mL/min CO2)

*Extrapolando

Pressure: P atmosférica

Pressure “backpressure_contr2” is set at 0.3 bars (before it was 0.6 bars) so the gases in the massflow are constant

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments
1	Ref	Cu	312.1	1	10;20	Filter3 = 22
2-5	EXAFS	Cu	312.1	1	10;23	Mode 4
6	Ref	Fe	312.1	1	10;53	Filter3 = 19
7-12	NEXAFS	Fe K-edge	312.1	1	10;57	Mode 2
13-18	NEXAFS	Ce L_3-edge	312.1	1	11;38	Mode 2
19	eiger	18 keV	312.1	1	12;30	60 sec

BIG PROBLEM WITH MASS FLOW OF CO2: SUDDENLY IT WENT TO 0 ML/MIN AND I HAD TO OPEN MORE THE VALVE OF THE GAS BOTTLE

THIS HAPPENED AROUND 11:40 and I didn’t noticed until 12:35, just before starting to ramp up to T2. Please consider that there was no CO2 for something like >40min

8 – Heating from 250 to 300°C (“008_CuFe-6Sm_Ramp_T2T3”)

Temperature: From 250 to 3000°C (T_sp= 312.1°C to 379.3 °C) starts at 10;00:16. Ends at

Flow: 4.69 mL/min H2*, 5 mL/min Ar, 1 mL CO2 (measured: 5.4 mL/min H2, 4.75 mL/min Ar, 1.35 mL/min CO2)

*Extrapolando

10°C/min de rampa de calentamiento

Pressure: atmospheric pressure

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments
1	Ref	Fe K-edge	312.1°C	1	12;35	Filter3 = 19
2-3	NEXAFS	Fe K-edge	312.1	1	12;45	Check after CO2 is there
5-8	NEXAFS	Fe K-edge	312.1 to 379.3	1	12;59	10°C/m heating, mode 4

9 – Reaction T3=300°C (“009_CuFe-6Sm_Reaction_T3”)

Temperature: 300°C (T_sp= 379.3°C) empieza a las 13;10

Flow: 4.69 mL/min H2*, 5 mL/min Ar, 1 mL CO2 (measured: 5.4 mL/min H2, 4.75 mL/min Ar, 1.35 mL/min CO2)

*Extrapolando

Pressure: P atmosférica

Pressure “backpressure_contr2” is set at 0.3 bars (before it was 0.6 bars) so the gases in the massflow are constant

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments
1	Ref	Cu	379.3	1	13;10	Filter3 = 22
2-5	EXAFS	Cu	379.3	1	13;14	Mode 4
6	Ref	Fe	379.3	1	13;48	Filter3 = 19
7-8	NEXAFS	Fe K-edge	379.3	1	13;51	Mode 2, abort 9
10-13	NEXAFS	Fe K-edge	379.3	1	14;08	Mode 2
14-19	NEXAFS	Ce L_3-edge	379.3	1	14;35	Mode 2
20-21	NEXAFS	Ce L_3-edge	379.3	1	15;22	Mode 6
22	eiger	18 keV	379.3	1	15;40	60 sec

Go to Argon while doing following operation:

BIG PROBLEM WITH MASS FLOW OF CO2: WRONG GAS BOTTLE

At 15:45, Dirk changed the CO2 bottle

10 – Heating from 300 to 350°C (“010_CuFe-6Sm_Ramp_T3T4”)

Temperature: From 300 to 350°C (T_sp= 379.3 °C to 444.4 °C) starts at 15:50. Ends at

Flow: 4.69 mL/min H2*, 5 mL/min Ar, 1 mL CO2 (measured: 5.4 mL/min H2, 4.75 mL/min Ar, 1.35 mL/min CO2)

*Extrapolando

10°C/min de rampa de calentamiento

Pressure: atmospheric pressure

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments
1	Ref	Fe K-edge	379.3°C	1	15;49	Filter3 = 19
2-	NEXAFS	Fe K-edge	379.3 to 444.4	1	15;54	In Ar (while changing CO2 bottle)
	NEXAFS	Fe K-edge	379.3 to 444.4	1		10°C/m heating, mode 4

12 – Heating from 300 to 350°C (“012_CuFe-6Sm_Ramp_T3T4B”)

Temperature: From 300 to 350°C (T_sp= 379.3 °C to 444.4 °C) starts at 16:50. Ends at 16:57

Flow: 4.69 mL/min H2*, 5 mL/min Ar, 1 mL CO2 (measured: 5.4 mL/min H2, 4.75 mL/min Ar, 1.35 mL/min CO2)

*Extrapolando

10°C/min de rampa de calentamiento

Pressure: atmospheric pressure

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments
1	Ref	Fe K-edge	379.3°C	1	16;41	Filter3 = 19, in 012_CuFe-6Sm_Ramp_T3T4
2-	NEXAFS	Fe K-edge	379.3 to 444.4	1	16;50	10°C/m heating, mode 4

13 – Reaction T4=350°C (“013_CuFe-6Sm_Reaction_T4”)

Temperature: 300°C (T_sp= 379.3°C) empieza a las 16;57

Flow: 4.69 mL/min H2*, 5 mL/min Ar, 1 mL CO2 (measured: 5.4 mL/min H2, 4.75 mL/min Ar, 1.35 mL/min CO2)

*Extrapolando

Pressure: P atmosférica

Pressure “backpressure_contr2” is set at 0.3 bars (before it was 0.6 bars) so the gases in the massflow are constant

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments
1	Ref	Cu	444.4	1	17;00	Filter3 = 22
2-5	EXAFS	Cu	444.4	1	17;03	Mode 4, Hubo por unos seg 90 ml/min en H2 en el reactor
6	Ref	Fe	444.4	1	17;34	Filter3 = 19
7-14	NEXAFS	Fe K-edge	444.4	1	17;37	Mode 6, 4 times
15-16	NEXAFS	Ce L_3-edge	444.4	1	18;31	Mode 6, 4 times, BAD no counts
17-18						Ropi and roll calibration for Ce
19-26		Ce L_3-edge	444.4	1	18;45	Mode 6, 4 times
27	eiger	18 keV	444.4	1	19;29	60 sec, parece que no lo guardo

14 – Heating from 350 to 400°C (“014_CuFe-6Sm_Ramp_T4T5”)

Temperature: From 350 to 400°C (T_sp= 444.4 °C to 508.9 °C) starts at 19:57. Ends at 20:05 (reached T)

Flow: 4.69 mL/min H2*, 5 mL/min Ar, 1 mL CO2 (measured: 5.4 mL/min H2, 4.75 mL/min Ar, 1.35 mL/min CO2)

*Extrapolando

10°C/min de rampa de calentamiento

Pressure: atmospheric pressure

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments
1	eiger	18Kev	444.4 °C	1	19;45	60 sec, parece que no guardó el anterior
2	Ref	Fe K-edge	444.4°C	1	19;53	Filter3 = 19
3-	NEXAFS	Fe K-edge	444.4 to 508.9	1	19;56	10°C/m heating, mode 4

15 – Reaction T4=400°C (“015_CuFe-6Sm_Reaction_T5”)

Temperature: 400°C (T_sp= 508.9°C) empieza a las 20;

Flow: 4.69 mL/min H2*, 5 mL/min Ar, 1 mL CO2 (measured: 5.4 mL/min H2, 4.75 mL/min Ar, 1.35 mL/min CO2)

*Extrapolando

Pressure: P atmosférica

Pressure “backpressure_contr2” is set at 0.3 bars (before it was 0.6 bars) so the gases in the massflow are constant

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments
1	Ref	Cu	508.9	1	20;07	Filter3 = 22
2-5	EXAFS	Cu	508.9	1	20;11	Mode 4,
6	Ref	Fe	508.9	1	20;46	Filter3 = 19
7-8	NEXAFS	Fe K-edge	508.9	1	20;45	Mode 6, 4 times, stop for calibration
9-11	Cr2ropi, cr2roll					Calibration for Fe with ionch1
12-19	NEXAFS	Fe K-edge	508.9	1	21;06	Mode 6, 4 times, maybe compare to 7-8 to see if the difference is due to reduction of Fe. Scan 17 tiene dos outsiders
20-27	NEXAFS	Ce L_3-edge	508.9	1	21;54	Mode 6, 4 times
28-29	NEXAFS	Ce L_3-edge	508.9	1	22;30	Mode 2, once
30	eiger	18 keV	508.9	1	22;52	60 sec

16 – Cool down from 400 to 20°C (“016_CuFe-6Sm_CD_T5RT”)

Temperature: From 400 to 20°C (T_sp= 25°C to 25°C) starts at 22:59. h.

Flow: 5 mL/min Ar (measured: 4.75 mL/min Ar)

10°C/min de rampa de calentamiento

Pressure: atmospheric pressure

	#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments
S1: 0-260		eiger	18keV	32	1		10 sec each scan; 22;59 starts cooldown

17 – Post Mortem at RT (“017_Cu-6Sm_PM_RT”)

Temperature: RT starts at 00:00.

Flow: 5 mL/min Ar (measured: 4.75 mL/min Ar) IMPORTANT in scan 3 there is no more Ar flow. Dirk was working with the gases so we have our reactor in Ar but no flow.

Pressure: atmospheric pressure

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments
1	eiger	18 keV	30	1	23;56	60 sec
2	Ref	Mo k edge	30	1	00;00	Filter3 = 14
3-4	NEXAFS	Zr k edge	30	1	00;02	3 XANES, Mode 2 . Stopped
5	Ref	Cu	30	1	00;10	Filter3 = 22, outsiders en EXAFS (scan9)
6-9	EXAFS	Cu K-edge	30	1	00;13	3 EXAFS, Mode 4 Se ve cómo se redujo respecto al pristino y sigue la forma del patron metalico
						Problems with beamline
16-20	EXAFS	Cu K-edge	30	1	01;05	3 EXAFS, Mode 4
21	Ref	Fe K-edge	30	1	1;29	Filter3 = 19
22-29	NEXAFS	Fe L_3-edge	30	1	1;36	4 NEXAFS, Mode 6
30-37	NEXAFS	Ce L_3-edge	30	1	2;21	4 NEXAFS, Mode 6
				End 27-7-2024		

2024_07_27

Sample: #3 Ru-CGO-ex

Plot PFY (cps): ROIX/mcaLt (Lifetime of MCA detector)

Plot Transmission: Diode1/AS_IC (average of the Ionch) for Cu K-edge mostly

Plot Ref: ionch1

Loading: 2-3 mg of sample in the capillary

Sample: $(Ce_{0.8}Gd_{0.2})_{0.94}Ru_{0.06}O_2$

Sinterizado: 700°C, 5h

Exsolución: 700°C, 4h, 5% H_2/Ar

1 – Pristine RT ("001_Ru-CGO-ex_Pristine_RT")

Temperature: 28°C

Flow: set-point at 9 mL/min Ar

roi: 1: Ce; 2: Sm; 3: Cu; 4:Gd, 5:Fe; 6:Ru

mz = -1.68 (scan 21 cambio a)

Pressure: atmospheric pressure

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments
5	Ref	Mo K edge	28	1	3;41	Filter3 = 14
6-9	EXAFS	Ru K edge	28	1	3;47	MIERDA
10	Ref	Mo K edge	28	1	4;02	Filter3 = 14
11-12	EXAFS	Ru K edge	28	1	4;10	MIERDA
13	Ref	Fe	28	1	4;40	BAD???
14	Ref	Mo K edge	28	1	3;41	Filter3 = 14. BAD
15	Acquire 10					BAD
16	Ref	Mo K edge	28	1	3;41	Filter3 = 14. BAD
22	Ref	Mo K edge	28	1	3;41	Filter3 = 14
26	Ref	Mo K edge	28	1	6;52	Filter3 = 14. BAD
35-36	Alignment	Ru K-edge	28	1	8;07	No filter, fhsopen
37	Capillary					
38	Mca det					
43-44	NEXAFS	Ru K-edge	28	1	8;45	
45-46	NEXAFS	Ru K-edge	28	1	8;55	
48-49	NEXAFS	Ru K-edge	28	1	9;05	New ROI6 (1830-1860 channels); 4 min
49-50	NEXAFS	Ru K-edge	28	1	9;09	ROI6 & 6 sec acq. (8 min)

56	eiger	Before edge	28	1	9;44	60 sec, 22.1 keV
57	eiger	At edge	28	1	9;47	60 sec, 22.16 keV
58	eiger	18 keV	28	1	9;54	10 sec
59	eiger	At edge	28	1	10;00	120 sec, 22.16 keV
60	eiger	After edge	28	1	10;07	60 sec, 22.3 keV
61	eiger	Before edge	28	1	10;10	60 sec, 22.1 keV
62-63	eiger	After edge	28	1	10;14	60 sec, 22.3 keV
64	eiger	At edge	28	1	10;00	60 sec, 22.16 keV

2 – Activation (“002_Ru-CGO-ex_Activation_ramp”)

Temperature: 28°C – 200°C (T_sp= 249.7°C) with a ramp of 10°C/minute. Starts at . Ends at

Flow: 4 mL/min H2, 5 mL/min Ar (measured: 4.60 mL/min H2, 4.75 mL/min Ar)

8 minutes delay between injection and MS signal

Pressure: atmospheric pressure

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments

3 – Methanation (“003_Ru-CGO-ex_Reaction_dwell”)

Temperature: 400°C (T_sp= 508.9°C)

Flow: 4.69 mL/min H2*, 5 mL/min Ar, 1 mL CO2 (measured: 5.4 mL/min H2, 4.75 mL/min Ar, 1.35 mL/min CO2) – METHANATION

8 minutes delay between injection and MS signal

Pressure: atmospheric pressure

#	Type	Scan info	Temp(T_{sp})	Pres (bar)	Time	Comments