



Supplement of

Spatially resolved sorption of Cm(III) on crystalline rock: influence of surface roughness and mineralogy

Maximilian Demnitz et al.

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Spatially resolved sorption of Cm(III) on crystalline rock: influence of surface roughness and mineralogy

Session 2F: Chemistry and migration behaviour of radionuclides

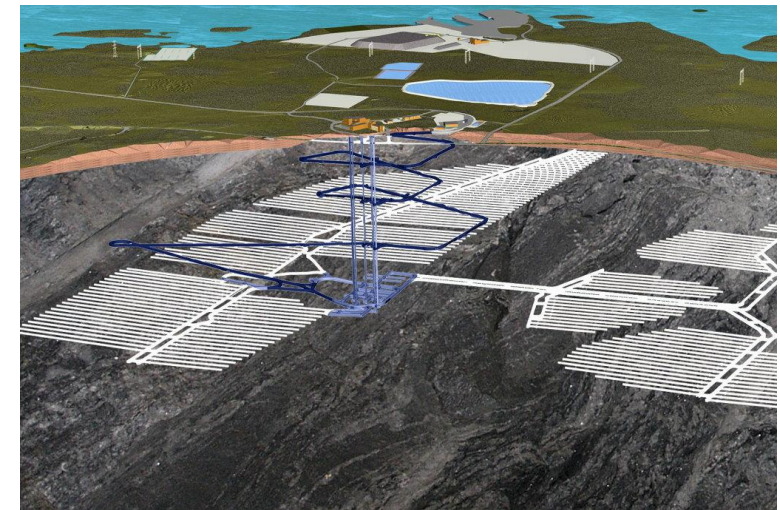
SafeND Workshop 11.11.2021

Maximilian Demnitz, Konrad Molodtsov, Stefan Schymura, Ariette Schierz, Katharina Müller,
Moritz Schmidt



Repositories for highly active nuclear waste

- Challenge for most countries worldwide: **highly active nuclear waste**
- **Safe deposition** of highly active nuclear waste for at least **100,000 to 1,000,000 years**
- Germany: 10,500 t or 27,000 m³
- Considered host rocks: salt, clay or **crystalline rocks**



<https://www.bge.de/de/abfaelle/aktueller-bestand/> Accessed: 18.06.2021

<https://sanjindumisc.com/wp-content/uploads/2013/12/onkalo-spent-nuclear-fuel-repository.jpg> Accessed: 18.06.2021

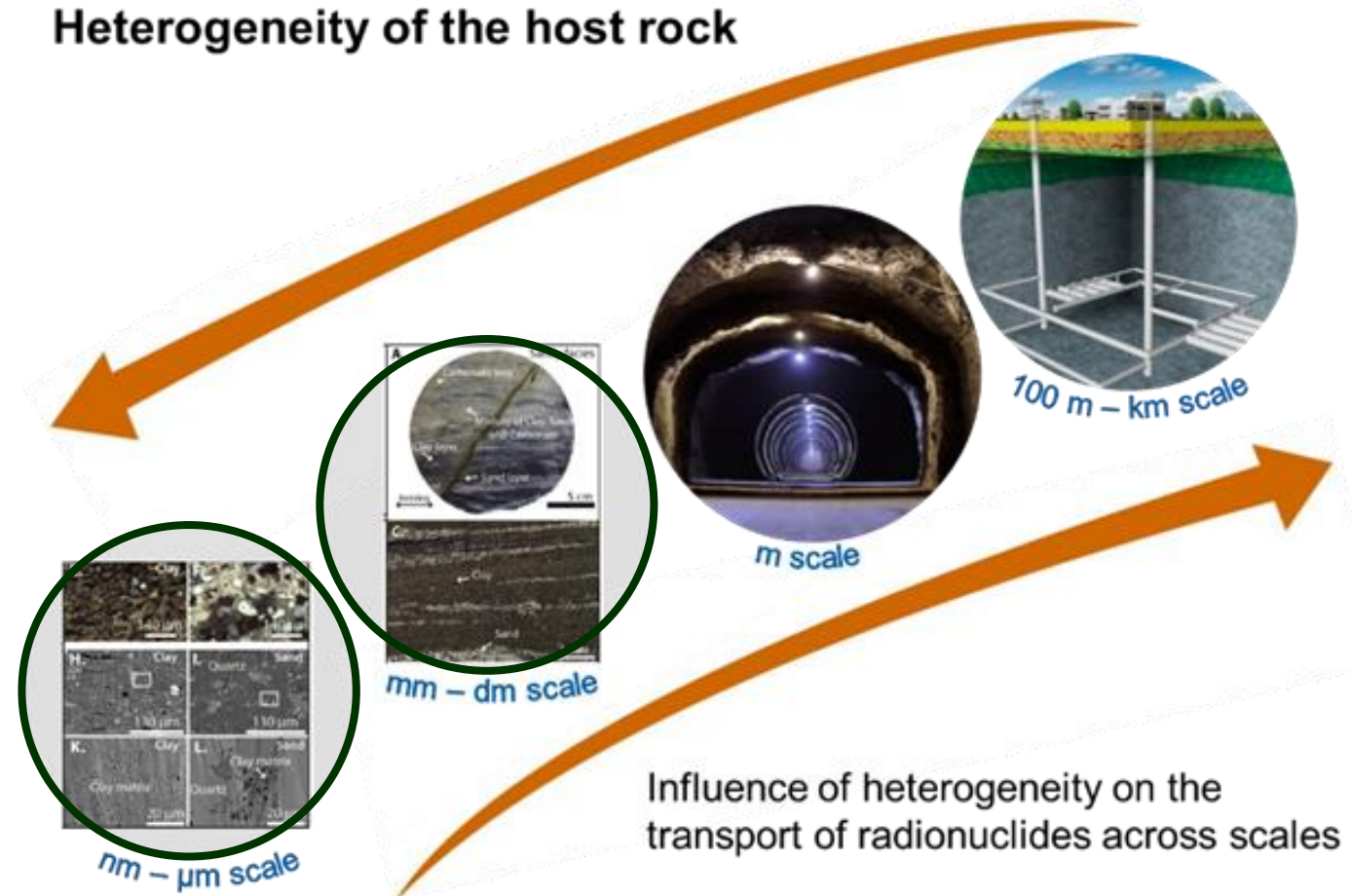
https://www.nsenergybusiness.com/wp-content/uploads/sites/3/2020/03/Image-3_Onkalo-Nuclear-Waste-Repository-Finland.jpg Accessed: 18.06.2021

Upscaling molecular scale findings

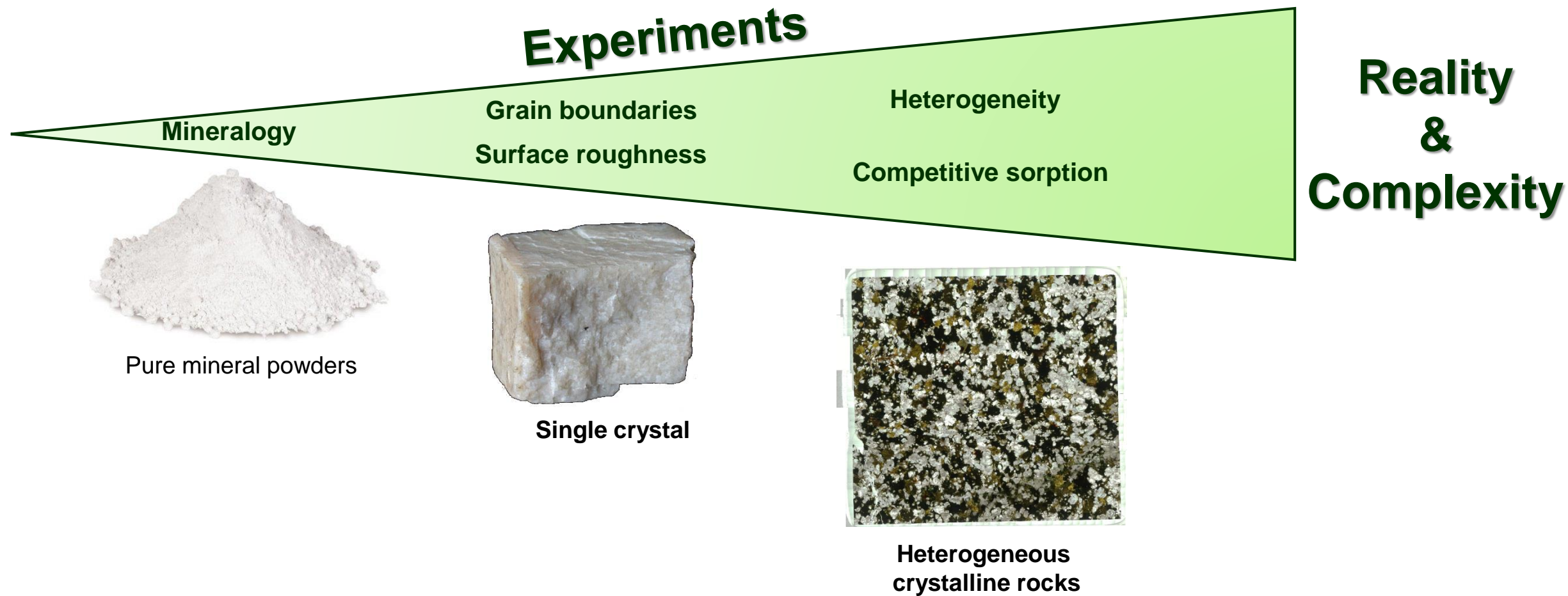
- Work is part of the iCross project
- **Radionuclide interaction and speciation** with rocks investigated on the **molecular scale**
(homogeneous mineral powders)
- **Nuclear waste repository** will be built on the **km scale**
- Need for experimental findings on larger scales

Can we straightforwardly apply findings from the molecular scale to larger scales?

What other factors influence the transport of radionuclides on larger scales?



Going larger makes things more complex



Cm(III) as a luminescent probe

- Curium is an actinide, which primarily exist in its **trivalent state Cm(III)**
- Cm(III) is a **chemical analogue to Am(III) and Pu(III)**
- **Reducing conditions are expected** in a deep geological repository
- After short lived fission products have decayed, **majority of activity** stems from **transuranium elements**

1

IA

1A

2

IIA

2A

3

IIIB

3B

4

IVB

4B

5

VB

5B

6

VIB

6B

7

VII B

7B

8

VIII

8

9

VIII

8

10

VIII

8

11

IB

1B

12

IIB

2B

13

IIIA

3A

14

IVA

4A

15

VA

5A

16

VIA

6A

17

VIIA

7A

18

VIIIA

8A

1

H

Hydrogen

1.008

2

He

Helium

4.003

3

Li

Lithium

6.941

4

Be

Beryllium

9.012

5

B

Boron

10.811

6

C

Carbon

12.011

7

N

Nitrogen

14.007

8

O

Oxygen

15.999

9

F

Fluorine

18.998

10

Ne

Neon

20.180

11

Na

Sodium

22.990

12

Mg

Magnesium

24.305

13

Al

Aluminum

26.982

14

Si

Silicon

28.086

15

P

Phosphorus

30.974

16

S

Sulfur

32.066

17

Cl

Chlorine

35.453

18

Ar

Argon

39.948

19

K

Potassium

39.098

20

Ca

Calcium

40.078

21

Sc

Scandium

44.956

22

Ti

Titanium

47.88

23

V

Vanadium

50.942

24

Cr

Chromium

51.996

25

Mn

Manganese

54.938

26

Fe

Iron

55.933

27

Co

Cobalt

58.933

28

Ni

Nickel

58.693

29

Cu

Copper

63.546

30

Zn

Zinc

65.39

31

Ga

Gallium

69.723

32

Ge

Germanium

72.61

33

As

Arsenic

74.922

34

Se

Selenium

78.972

35

Br

Bromine

79.904

36

Kr

Krypton

84.80

37

Rb

Rubidium

84.468

38

Sr

Strontium

87.62

39

Y

Yttrium

88.906

40

Zr

Zirconium

91.224

41

Nb

Niobium

92.906

42

Mo

Molybdenum

95.95

43

Tc

Technetium

98.907

44

Ru

Ruthenium

101.07

45

Rh

Rhodium

102.906

46

Pd

Palladium

106.42

47

Ag

Silver

107.868

48

Cd

Cadmium

112.411

49

In

Indium

114.818

50

Sn

Tin

118.71

51

Sb

Antimony

121.760

52

Te

Tellurium

127.6

53

I

Iodine

126.904

54

Xe

Xenon

131.29

55

Cs

Cesium

132.905

56

Ba

Barium

137.327

57-71

Lanthanide Series

72

Hf

Hafnium

178.49

73

Ta

Tantalum

180.948

74

W

Tungsten

183.85

75

Re

Rhenium

186.207

76

Os

Osmium

190.23

77

Ir

Iridium

192.22

78

Pt

Platinum

195.08

79

Au

Gold

196.967

80

Hg

Mercury

200.59

81

Tl

Thallium

204.383

82

Pb

Lead

207.2

83

Bi

Bismuth

208.980

84

Po

Polonium

[209]

85

At

Astatine

[210]

86

Rn

Radon

[222]

87

Fr

Francium

223.020

88

Ra

Radium

226.025

89-103

Actinide Series

104

Rf

Rutherfordium

[261]

105

Db

Dubnium

[262]

106

Sg

Seaborgium

[266]

107

Bh

Bohrium

[264]

108

Hs

Hassium

[269]

109

Mt

Meitnerium

[268]

110

Ds

Darmstadtium

[269]

111

Rg

Roentgenium

[272]

112

Cn

Copernicium

[277]

113

Nh

Nihonium

[284]

114

Fl

Flerovium

[289]

115

Mc

Moscovium

[288]

116

Lv

Livermorium

[293]

117

Ts

Tennessine

[294]

118

Og

Oganesson

[294]

57

La

Lanthanum

138.906

58

Ce

Cerium

140.115

59

Pr

Praseodymium

140.908

60

Nd

Neodymium

144.24

61

Pm

Promethium

144.913

62

Sm

Samarium

150.36

63

Eu

Europium

151.966

64

Gd

Gadolinium

157.25

65

Tb

Terbium

158.925

66

Dy

Dysprosium

162.50

67

Ho

Holmium

164.930

68

Er

Erbium

167.26

69

Tm

Thulium

168.934

70

Yb

Ytterbium

173.04

71

Lu

Lutetium

174.967

89

Ac

Actinium

227.028

90

Th

Thorium

232.038

91

Pa

Protactinium

231.036

92

U

Uranium

238.029

93

Np

Neptunium

237.048

94

Pu

Plutonium

244.064

95

Am

Americium

243.061

96

Cm

Curium

247.070

97

Bk

Berkelium

247.070

98

Cf

Californium

251.080

99

Es

Einsteinium

[254]

100

Fm

Fermium

257.095

101

Md

Mendelevium

258.1

102

No

Nobelium

259.101

103

Lr

Lawrencium

[262]

Alkali Metal

Alkaline Earth

Transition Metal

Basic Metal

Semimetal

Nonmetal

Halogen

Noble Gas

Lanthanide

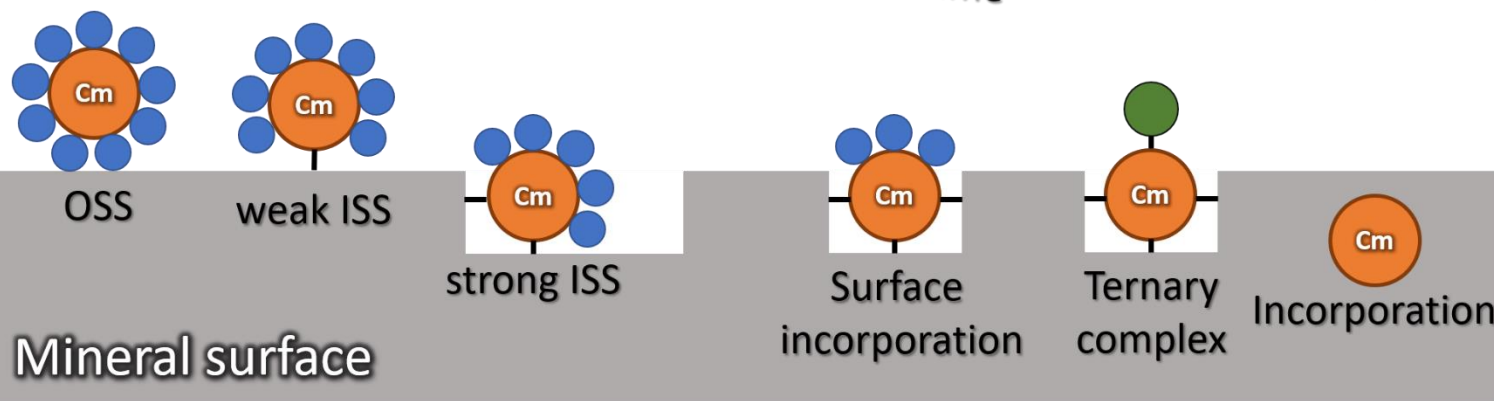
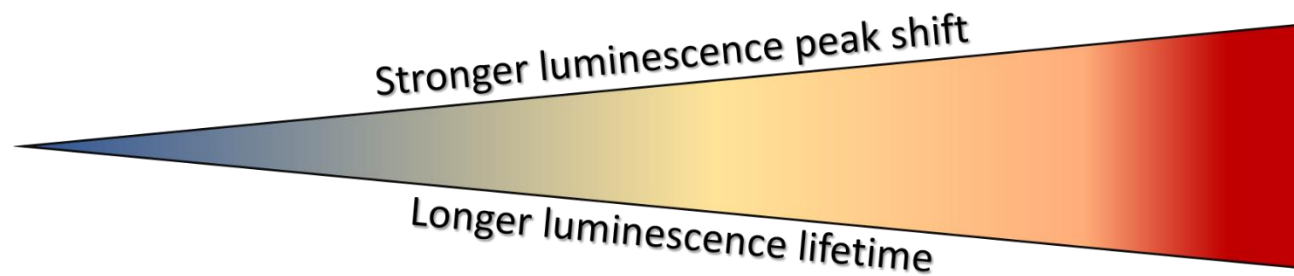
Actinide

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sciencesonline.com

<https://sciencenotes.org/wp-content/uploads/2014/11/PeriodicTableBoilingPoint.png> Accessed: 14.06.2021

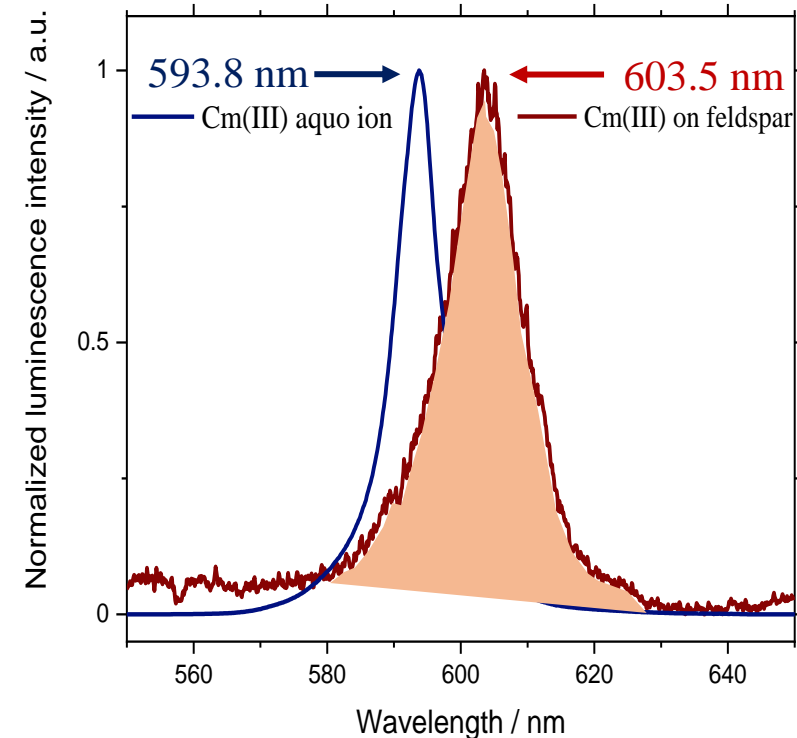
Cm(III) as a luminescent probe

- Cm(III) possesses excellent luminescence properties
- Cm(III) bound to the surface experiences a **luminescence peak shift**
- Cm(III) **peak position** is an indication of **sorption strength**



● H₂O

● Ligand (e.g. silicate, carbonate)

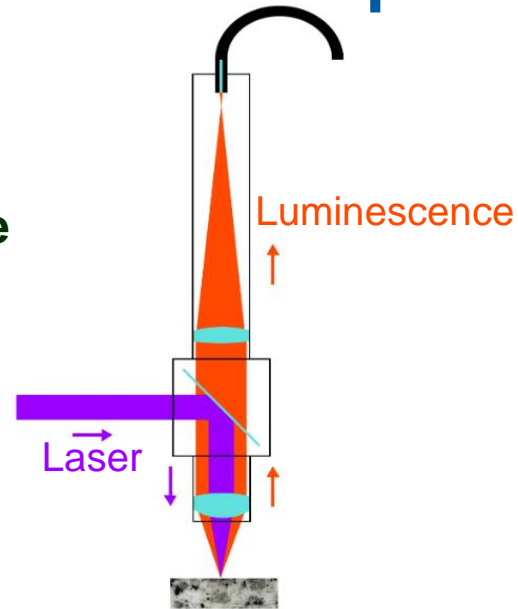


- Water is a luminescence quencher; **less water** means **longer lifetimes**
- From **lifetime** we can **calculate** the amount of **water bound to Cm(III)**
- Combination of **peak shift** and **lifetime** gives us information about **chemical speciation of Cm(III)** on a mineral surface

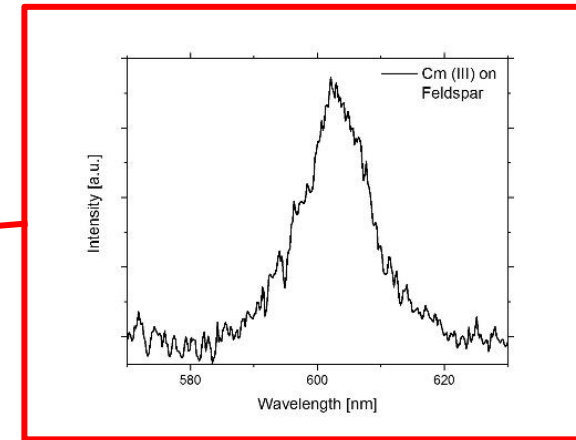
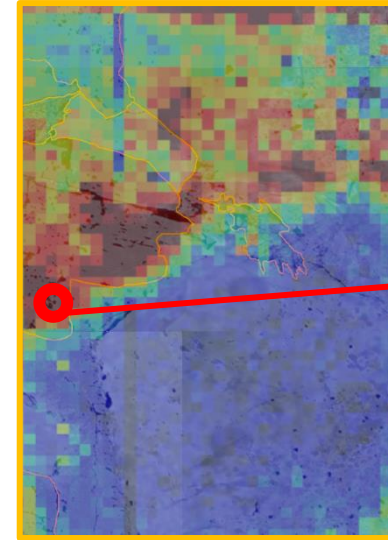
Spatially-resolved correlative spectroscopy

μTRLFS

- Allows **spatially resolved** investigation of **luminescence** (Cm(III)/Eu(III))
- Scan surface pixel by pixel
- **Each pixel** represents a **luminescence spectrum**

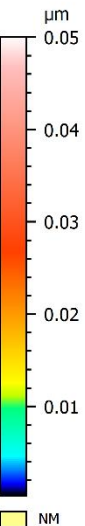
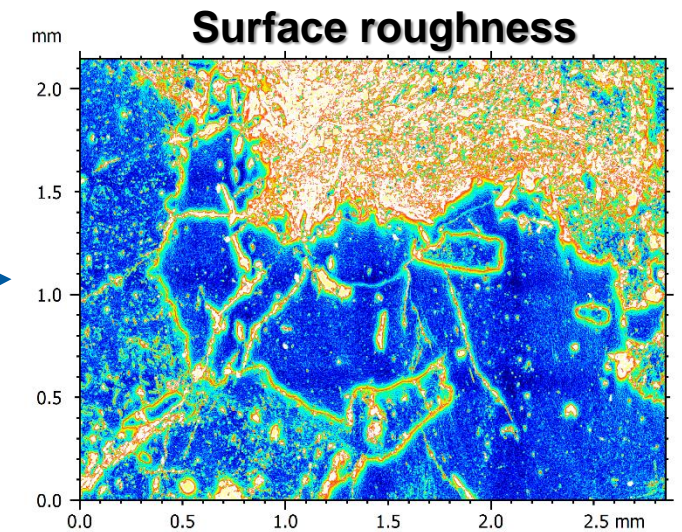
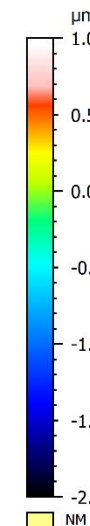
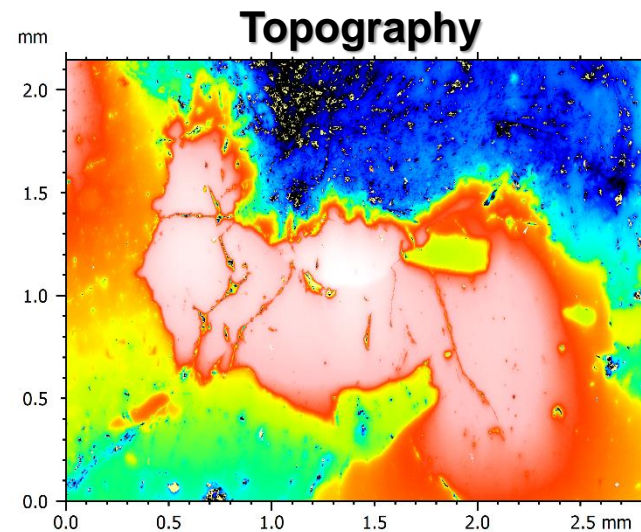


Luminescence intensity



Interferometry

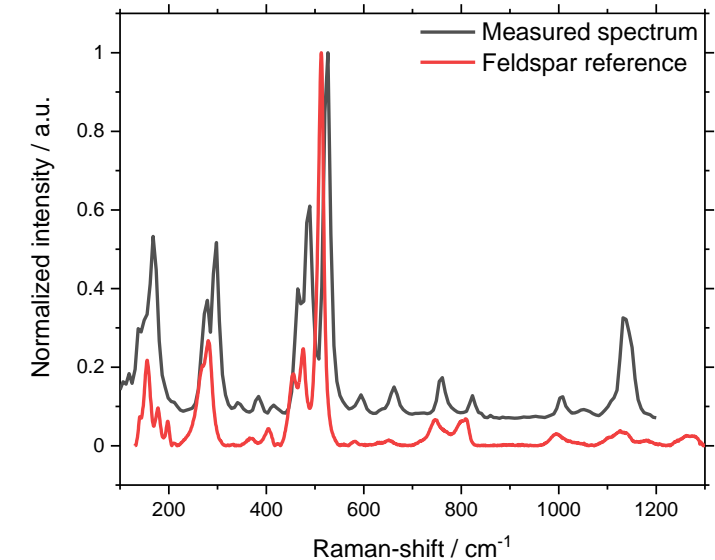
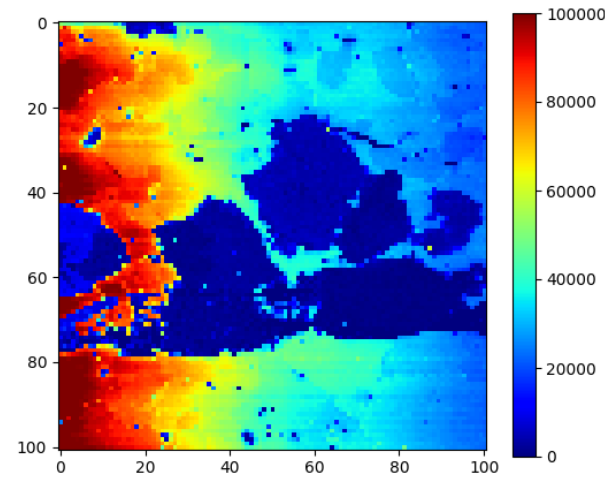
- Method to determine topography (“height profile”) of a surface
- Nanometer lateral resolution; micrometer spatial resolution
- From topography surface roughness can be calculated



Spatially-resolved correlative spectroscopy

Raman-microscopy

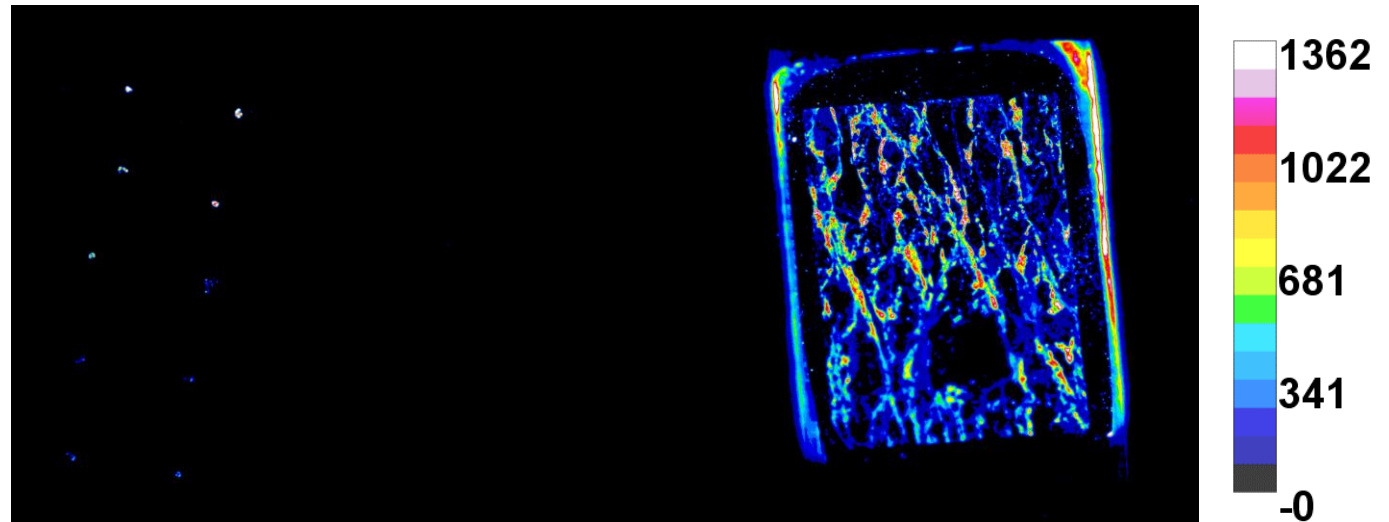
- Scanning of surface via Raman-microscope
- Comparison of spectra to RRUFF-database



Autoradiography

- Measurement of the activity (alpha, beta) on the sample
- Calibration of autoradiography allows numerical quantification of sorbed radionuclide

Sorption uptake / pmol/cm^2



Cm(III) sorption on granite and gneiss

Effects of surface roughness and mineralogy on the sorption of Cm(III) on crystalline rock

August 2021 Journal of Hazardous Materials 423(Pt A):127006

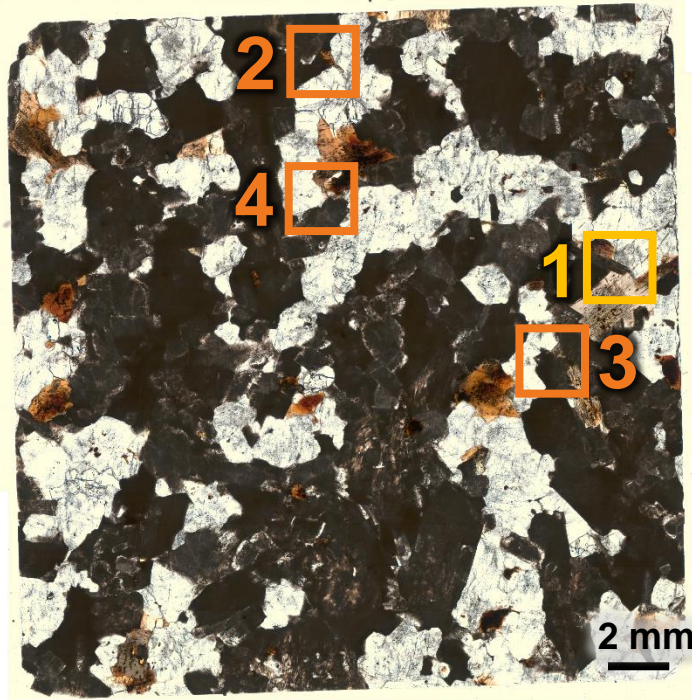
DOI: 10.1016/j.jhazmat.2021.127006

Cm(III) sorption on granite and gneiss

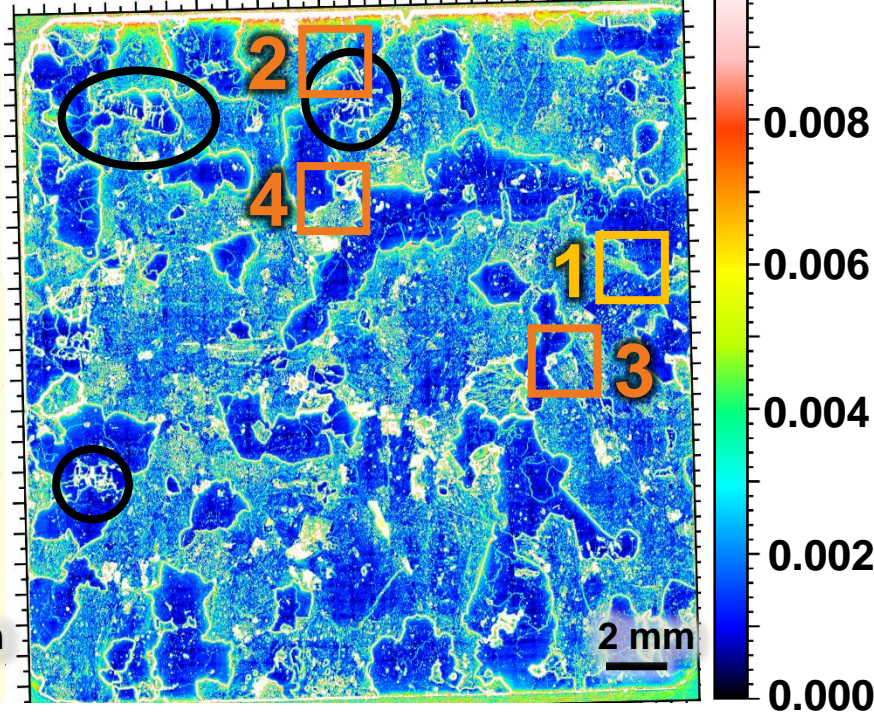
Granite – thin section

I = 0.1 M NaCl pH = 8.0
[Cm(III)] = 10^{-6} M t = 7 d

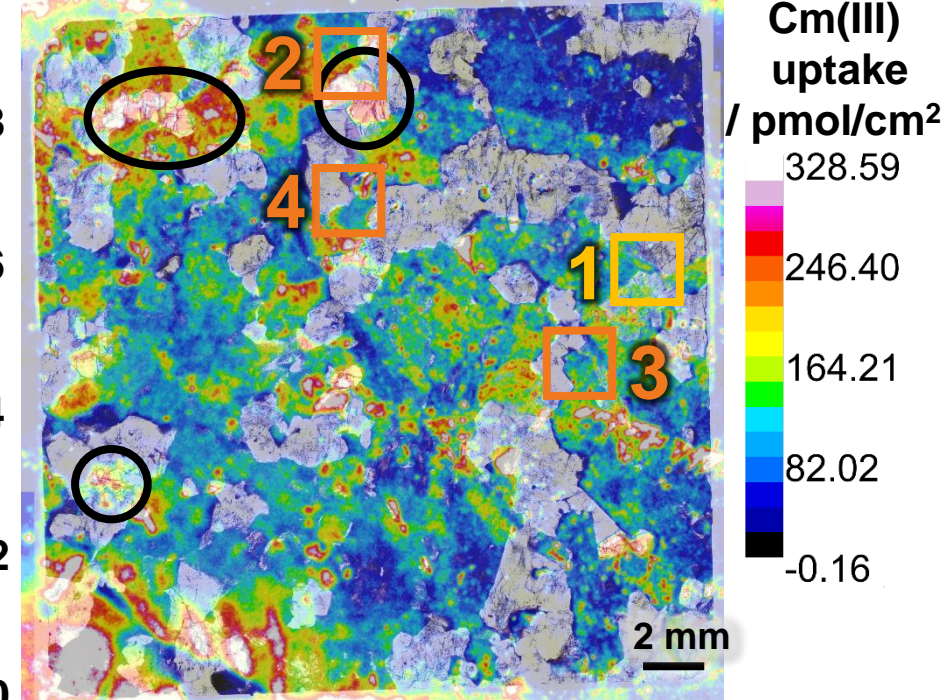
Mineral composition



Surface roughness



Cm(III) uptake



Sorption uptake:

- Mica > Feldspar > Quartz
- **Surface roughness increases sorption uptake** by almost one order of magnitude

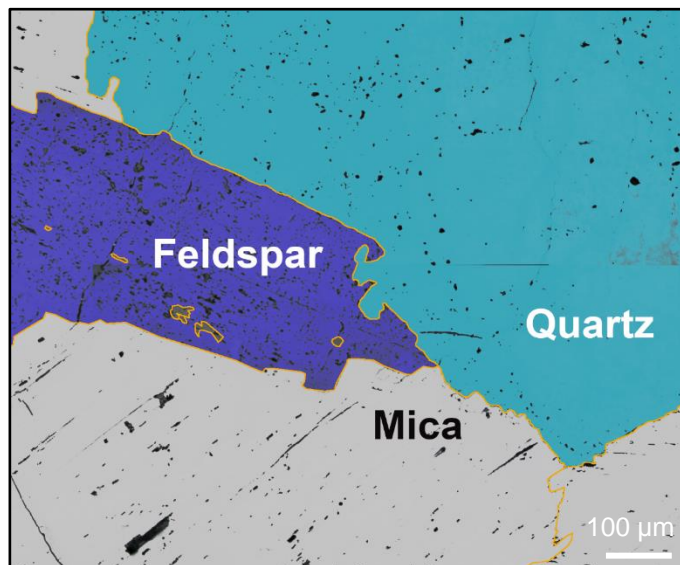
Mica: $\text{KAl}_2(\text{AlSi}_3\text{O}_{10})(\text{F,OH})_2$ / $\text{K}(\text{Mg,Fe})_3(\text{AlSi}_3\text{O}_{10})(\text{F,OH})_2$
Feldspar: KAlSi_3O_8 / $\text{NaAlSi}_3\text{O}_8$ / $\text{CaAl}_2\text{Si}_2\text{O}_8$
Quartz: SiO_2

Cm(III) sorption on granite and gneiss

Granite – thin section ROI 1

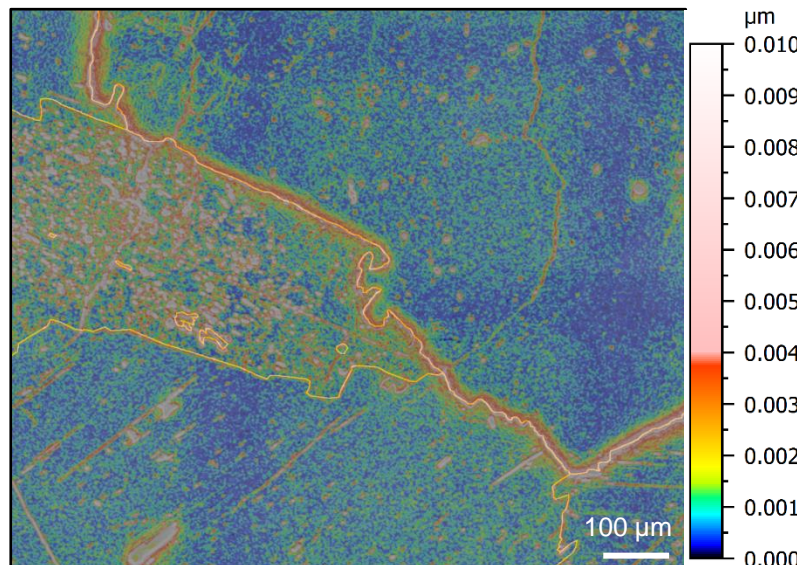
$I = 0.1 \text{ M NaCl}$ $\text{pH} = 8.0$
 $[\text{Cm(III)}] = 10^{-6} \text{ M}$ $t = 7 \text{ d}$

Mineral phases

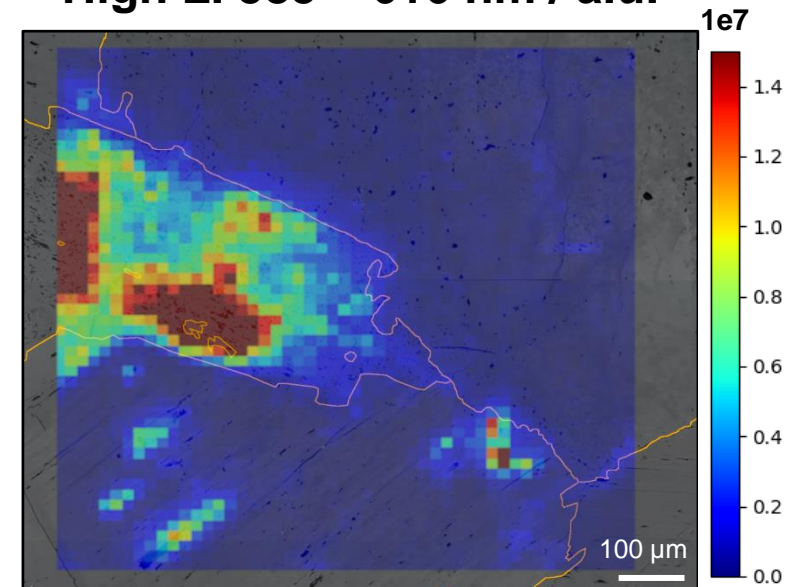


Mica: $\text{KAl}_2(\text{AlSi}_3\text{O}_{10})(\text{F,OH})_2$ / $\text{K}(\text{Mg,Fe})_3(\text{AlSi}_3\text{O}_{10})(\text{F,OH})_2$
Feldspar: KAlSi_3O_8 / $\text{NaAlSi}_3\text{O}_8$ / $\text{CaAl}_2\text{Si}_2\text{O}_8$
Quartz: SiO_2

Surface roughness

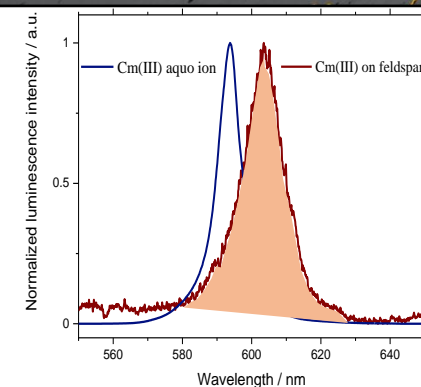


High LI 585 – 616 nm / a.u.



Luminescence intensity:

- Feldspar > Mica (quenching) > Quartz
- **Surface roughness increases sorption uptake** on feldspar and mica by around one order of magnitude

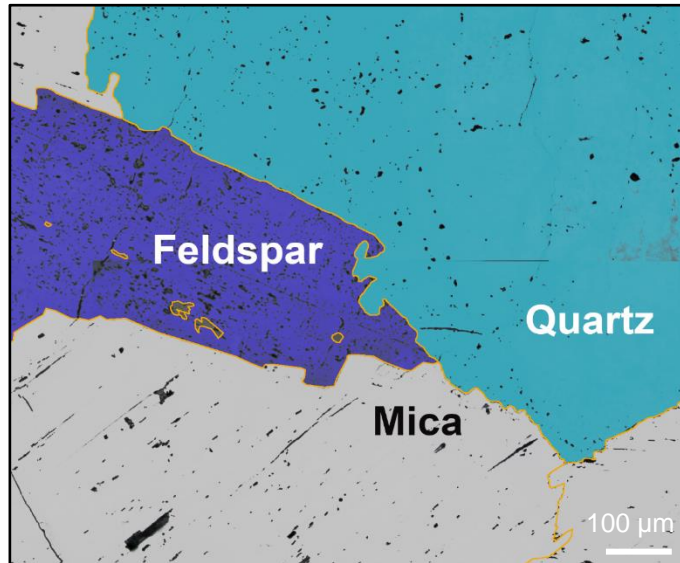


Cm(III) sorption on granite and gneiss

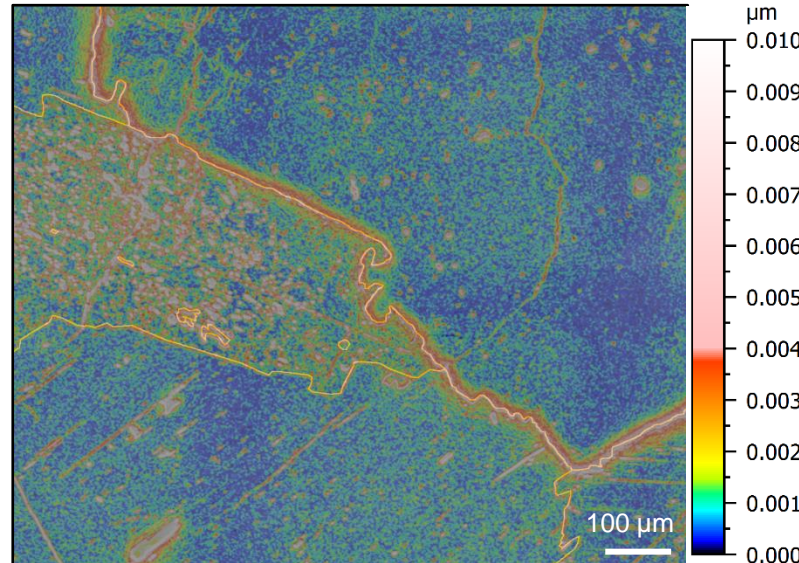
$I = 0.1 \text{ M NaCl}$ $\text{pH} = 8.0$
 $[\text{Cm(III)}] = 10^{-6} \text{ M}$ $t = 7 \text{ d}$

Granite – thin section ROI 1

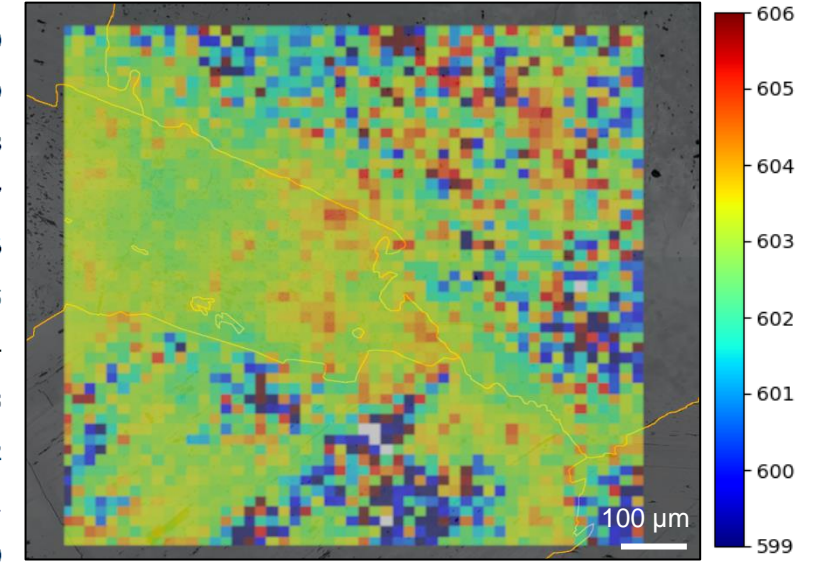
Mineral phases



Surface roughness



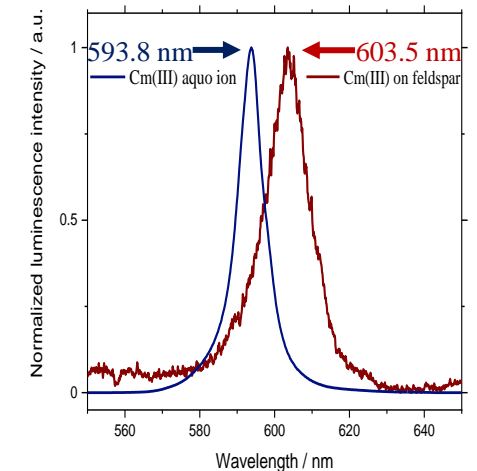
Peak position / nm



More **pronounced red-shifts** in regions **with higher surface roughness**

Feldspar: 602 – 605 nm Quartz: 602 – 605 nm Mica: 603 – 604 nm

Confirmation through lifetime: higher surface roughness leads to stronger complexation

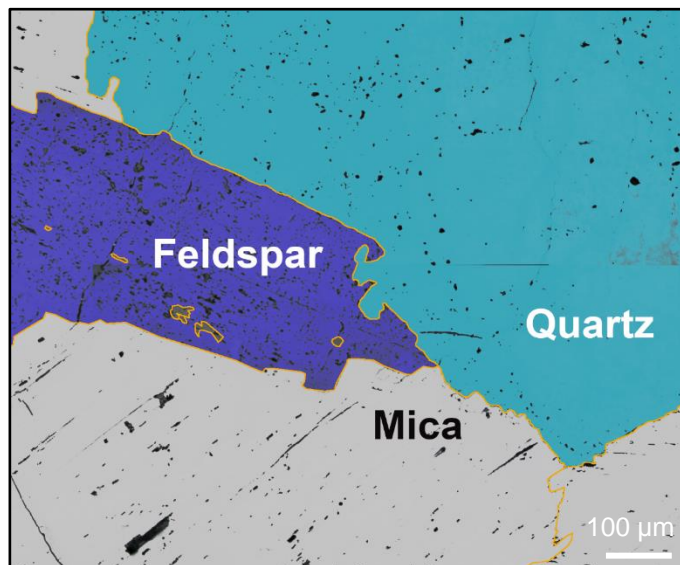


Cm(III) sorption on granite and gneiss

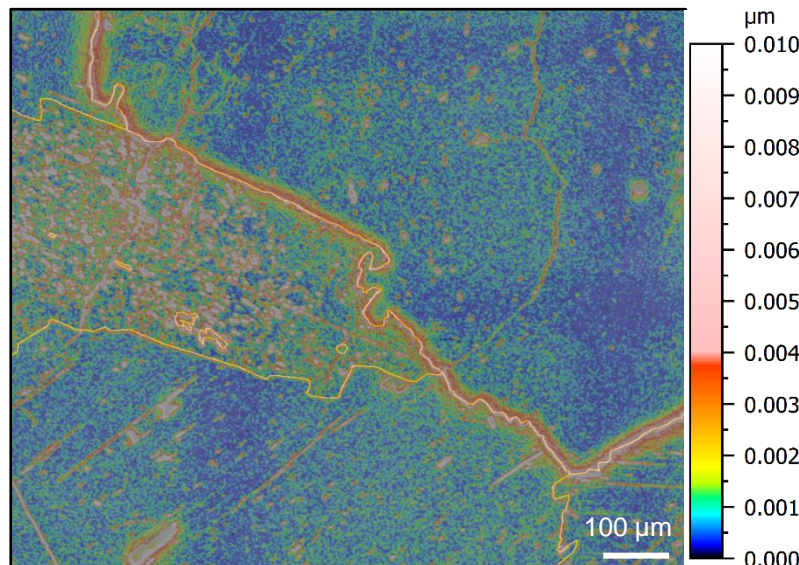
$I = 0.1 \text{ M NaCl}$ $\text{pH} = 8.0$
 $[\text{Cm(III)}] = 10^{-6} \text{ M}$ $t = 7 \text{ d}$

Granite – thin section ROI 1

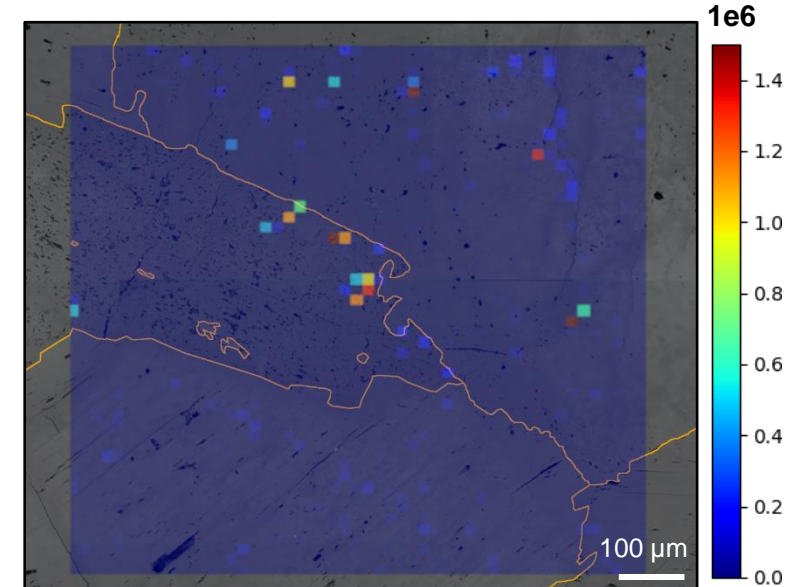
Mineral phases



Surface roughness



LI 614 – 640 nm / a.u.

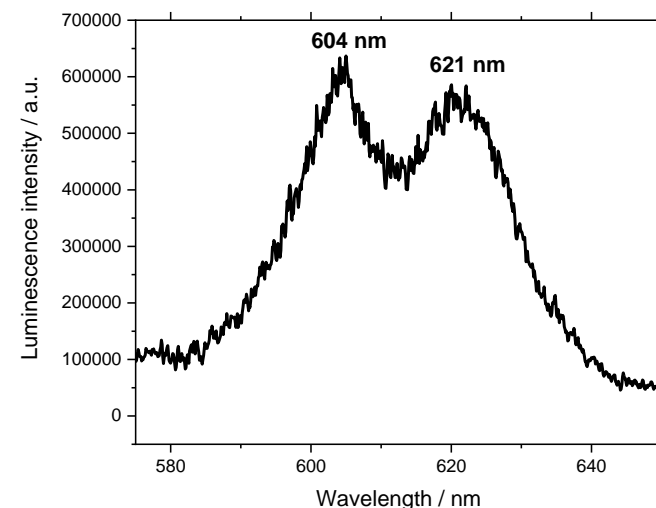


Regions with **high surface roughness** occasionally show **2nd Cm(III) peak**

On feldspar, quartz and their grain boundary with each other

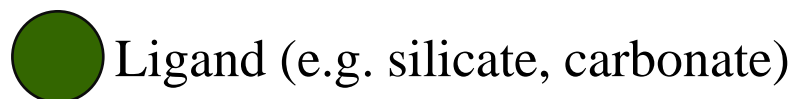
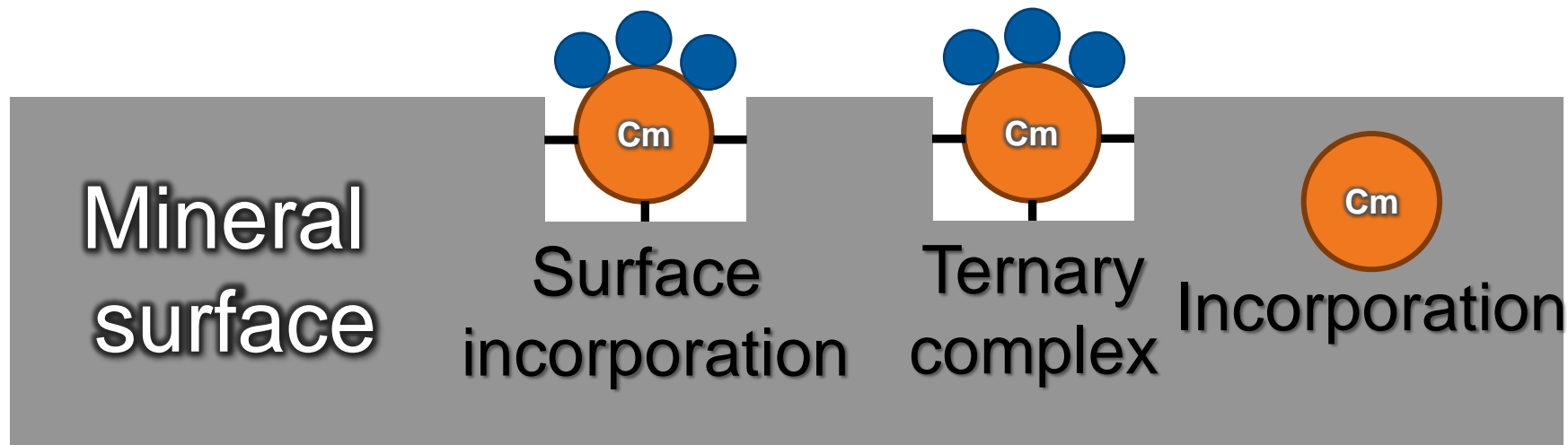
Feldspar: 621 – 625 nm Quartz: 625 – 630 nm

High peak shift suggest incorporation, which is unlikely

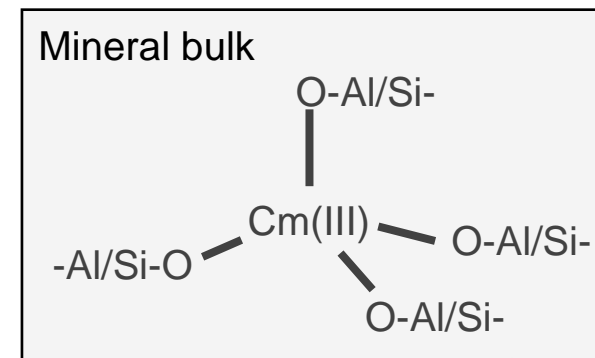
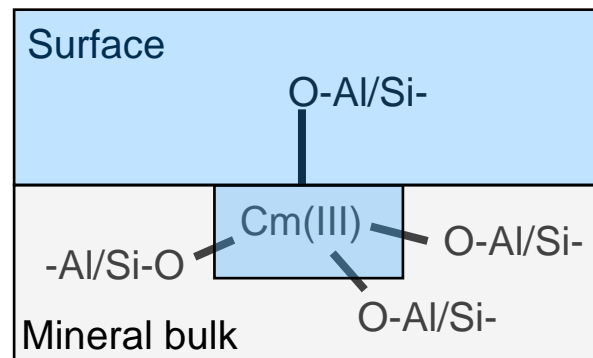


Cm(III) sorption on granite and gneiss

Granite – thin section ROI 1



Chemical environment of Cm(III) ternary complex and bulk incorporated Cm(III) might be similar

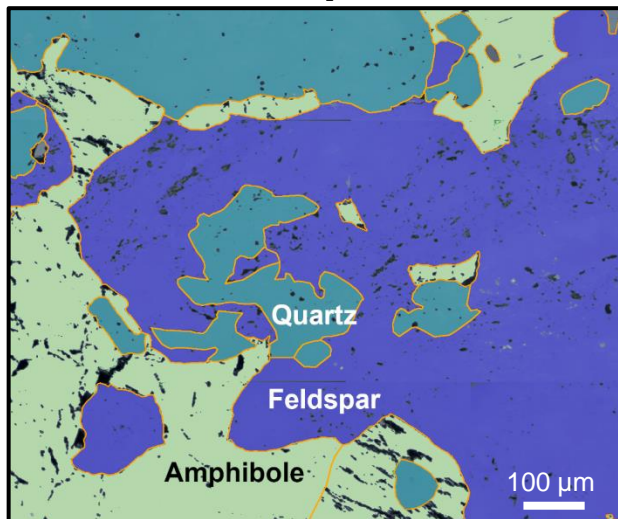


Cm(III) sorption on granite and gneiss

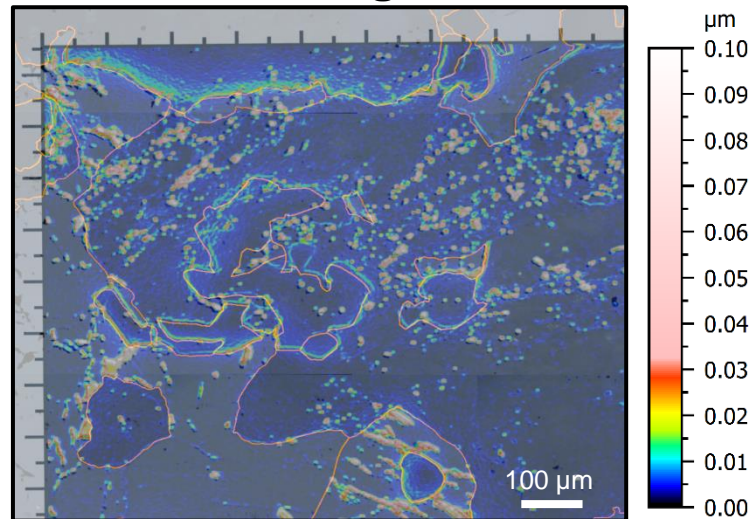
I = 0.1 M NaCl pH = 8.0
[Cm(III)] = 10^{-6} M t = 7 d

Gneiss – thin section ROI 1

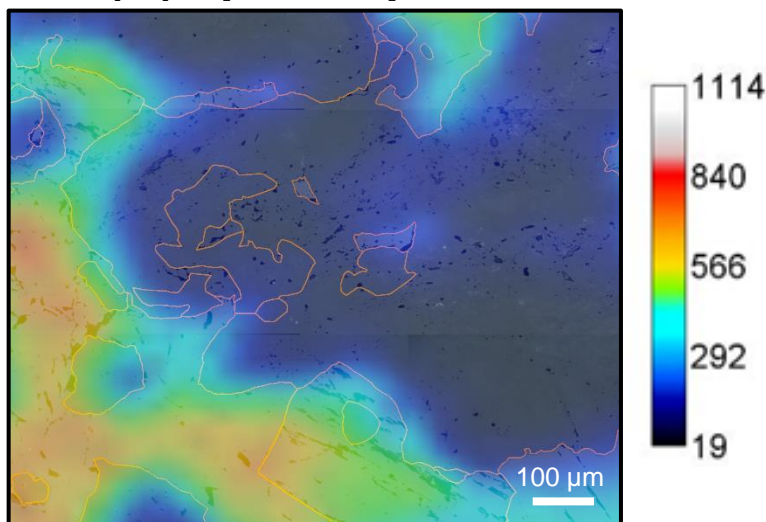
Mineral phases



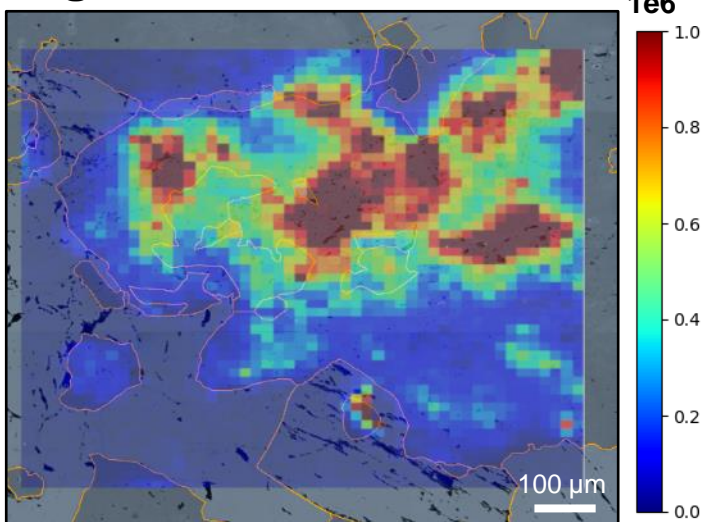
Surface roughness



Cm(III) uptake / pmol/cm²



High LI 585 – 616 nm / a.u.



- Sorption on gneiss almost exclusively occurs on amphibole
- Sorption on feldspar and quartz mineral phases is far lower than on granite sample
- Sorption on feldspar and quartz occurs **only** in regions with a high surface roughness
- Surface roughness becomes driving parameter for sorption on low uptake mineral phases

Amphibole: $(K,Na)_{0-1}(Ca,Na,Fe,Mg)_2(Mg,Fe,Al)_5(Al,Si)_8O_{22}(OH)_2$
Feldspar: $KAlSi_3O_8$ / $NaAlSi_3O_8$ / $CaAl_2Si_2O_8$
Quartz: SiO_2

Conclusions and implications

Cm(III) sorption on crystalline rock surfaces:

- Mineralogy is the most important parameter; closely followed by surface roughness
- Higher surface roughness leads to higher sorption uptake and stronger surface complexes
- Surface roughness becomes driving parameter on minerals in a competitive sorption environment
- pH dependent sorption behavior leads to strong preferential sorption at lower pH
- Observation of Cm(III) ternary complex formation/incorporation

During an upscaling process new parameters need to be considered

Parameters determined at smaller scales will not necessarily behave linearly or additively at larger scales

Thank you for your attention 😊

Sample locations

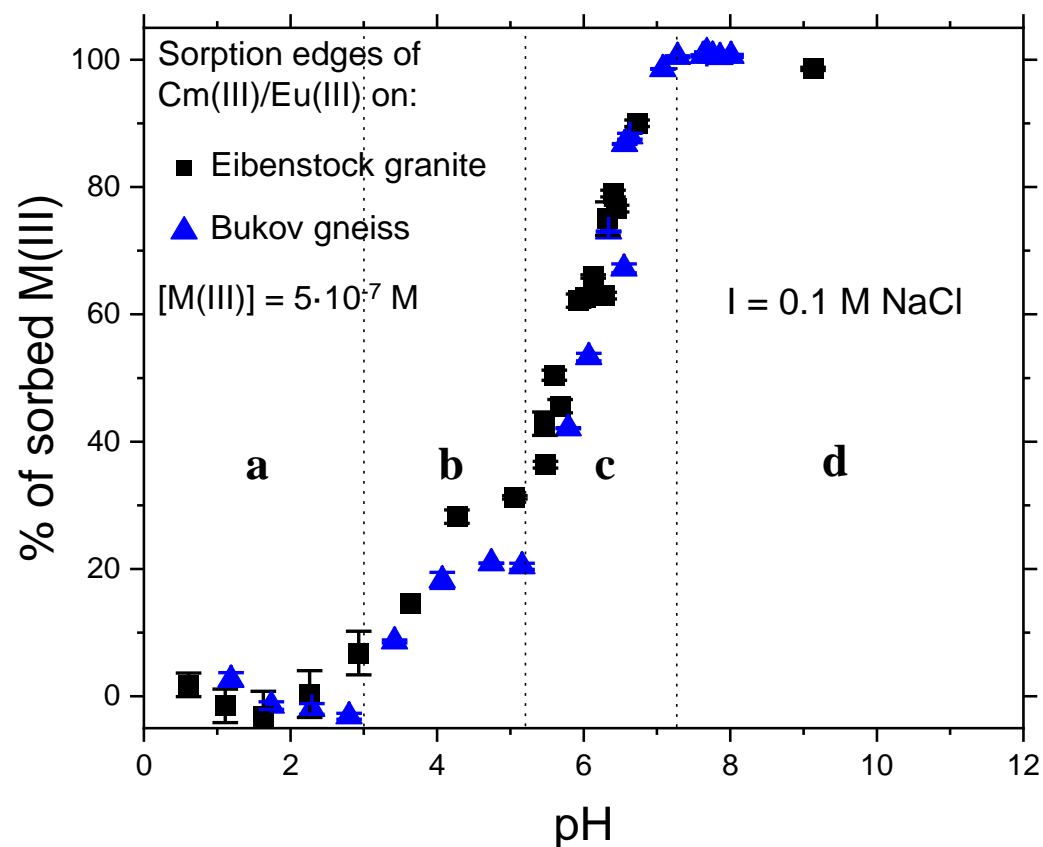
- Crystalline rock samples from different locations in Europe
- **Eibenstock:** granite, former uranium mine in Erzgebirge, Germany
- **Bukov:** migmatized gneiss, underground research lab in Czech Republic
- **Onkalo:** granitic pegmatite, underground research lab in Finland
- **Grimsel:** likely granodiorite/mylonite/fault gouge, Grimsel test site in Switzerland



Cm(III) sorption on granite and gneiss

Granite/Gneiss – powder

I = 0.1 M NaCl pH = 8.0
[Cm(III)] = 10^{-6} M t = 7 d



- a) No significant sorption
- b) Slight increase to 20/30% sorption
- c) Accelerated sorption up to 100% sorption
- d) Complete sorption

Chosen pH for thin section experiments: pH 8.0

Appendix

Mineral Granite	Percentage [%]
Quartz	45.0
Feldspar	42.5
Mica	11.5
Minor	0.9

Mineral Gneiss	Percentage [%]
Feldspar	44.8
Amphibole	33.3
Quartz	13.5
Mica	9.2
Chlorite	1.5
Alkali-feldspar	0.7

Mineral Granitic pegmatite	Percentage [%]
Albite	35.0
Illite	18
Quartz	16.0
Microcline	16.0
Babingtonite?	9.0
Minor	5.7

Mineral Grimsel	Percentage [%]
Orthopyroxene	27.0
Quartz	24.9
Albite	16.4
Microcline	9.8
Aragonite	9.4
Polyolithionite	5.7
Orthoclase	2.5
Phlogopite	2.5
Titanite	1.8

Lifetime analysis

Granite minerals	n(H ₂ O)	Species
Feldspar (smooth)	7.4 ± 0.5	Weak ISS (23%)
	5.7 ± 0.5	Strong ISS (47%)
	3.3 ± 0.7	SF incorporation (30%)
Feldspar (rough)	9.0 ± 0.5	OSS (5%)
	7.5 ± 0.5	Weak ISS (6%)
	5.1 ± 0.3	Strong ISS (26%)
	3.4 ± 0.8	SF incorporation (63%)
	0.0 ± 0.5	Ternary complex/ Bulk incorporation
Quartz (smooth)	5.5 ± 0.5	Strong ISS
	3.0 ± 1.0	SF incorporation
Quartz (rough)	5.7 ± 0.5	Strong ISS
	0.0 ± 0.5	Ternary complex/ Bulk incorporation
Topaz (smooth)	4.0 ± 0.5	SF incorporation
Topaz (rough)	6.0 ± 0.5	Strong ISS
	2.8 ± 1.6	SF incorporation
Mica (smooth)*	9.0 ± 0.5	OSS
	7.0 ± 0.5	Weak ISS
	6.0 ± 0.5	Strong ISS
	3.0 ± 1.0	SF incorporation
Mica (rough)*	7.5 ± 0.5	Weak ISS
	5.0 ± 0.5	Strong ISS
	3.0 ± 1.0	SF incorporation

Gneiss minerals	n(H ₂ O)	Species
Feldspar (smooth)	9.0 ± 0.5	OSS (14%)
	7.2 ± 0.4	Weak ISS (24%)
	5.7 ± 0.5	Strong ISS (33%)
	3.5 ± 0.8	SF incorporation (29%)
Feldspar (rough)	7.3 ± 0.5	Weak ISS (23%)
	5.3 ± 0.4	Strong ISS (31%)
	2.5 ± 1.3	SF incorporation (46%)
Quartz (smooth)	7.5 ± 0.5	Weak ISS
	6.0 ± 0.5	Strong ISS
	3.0 ± 0.5	SF incorporation
Quartz (rough)	3.0 ± 1	SF incorporation
	0.0 ± 0.5	Ternary complex/ Bulk incorporation
Amphibole*	7.7 ± 0.5	Weak ISS
	6.0 ± 0.5	Strong ISS

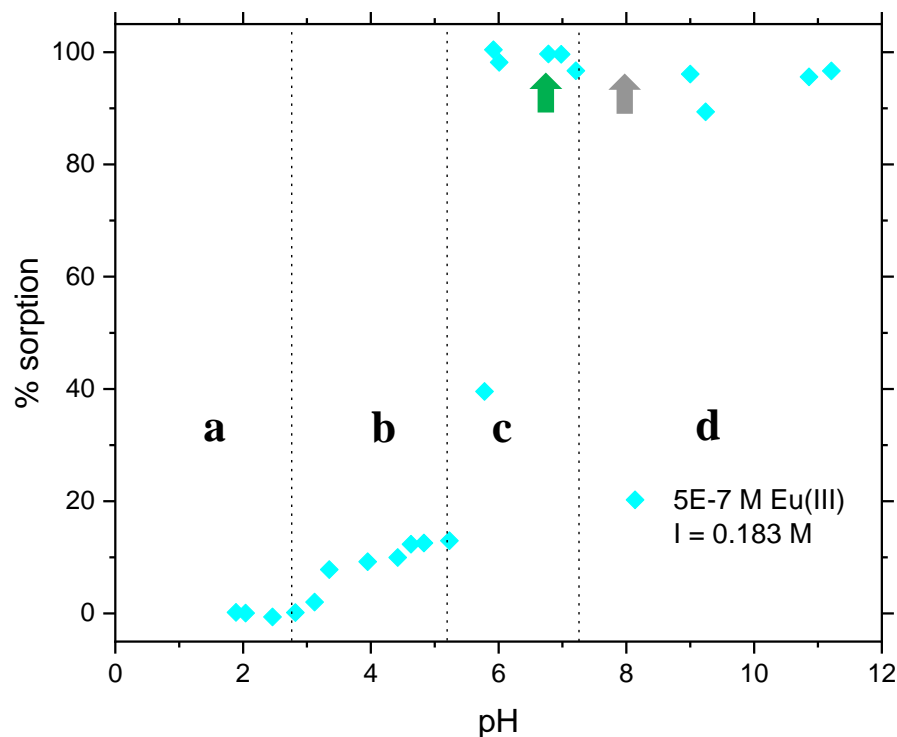
Cm(III) sorption on granitic pegmatite

Cm(III) sorption on granitic pegmatite

Granitic pegmatite – powder

Sample from Onkalo, Finland

$I = 0.183 \text{ M NaCl}$ $\text{pH} = 6.83$
 $[\text{Cm(III)}] = 10^{-5} \text{ M}$ $t = 7 \text{ d}$



- a) No significant sorption
- b) Slight increase to 15% sorption
- c) Abrupt sorption step to 100% sorption
- d) Complete sorption

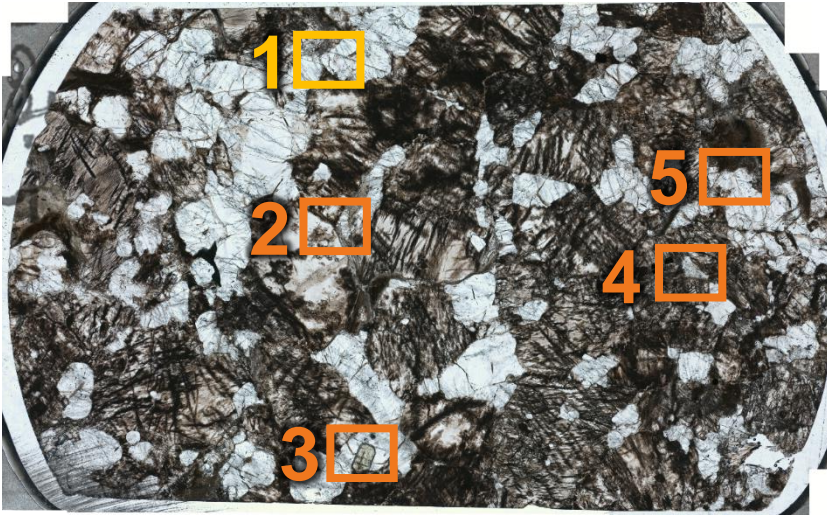
Chosen pH for thin section experiments: **pH 6.83**
(compared to **pH 8.0** in previous experiments)

Cm(III) sorption on granitic pegmatite

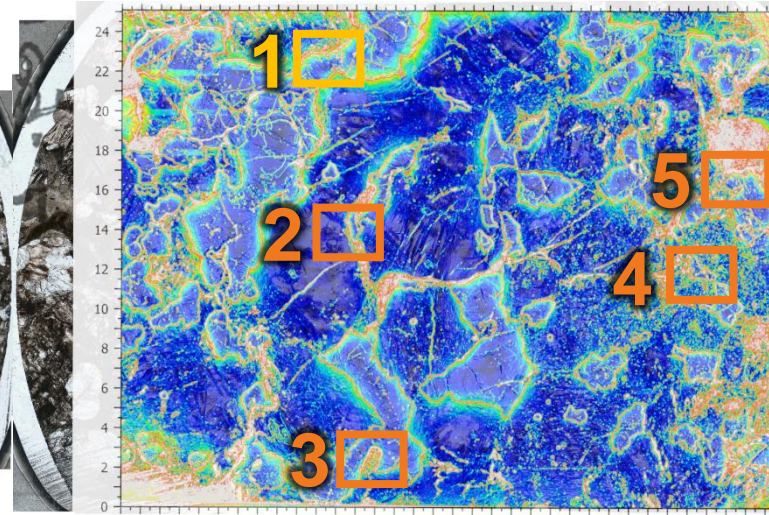
Granitic pegmatite – thin section

$I = 0.183 \text{ M NaCl}$ $\text{pH} = 6.83$
 $[\text{Cm(III)}] = 10^{-5} \text{ M}$ $t = 7 \text{ d}$

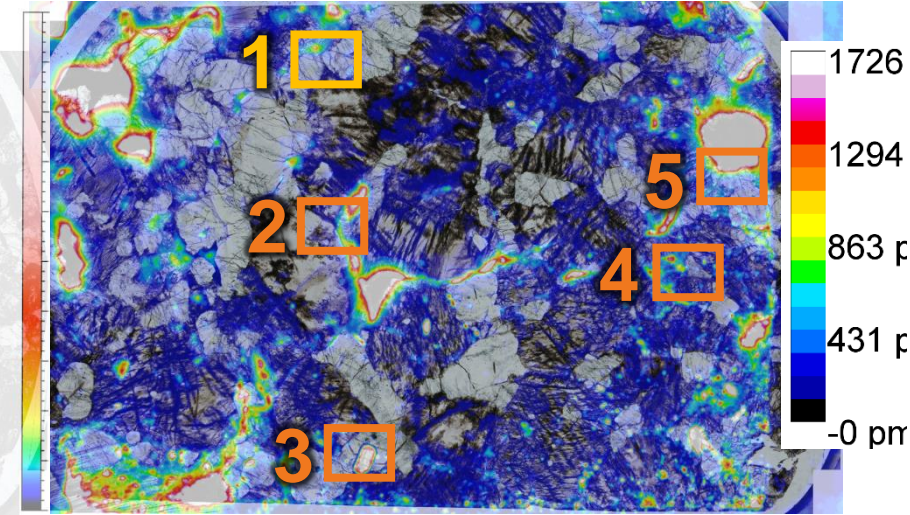
Mineral composition



Surface roughness



Cm(III) uptake / pmol/cm²



- Sorption occurs in primarily **“hot spots”** (identified as mica/apatite)
- Hot spot regions also exhibit a high surface roughness

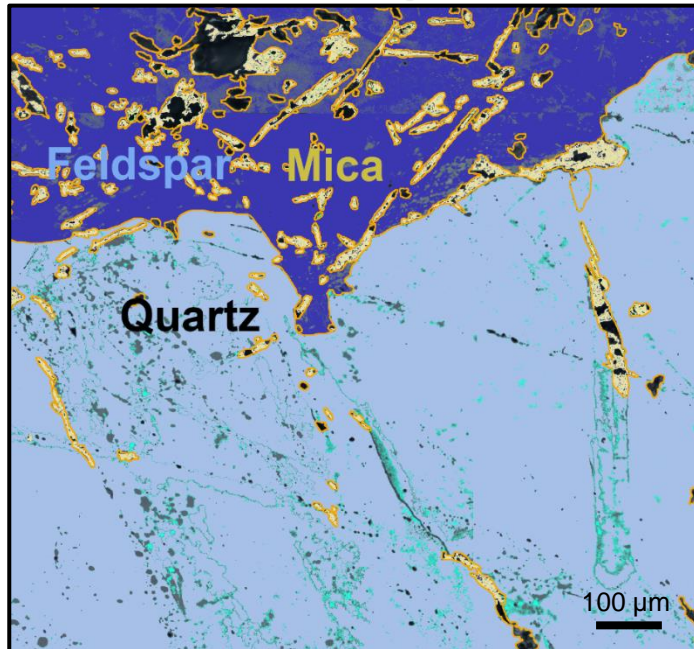
Cm(III) sorption on granitic pegmatite

Granitic pegmatite – thin section ROI 1

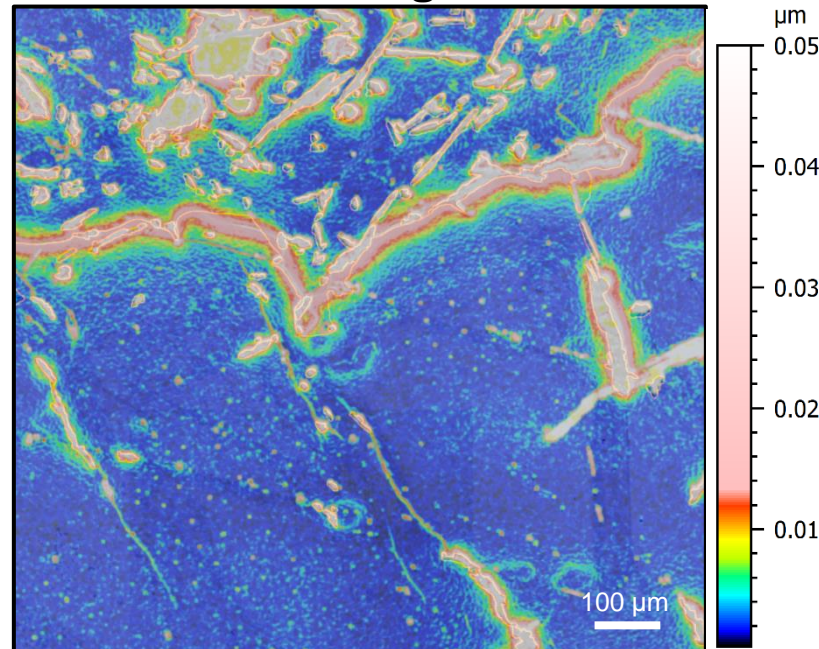
$I = 0.183 \text{ M NaCl}$ $\text{pH} = 6.83$

$[\text{Cm(III)}] = 10^{-5} \text{ M}$ $t = 7 \text{ d}$

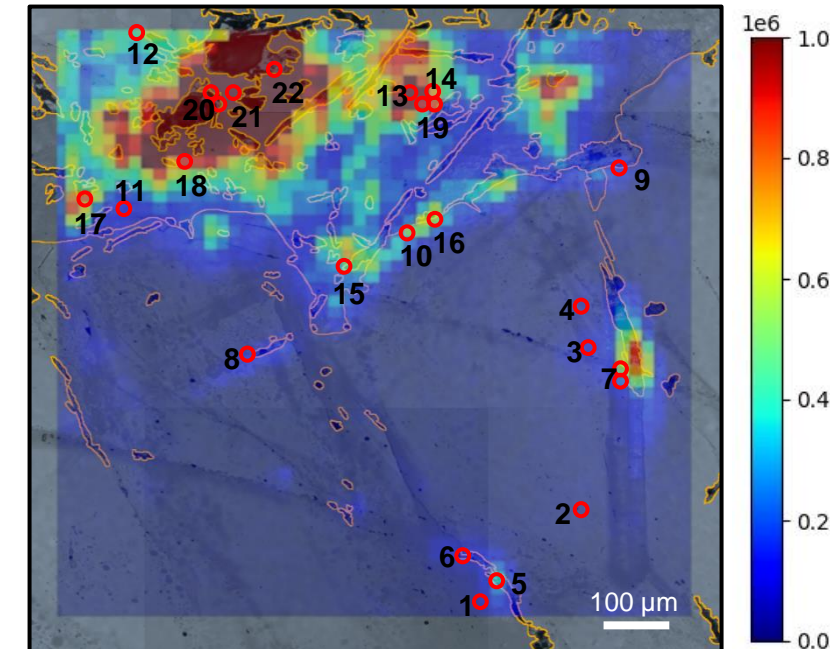
Mineral composition



Surface roughness



LI 580 – 630 nm / a.u.



Mica: $\text{KAl}_2(\text{AlSi}_3\text{O}_{10})(\text{F,OH})_2$ / $\text{K}(\text{Mg,Fe})_3(\text{AlSi}_3\text{O}_{10})(\text{F,OH})_2$
Feldspar: KAlSi_3O_8 / $\text{NaAlSi}_3\text{O}_8$ / $\text{CaAl}_2\text{Si}_2\text{O}_8$
Quartz: SiO_2

- Luminescence intensity: Mica > Feldspar > Quartz
- Luminescence on mica measurable

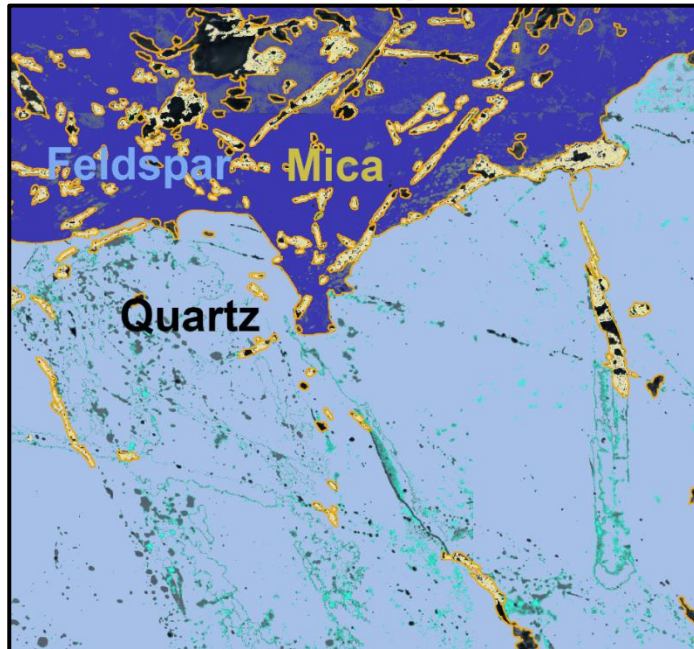
Cm(III) sorption on granitic pegmatite

Granitic pegmatite – thin section ROI 1

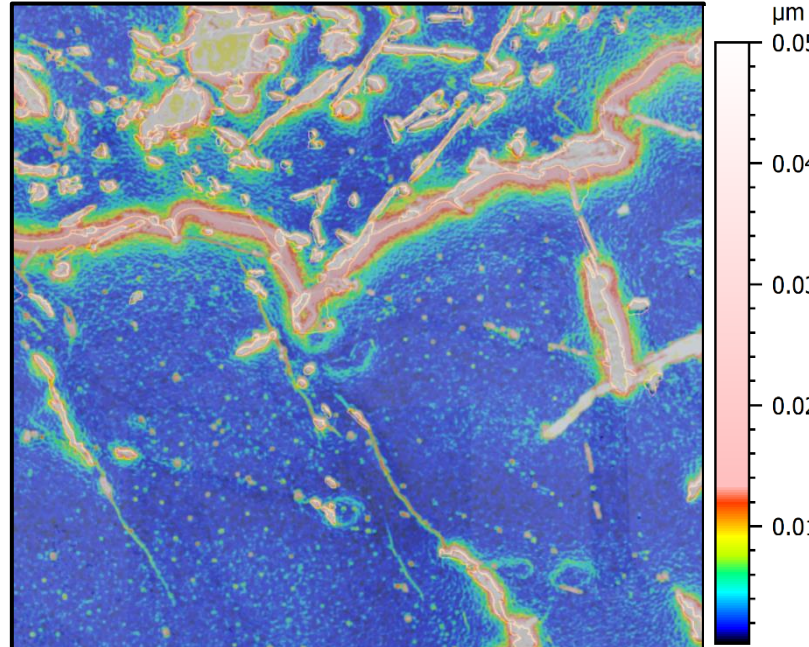
$I = 0.183 \text{ M NaCl}$ $\text{pH} = 6.83$

$[\text{Cm(III)}] = 10^{-5} \text{ M}$ $t = 7 \text{ d}$

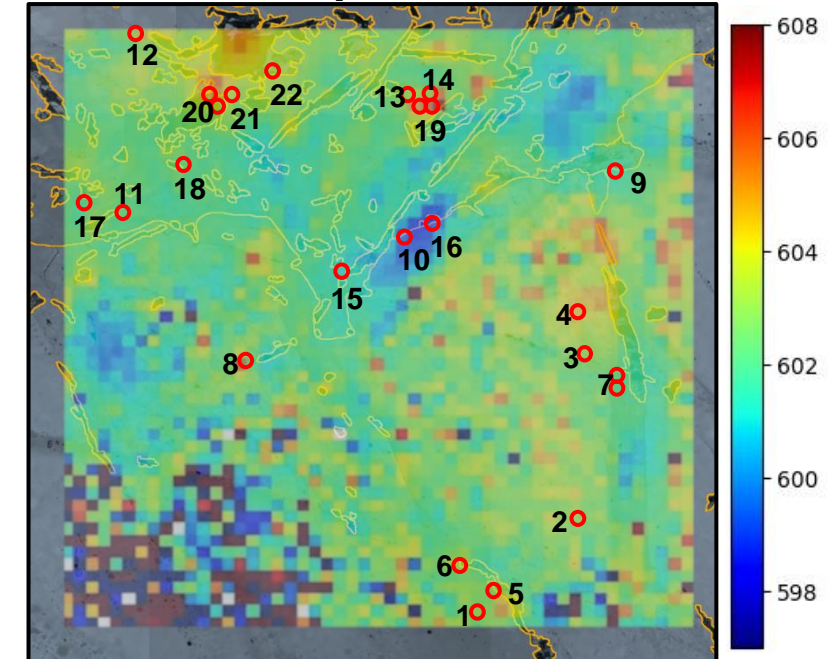
Mineral composition



Surface roughness



Peak position / nm



Mica: $\text{KAl}_2(\text{AlSi}_3\text{O}_{10})(\text{F,OH})_2$ / $\text{K}(\text{Mg,Fe})_3(\text{AlSi}_3\text{O}_{10})(\text{F,OH})_2$
Feldspar: KAlSi_3O_8 / $\text{NaAlSi}_3\text{O}_8$ / $\text{CaAl}_2\text{Si}_2\text{O}_8$
Quartz: SiO_2

Quartz: individual bond strengths per pixel

Feldspar: homogeneous sorption strength

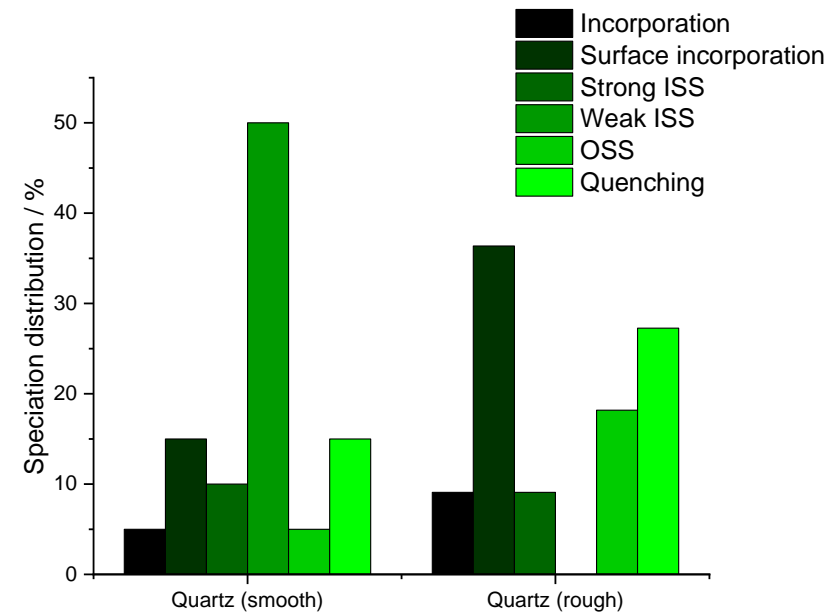
Mica: clusters of high/low strength

Cm(III) sorption on granitic pegmatite

Granitic pegmatite – thin section lifetimes

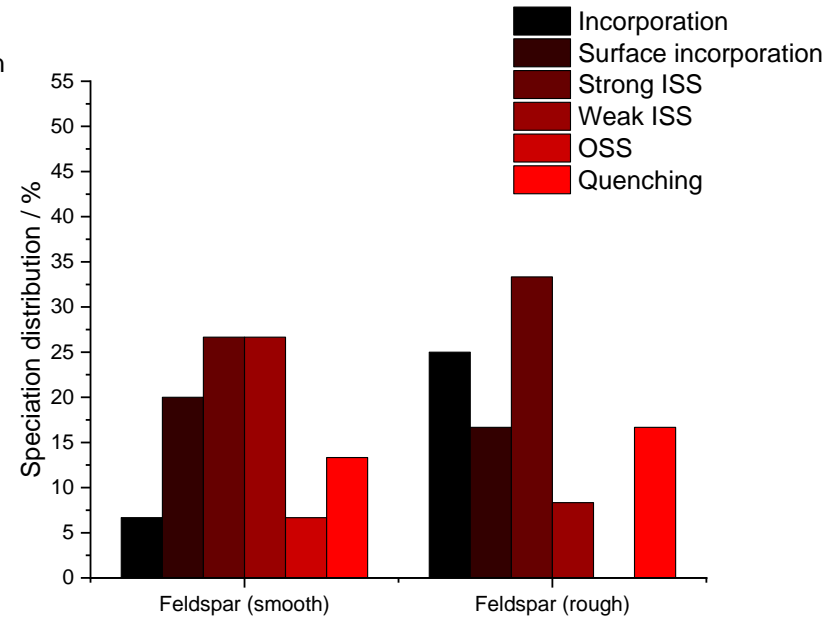
$I = 0.183 \text{ M NaCl}$ $\text{pH} = 6.83$

$[\text{Cm(III)}] = 10^{-5} \text{ M}$ $t = 7 \text{ d}$



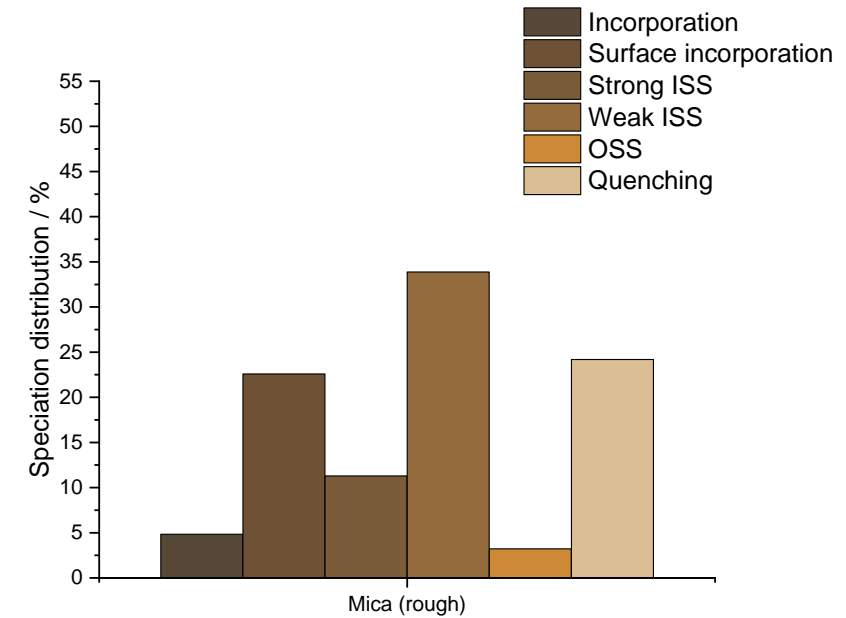
Quartz

Speciation largely depended on surface roughness



Feldspar

Trend towards stronger complexation (not as prominent)



Mica

Speciation depended on how many sorption sites are taken

Presumably: strong sites are occupied first, then weak sites

pH dependent sorption on different crystalline rocks

	Granitic pegmatite pH 6.83	Granite pH 8	Gneiss pH 8
Quartz:	598 – 605 nm (598 – 600; 602 – 605)	602 – 605 nm	602 – 605 nm
Feldspar:	599 – 604 nm (599 – 600; 601 – 604)	602 – 605 nm	602 – 605 nm
Mica:	598 – 605 nm (598 – 601; 602 – 605)	603 – 604 nm	

- Tendency of **weaker sorption** strength at **lower pH**
- Sorption **primarily** occurs on **hot spots** (mica)
- Almost no sorption on feldspar and quartz (compared to granite/gneiss) except for high roughness regions

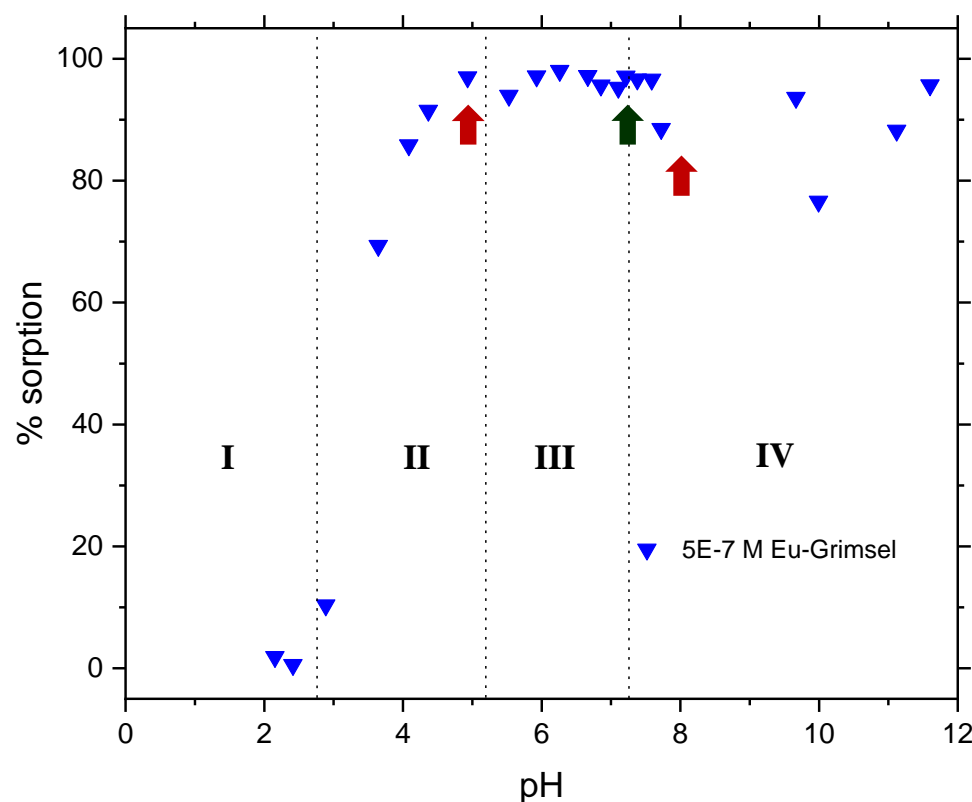
Planned: Cm(III) sorption on Grimsel sample

WIP: Cm(III) sorption on Grimsel sample

$I = 0.0012 \text{ M NaCl}$
 $[Cm(III)] = 5 \cdot 10^{-7} \text{ M}$

$pH = 7.30$
 $t = 7 \text{ d}$

Most likely granodiorite/mylonite/fault gouge



$I = 0.0012 \text{ M}$

Two pH values on same sample:

1.) pH 5.0

2.) pH 8.0

Effect of pH dependent change of sorption preference and speciation

Strong buffering effect shifted **pH from 5.0 to 7.3**
(low ionic strength?)

WIP: Cm(III) sorption on Grimsel sample

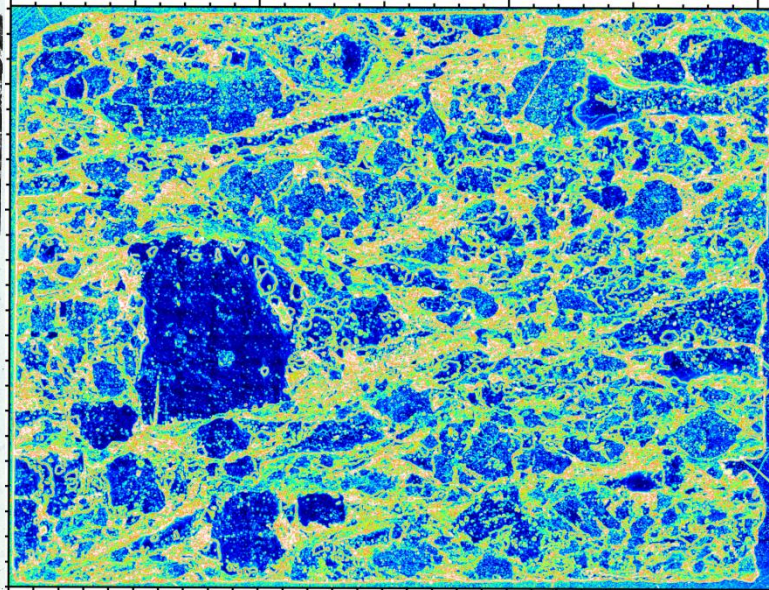
$I = 0.0012 \text{ M NaCl}$
 $[\text{Cm(III)}] = 5 \cdot 10^{-7} \text{ M}$

$\text{pH} = 7.30$
 $t = 7 \text{ d}$

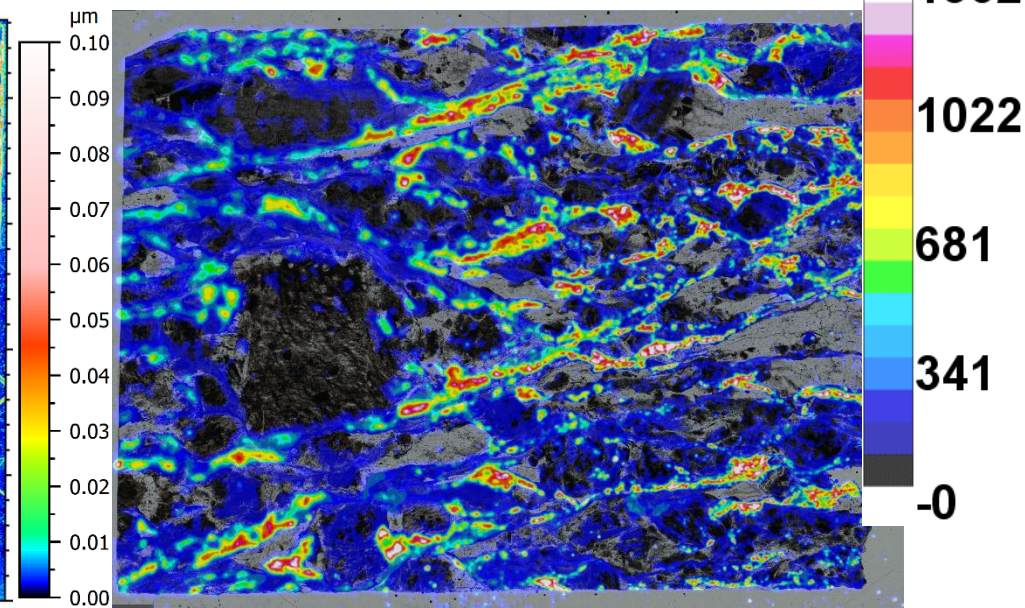
Mineral composition



Surface roughness



Cm(III) uptake / pmol/cm²



Preliminary results:

- Major sorption preference on black mineral phases (so far identified as different mica)
- Little sorption on feldspar
- Almost no sorption on quartz

pH dependent Cm(III) sorption studies on granitic pegmatite and Grimsel rock

pH dependent Cm(III) sorption studies on crystalline rocks

	Granitic pegmatite pH 6.83	Granite pH 8	Gneiss pH 8
Quartz:	598 – 605 nm (598 – 600; 602 – 605)	602 – 605 nm	602 – 605 nm
Feldspar:	599 – 604 nm (599 – 600; 601 – 604)	602 – 605 nm	602 – 605 nm
Mica:	598 – 605 nm (598 – 601; 602 – 605)	603 – 604 nm	

- Tendency of **weaker sorption** strength at **lower pH**
- Sorption **primarily** occurs on **hot spots** (mica)
- Almost no sorption on feldspar and quartz (compared to granite/gneiss) except for high roughness regions
- Speciation dependency on surface roughness:

Quartz > Feldspar > Mica

