Life Cycle of Critical Metals:
Compositional quantification of REE-bearing materials

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Compositional quantification of REE-bearing materials

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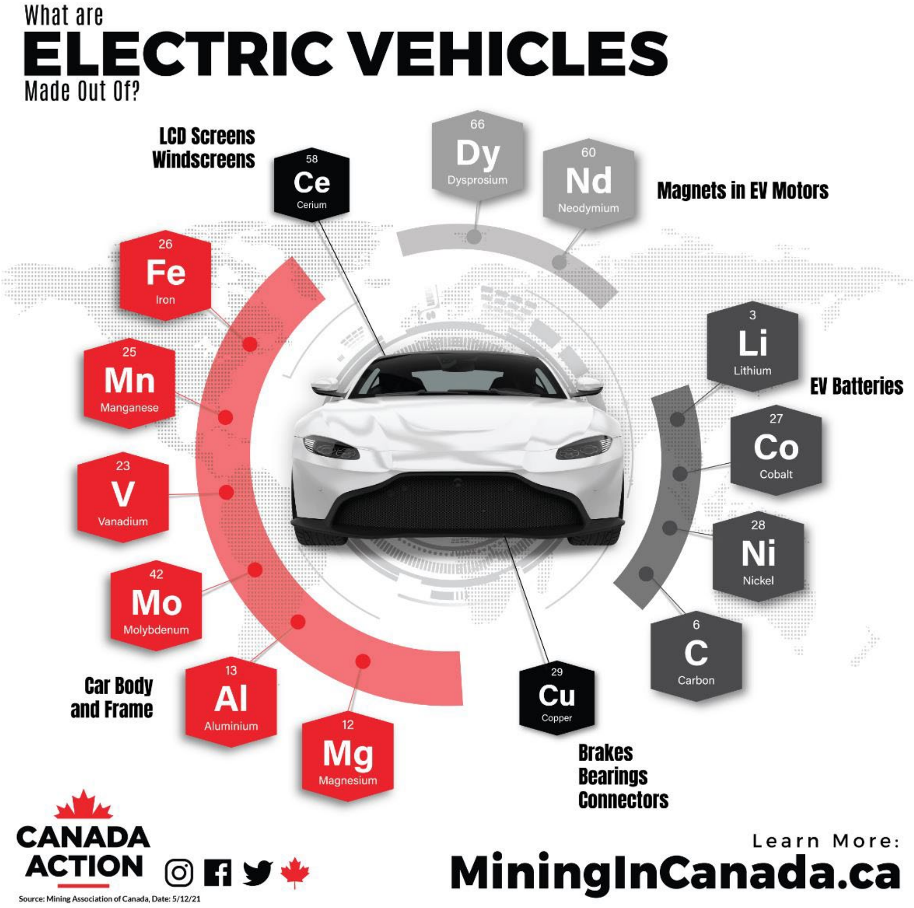
Critical Metals and the Green Technology Future

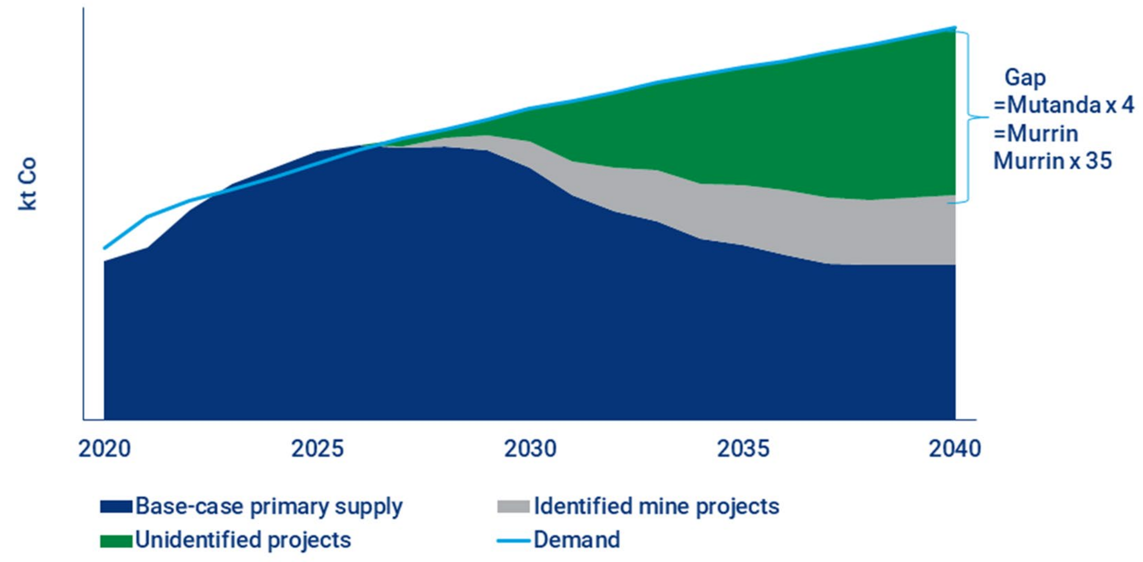
- Transition to the new technologies, lower carbon footprints, and a more sustainable future requires increased supply of metals
  - Battery metals
  - Magnets
  - Touch screens, glass
  - Body / frame materials
  - Wiring for technology and infrastructure



Reproduced from: Canada Action (https://www.canadaaction.ca/canada-electric-vehicle-tech-minerals-opportunity); original work by: Jason Burton

Critical Metals and the Green Technology Future

- One issue we face is the projected gap between existing and projected production from known resources
- To improve this position, several strategies can be considered
  - Discovery of new resources
  - Improved and efficient extraction as by-product metals in deposits of other commodities
  - Re-mining of waste and tailings for commodities previously not considered of economic importance
  - Recycling



Source: Wood Mackenzie

Example of the projected gap between existing and projected supply of cobalt
Similar short-falls are predicted for other commodities critical to green technologies

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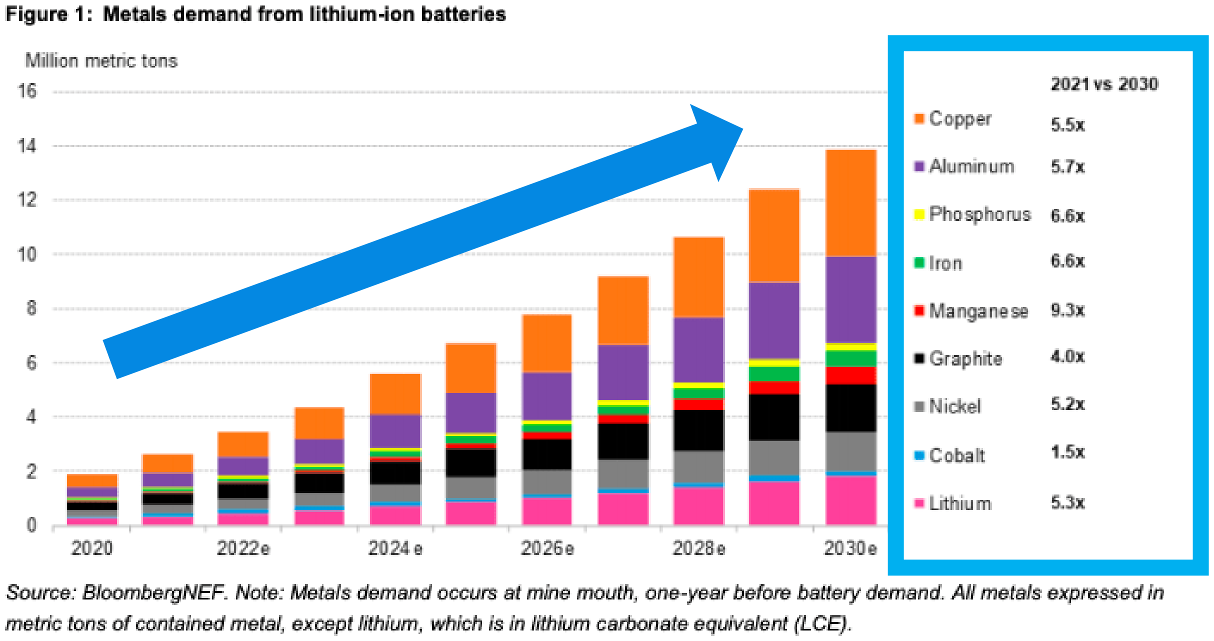
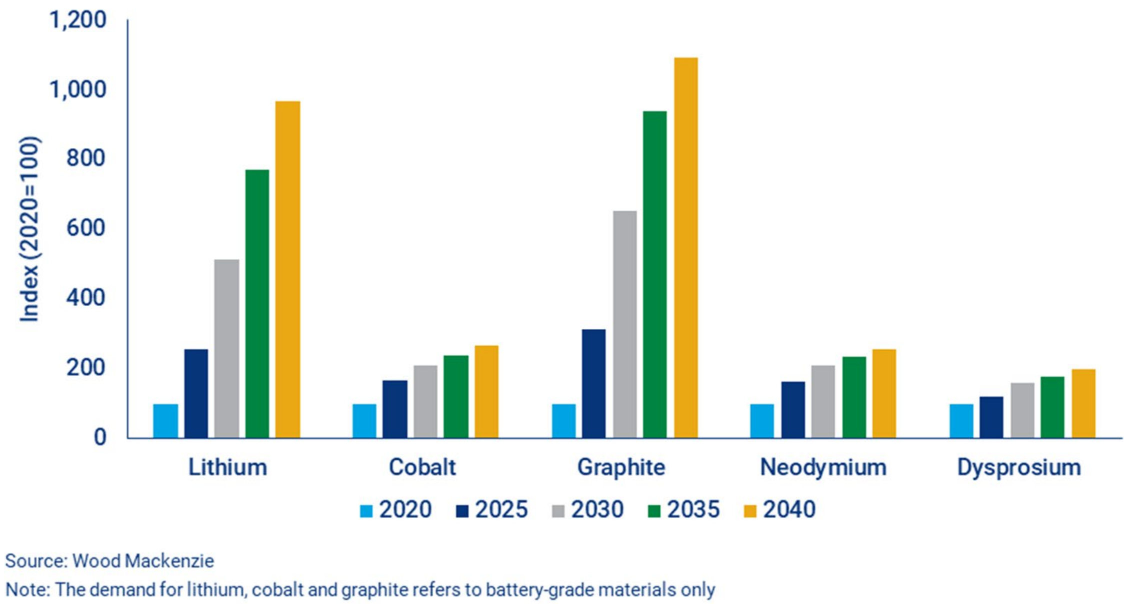
Importance of Critical Metals to the Green Technology Future

Lithium-ion Battery Requirements



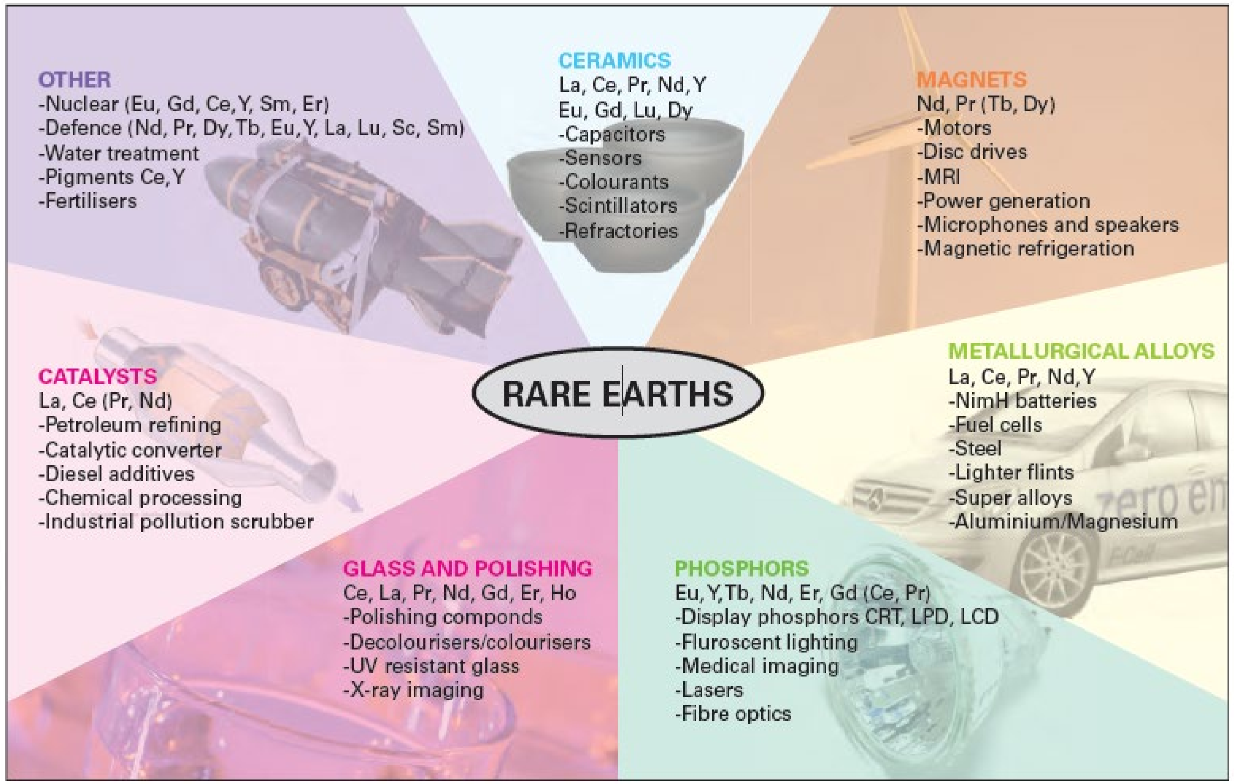
Source: BloombergNEF

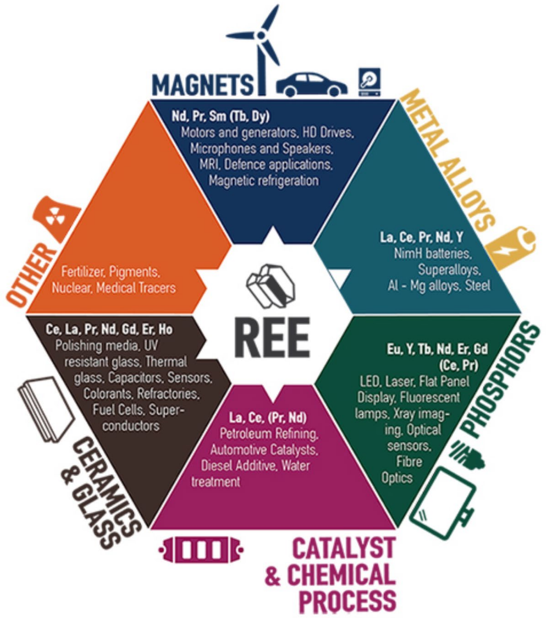
Demand outlook for key transition metals



Source: Wood & Mackenzie, 2022 report, https://www.woodmac.com/news/opinion/energy-transition-metals-the-esg-dilemma/

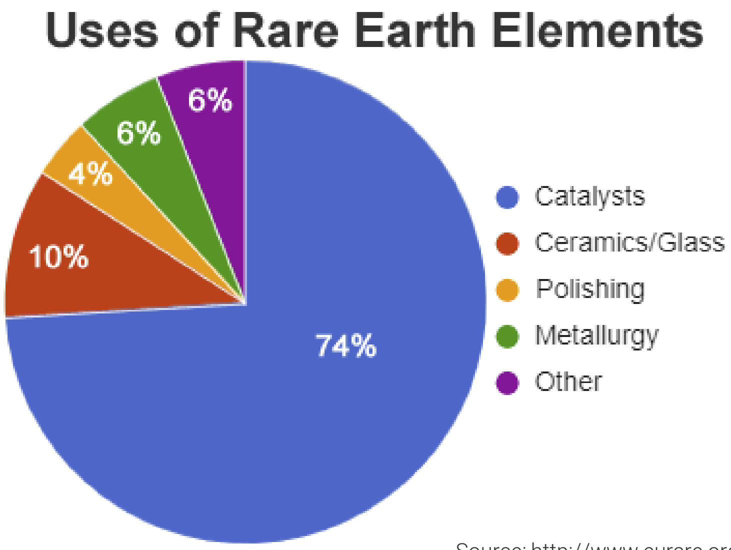
Rare Earth Elements (REE´s): Essential to modern-day technologies





metallic state

oxidized state

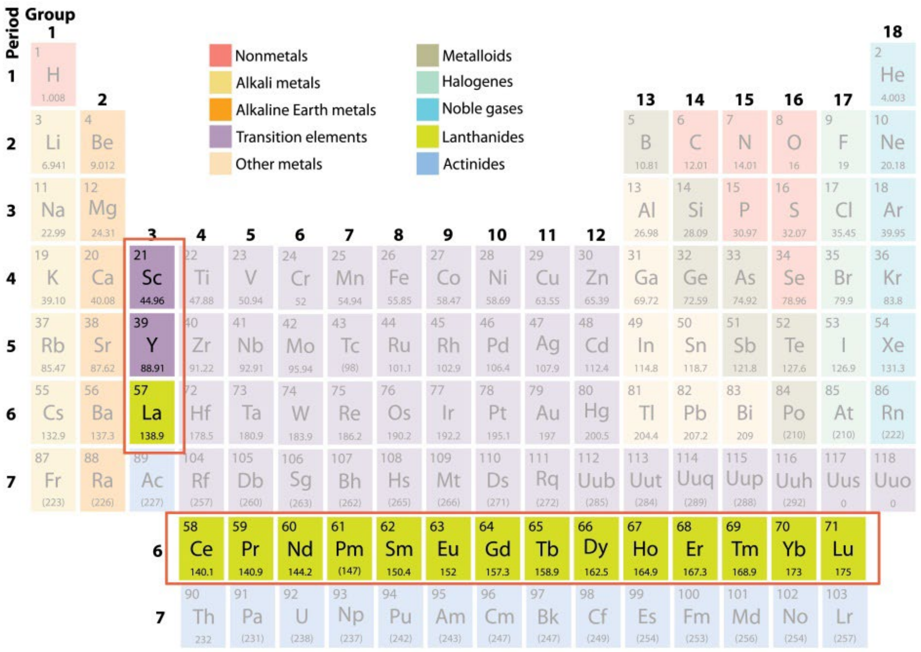


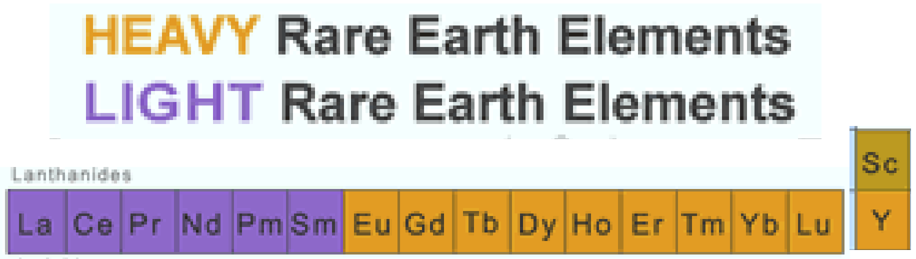
Source: http://www.eurare.org/RareEarthElements.html

Source: https://geology.com/articles/rare-earth-elements/

Rare Earth Elements (REE´s): **Introduction**

- Group of 17 chemically similar metallic elements, including the 15 lanthanides as well as scandium and yttrium.
- Chemically similar: easily substitute for each other and often occur together within various minerals
- Earth’s crust abundance of < 10 ppm (Rudnick et al., 2003)
- Do NOT occur naturally as metallic elements, but
- Do occur in a wide range of mineral types, for example halides, carbonates, oxides, phosphates and silicates.





Source: https://geology.com/articles/rare-earth-elements/

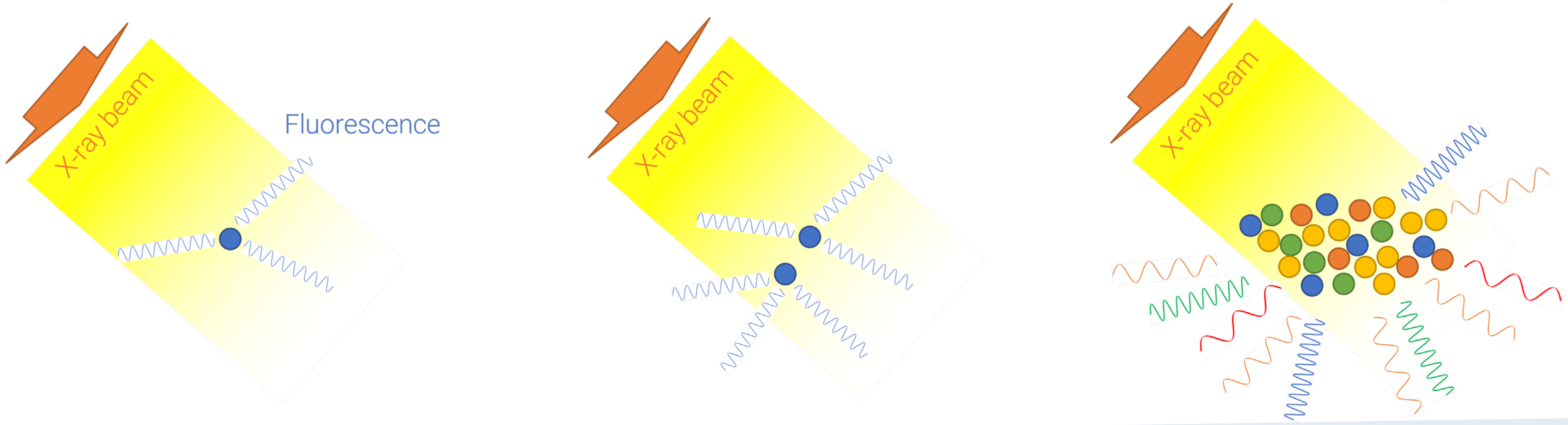
15 lanthanides
Lanthanum [La]
Cerium [Ce]
Praseodymium [Pr]
Neodymium [Nd]
Promethium [Pm]
Samarium [Sm]
Europium [Eu]
Gadolinium [Gd]
Terbium [Tb]
Dysprosium [Dy]
Holmium [Ho]
Erbium [Er]
Thulium [Tm]
Ytterbium [Yb]
Lutetium [Lu]
And
Scandium [Sc] and
Yttrium [Y]

WEBINAR: MICRO-XRF – REE QUANTIFICATION

What is micro-XRF?

Qualitative and quantitative X-ray fluorescence analysis
Counting atoms

- XRF is widely known for quantitative analysis. Why? Because it works so straightforwardly.
- An atom in an X-ray beam will produce element specific fluorescence radiation.
- Two atoms of the same type will produce twice as much fluorescence radiation.
- Many different atoms in the X-ray beam will all produce their characteristic fluorescence radiation.
  - Detecting the radiation with wavelength- or energy-dispersive detectors enables qualitative analysis.
  - Counting the number of incoming photons allows to **count the atoms** → quantitative analysis.



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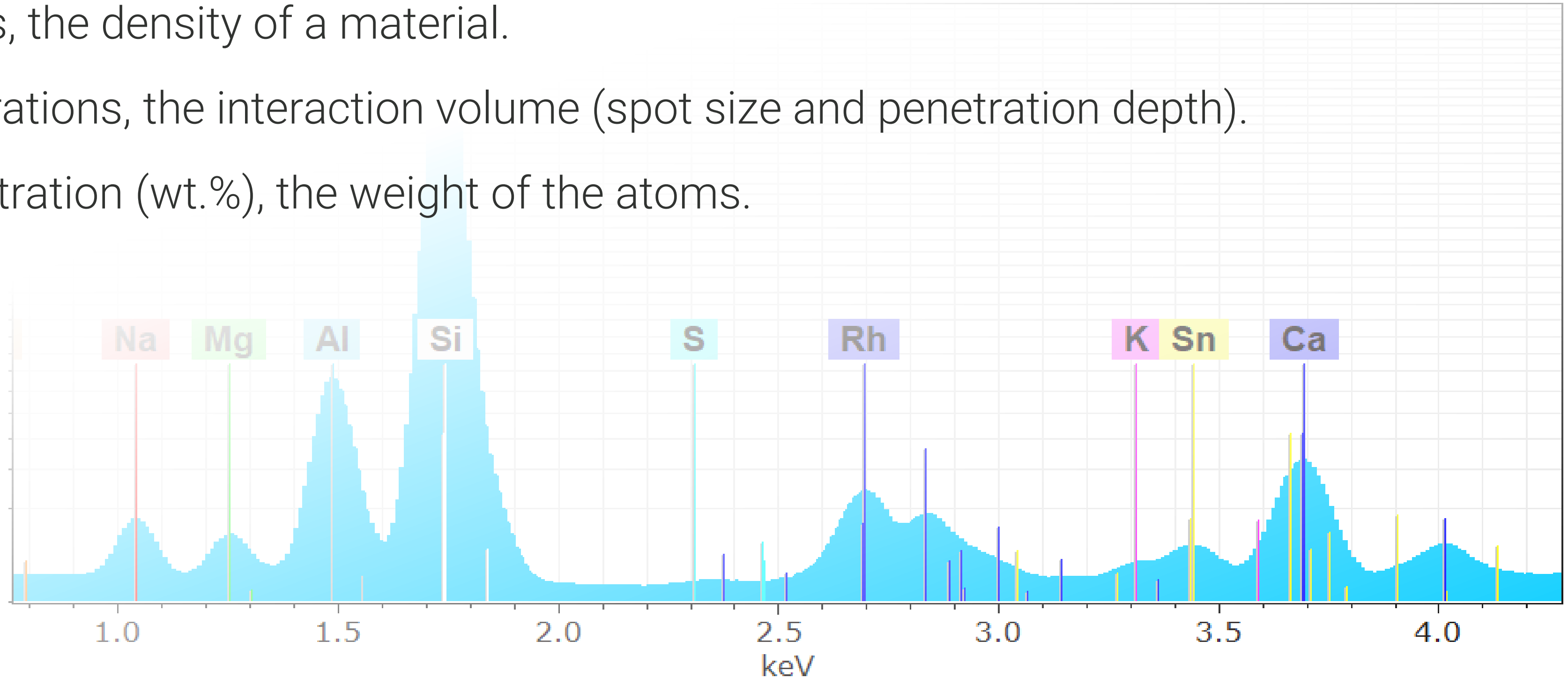
Quantitative X-ray fluorescence analysis
From atoms to wt.%

- With additional information, it is possible to convert this number of "atoms in an X-ray beam" to meaningful units:
  - To get the mass coverage, the size of the irradiated area.
  - To get a layer thickness, the density of a material.
  - To get atomic concentrations, the interaction volume (spot size and penetration depth).
  - To get to mass concentration (wt.%), the weight of the atoms.



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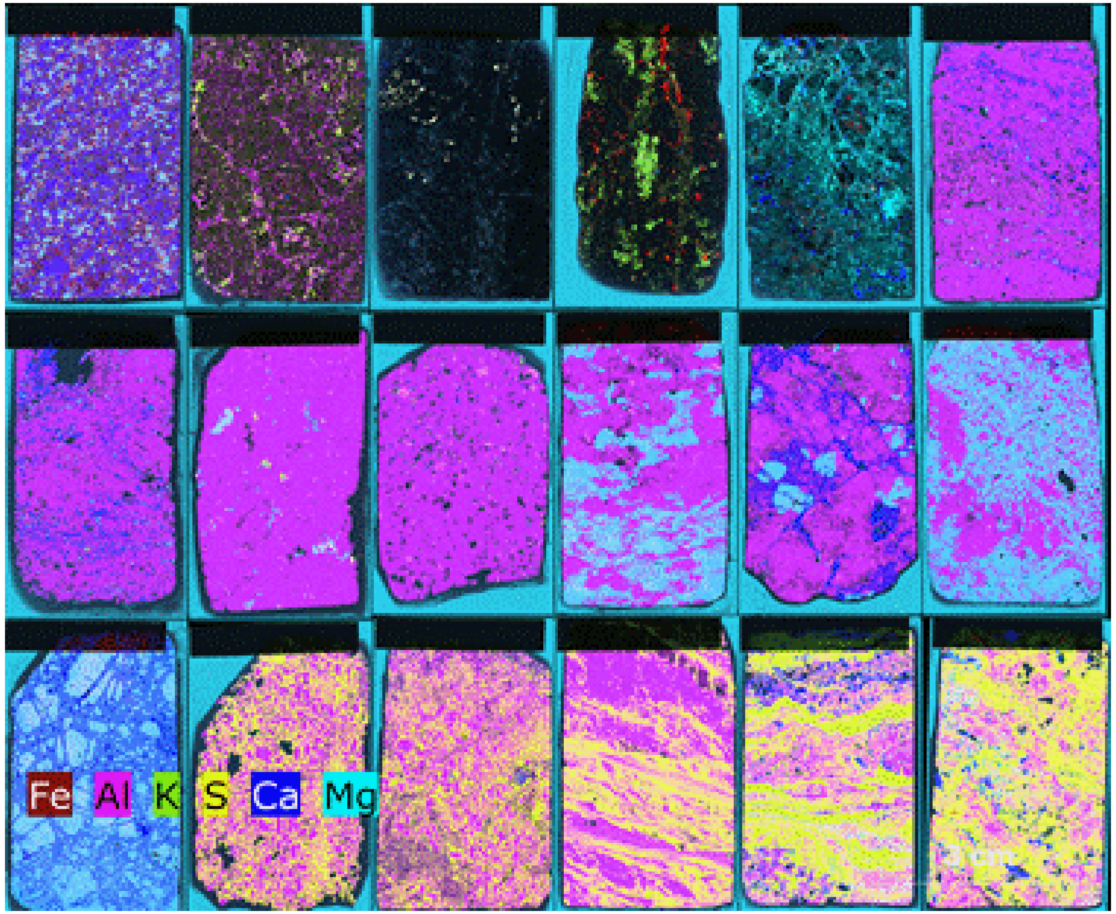
Micro-XRF

- Micro-XRF is XRF with a small spot (typically < 20 µm)

  → Micro-XRF reveals where elements are

  → Micro-XRF is ideal for non-homogeneous samples
- It usually requires minimal or no sample preparation.
- Quantitative micro-XRF is feasible for sufficiently homogeneous areas of the sample, which can be even below 100 µm in diameter.
- The measurement conditions are very flexible in order to address different analytical tasks or requirements posed by the sample.



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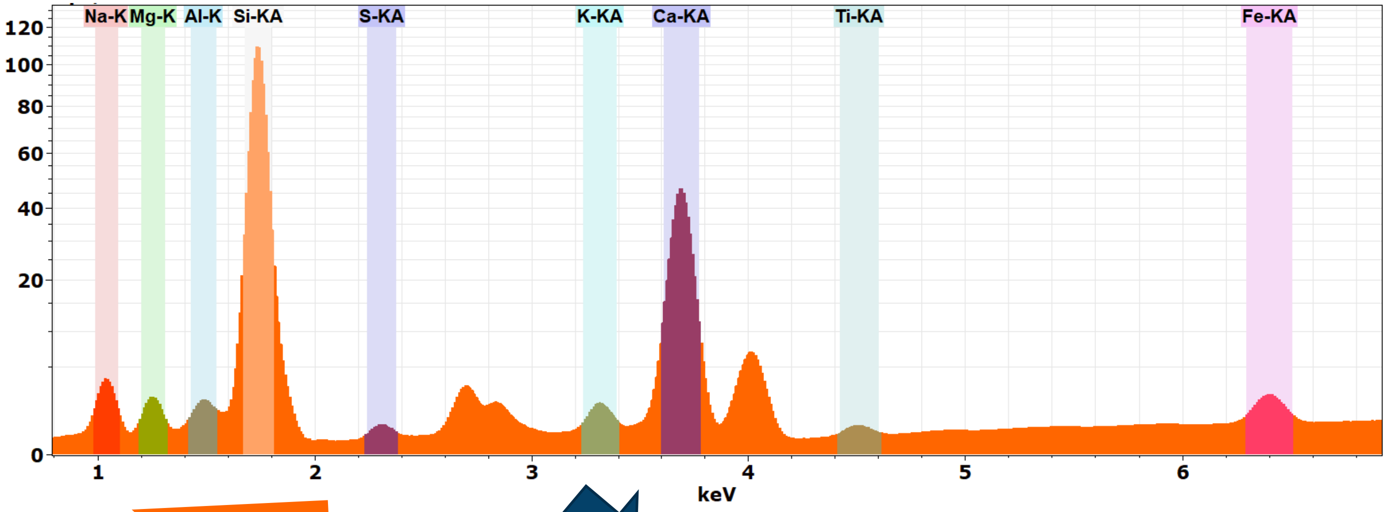
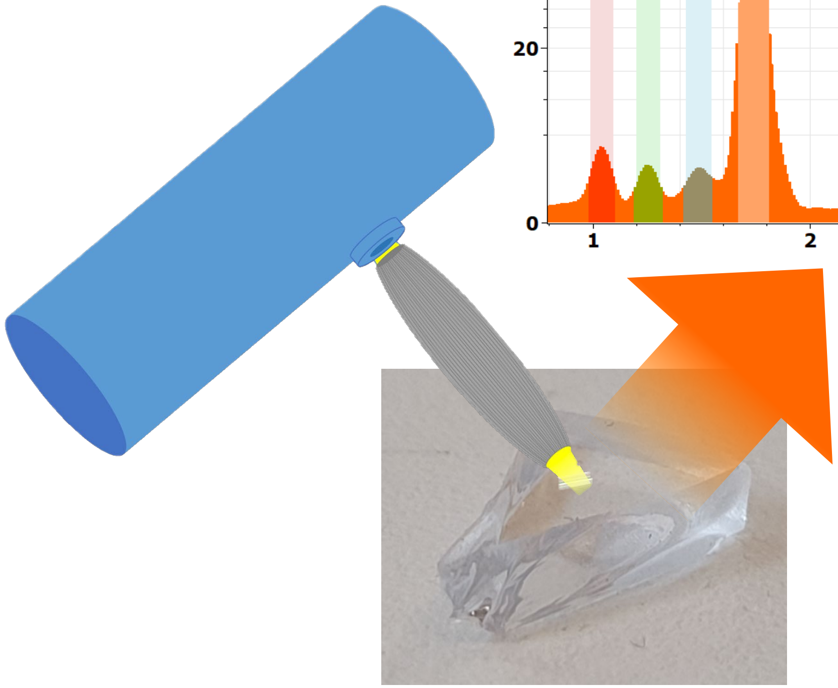
WEBINAR: MICRO-XRF – REE QUANTIFICATION

Optimising REE Quantification via Micro-XRF

Quantification techniques in XRF
Flow of Information





Element	AN	Series	Net intensity [cps]
O	8	K series	0.00
Na	11	K series	340.84
Mg	12	K series	172.30
Al	13	K series	153.60
Si	14	K series	12481.62
S	16	K series	49.57
K	19	K series	184.71
Ca	20	K series	7147.86
Ti	22	K series	56.83
Mn	25	K series	10.70
Fe	26	K series	327.96
As	33	K series	6.21
Rb	37	K series	17.30
Sr	38	K series	32.57
Zr	40	K series	23.02

Element	AN	Series	Compound	norm. stoich. C [wt.%]
O	8	K series		0.000
Na	11	K series	Na2O	14.282
Mg	12	K series	MgO	3.870
Al	13	K series	Al2O3	1.521
Si	14	K series	SiO2	71.338
S	16	K series	SO3	0.175
K	19	K series	K2O	0.351
Ca	20	K series	CaO	8.331
Ti	22	K series	TiO2	0.045
Mn	25	K series	MnO	0.003
Fe	26	K series	Fe2O3	0.076
As	33	K series	As2O3	0.001
Rb	37	K series	Rb2O	0.002
Sr	38	K series	SrO	0.003
Zr	40	K series	ZrO2	0.003

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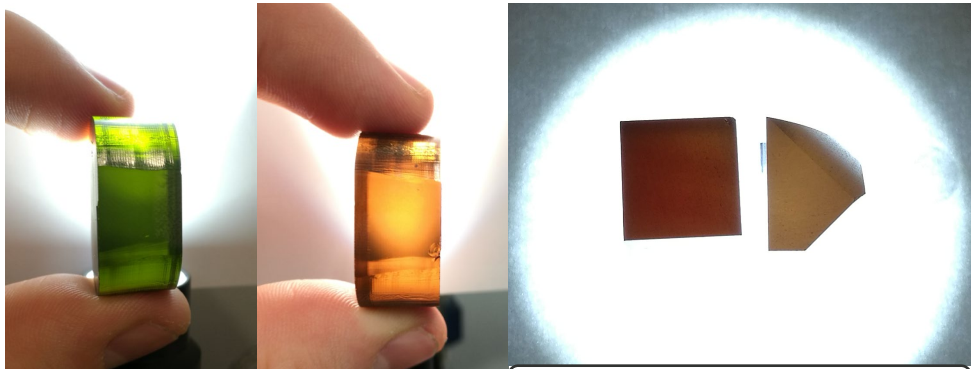
Optimizing measurement conditions for quantification of *REE*
Filters

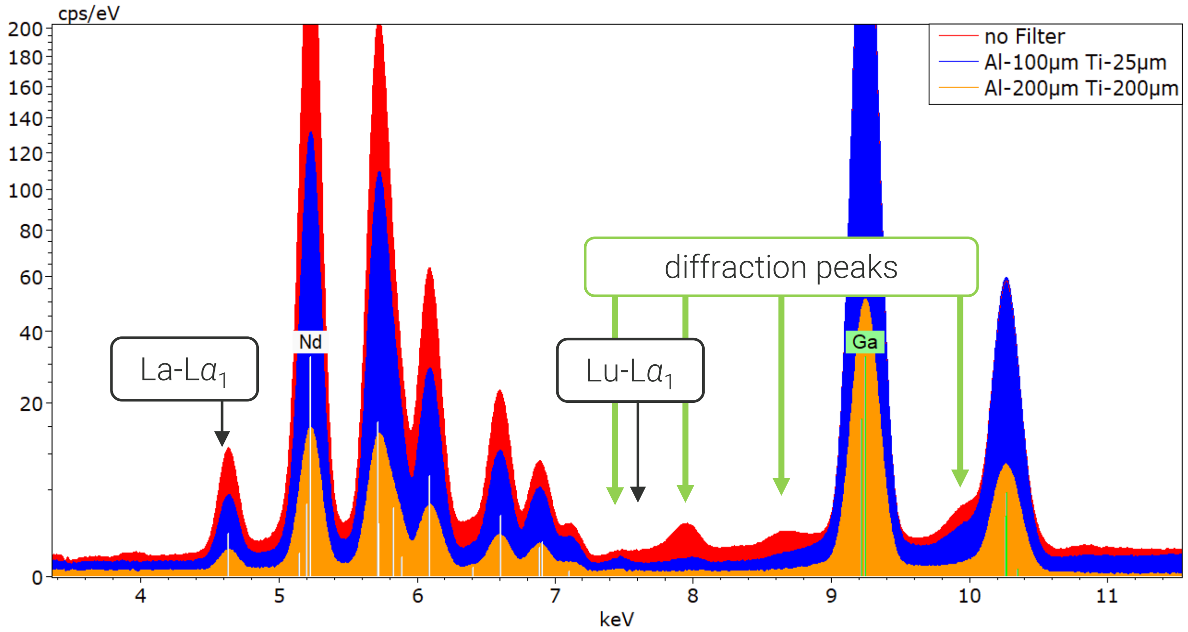
Materials are often crystalline. XRF spectra will contain both fluorescence peaks and diffraction peaks!

→ Problem for accurate quantification: intensity from diffraction peaks cannot be separated.

→ Filters should be used to inhibit diffraction.



NdGaO_3 single crystals



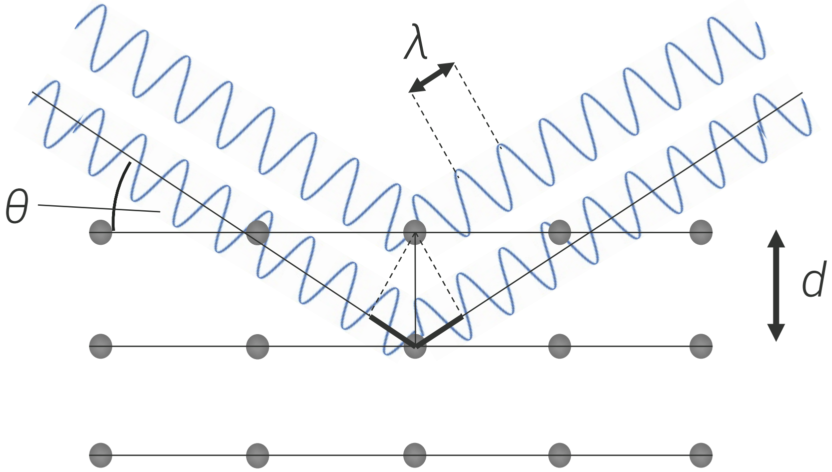
Comparing filter materials:

→ An Al-200µm Ti-200µm filter inhibits diffraction peaks in the relevant energy range.

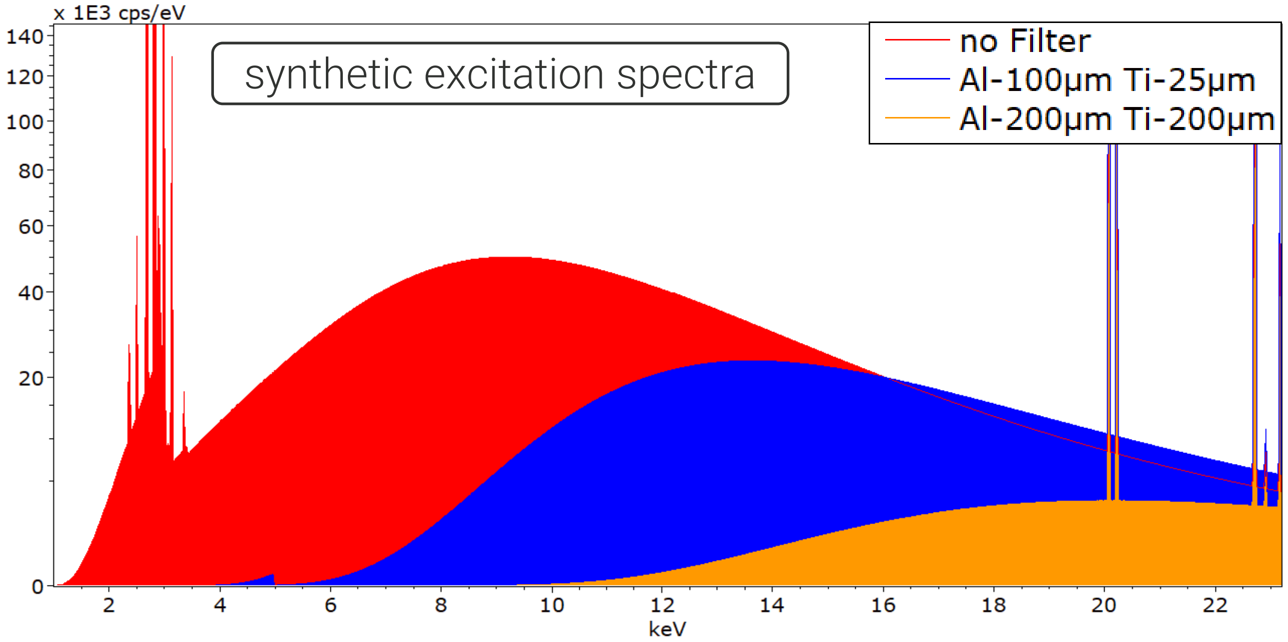
Optimizing measurement conditions for quantification of *REE*
Effect of filters on diffraction?

Diffraction requires that the glancing angle θ, X-ray energy $E = hc/\lambda$ and lattice spacing d follow Bragg's law.

However: The excitation spectrum is continuous!
→ X-rays with "correct" energy **will** be present!



$$n\lambda = 2d\sin\theta$$



Filters absorb lower-energy photons of excitation spectra.
→ No photons in these energy ranges to be diffracted.

Fluorescence can still be induced via higher energy X-rays.

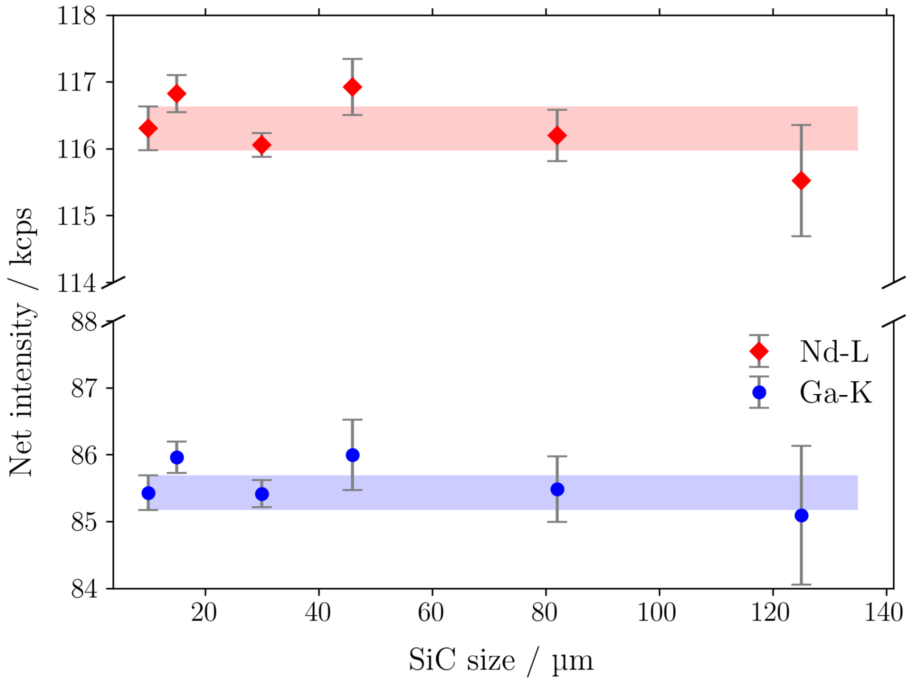
Optimizing measurement conditions for quantification of *REE*

Polishing – Implications for quantification?

$NdGaO_3$ surface with controlled roughness was prepared by polishing on SiC foils.

→ Net intensities do not change within the uncertainties.
→ The data scatter less with increasingly fine polishing.
→ Finer grit than paper with SiC size of 30 µm does not improve scatter further.

- $NdGaO_3$ single crystal.
- Average of 9 point measurements each.
- Error bars show ±1 standard deviation.



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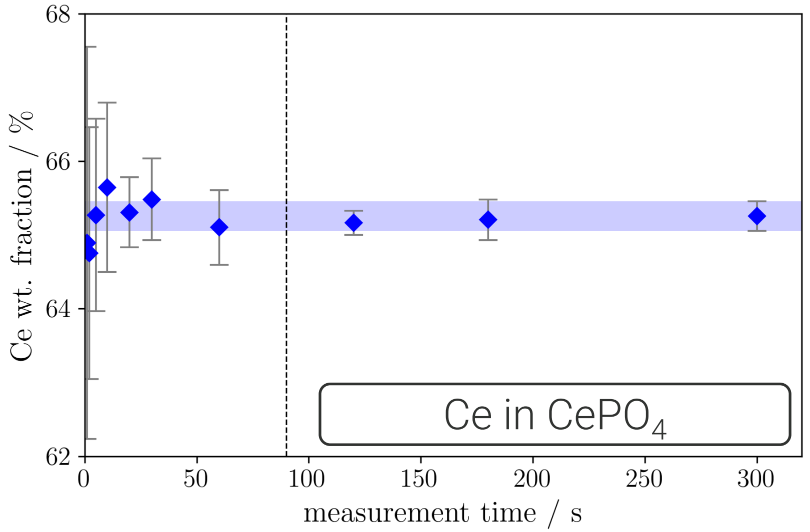
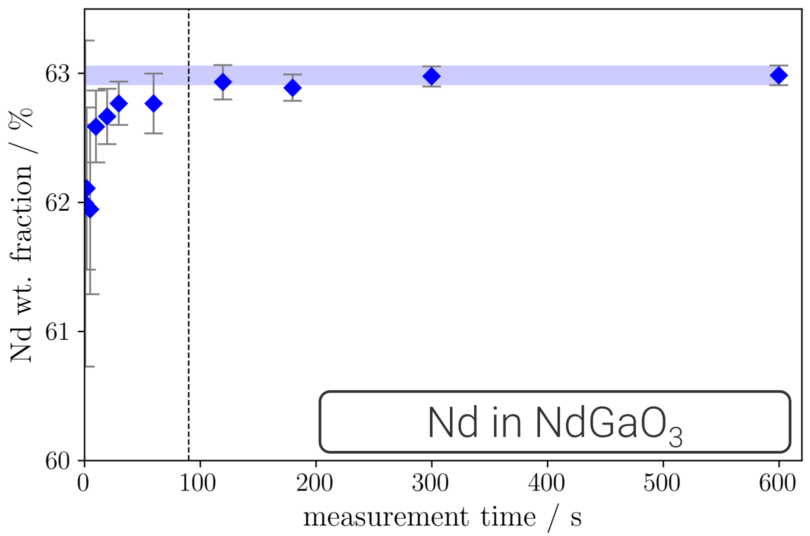
Optimizing measurement conditions for quantification of *REE*
Measurement time?

- Point measurements on $NdGaO_3$ and $CePO_4$ single crystals with varying real time.
- 10 measurements for each condition.
- Al-200µm Ti-200µm filter; appropriate surface finish.
- Quantification as oxides; Nd_2O_3 + Ga_2O_3 and Ce_2O_3 + P_2O_5.

→ Results converge with increasing measurement time.
→ For both materials, 90 seconds of real time appear sufficient to ensure accurate and precise results.





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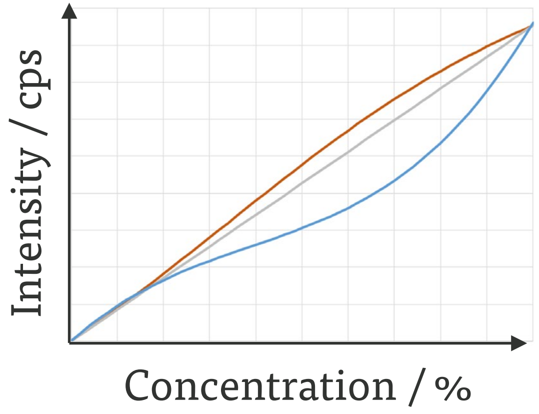
Quantification pathways in the M4 TORNADO

Fundamental Parameters (FP – M4 TORNADO)

- Forward calculation (M4 TORNADO): Intensities calculated based on iteratively-adjusted concentrations of elements, known physics, instrument, … .
- Calibration possible:
  - Correction for uncertainties of fundamental parameters and interactions with incomplete descriptions.
  - Compensation for deviations from idealized sample state.

Empirical (XMethod)

- Concentrations derived from comparing intensities of the sample and reference materials with known composition.



→ Empirical and FP based quantification both need reference materials for high accuracy/validation.
FP based quantification chosen here: higher flexibility, more convenient workflow.

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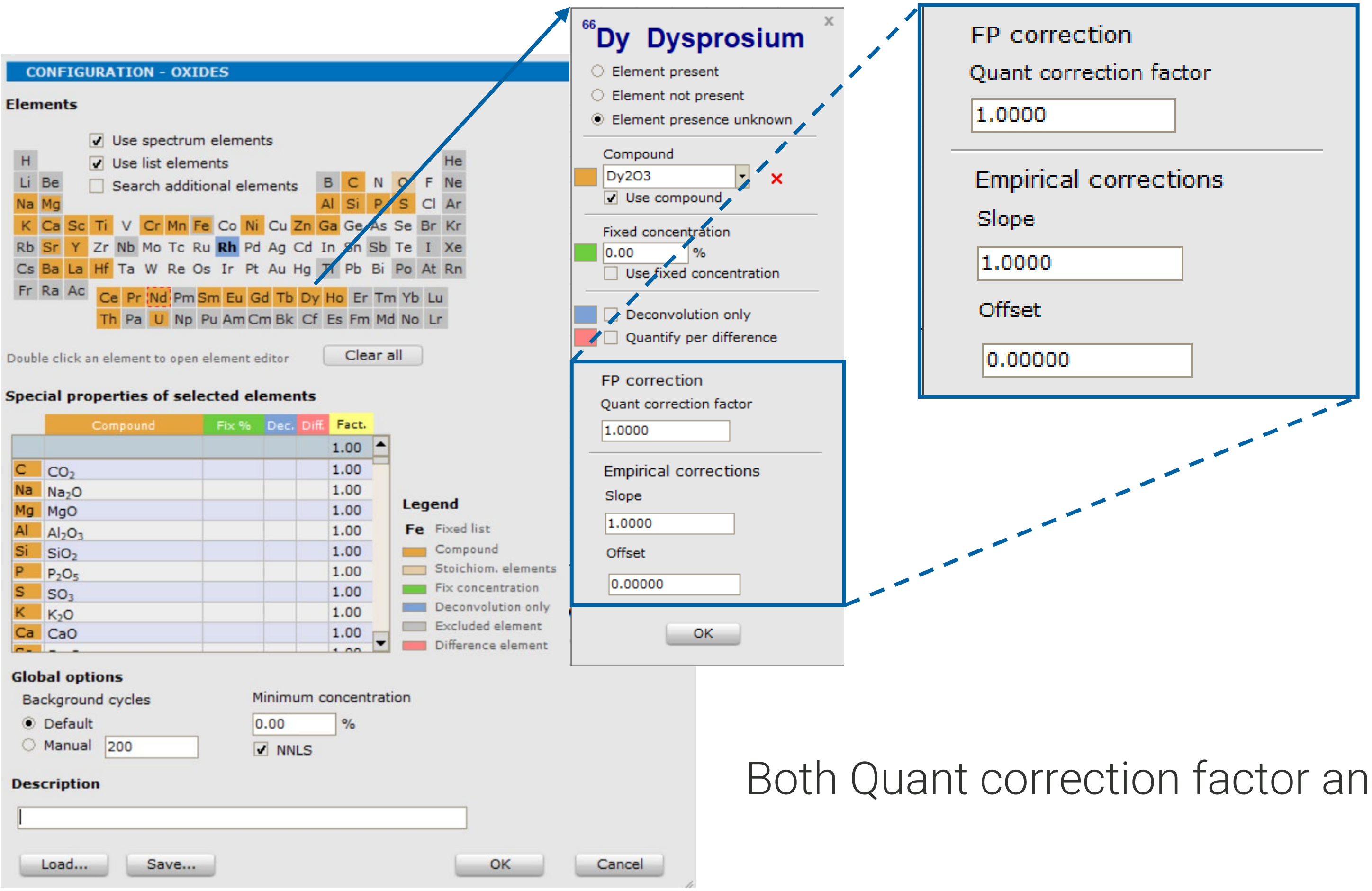
Calibrating FP based quantification in the M4 TORNADO



FP Quant correction factor:

- Adjusts sensitivities for specific transitions, which are part of the FP model.

Empirical corrections:

- Apply a linear correction to the quantified concentration after the FP quantification has concluded.

Both Quant correction factor and Empirical corrections can be used.

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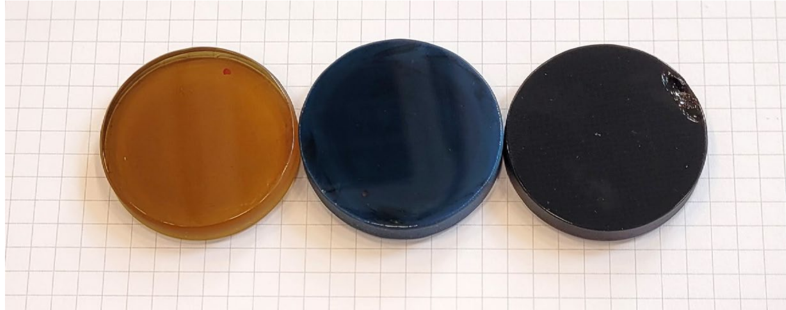
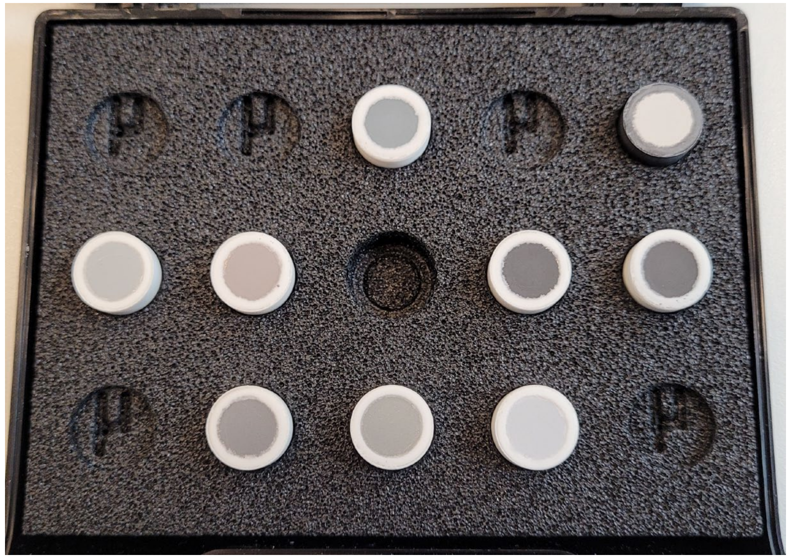
Reference Materials

Possible reference materials

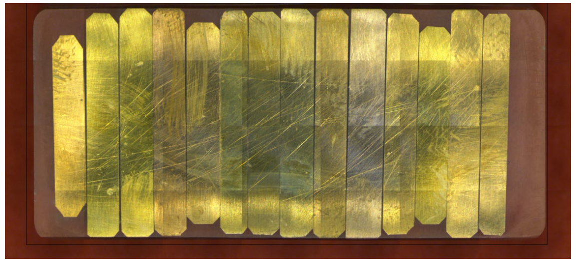
- Commercial reference materials
  → Do these exist for these materials?
- Materials with „known“ composition
  → In-house standards; composition from other method?
- Self-made reference materials.





Requirements:

- Composition similar to sample.
- Homogeneous in appropriate volume.
- Ideally certified.



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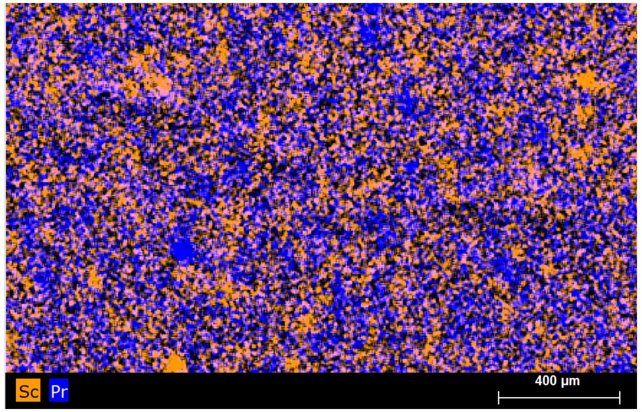
Creating your own reference materials

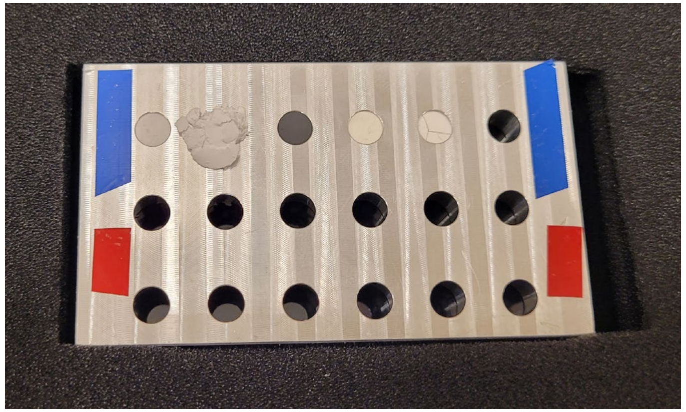
Pressed pellets as reference materials:

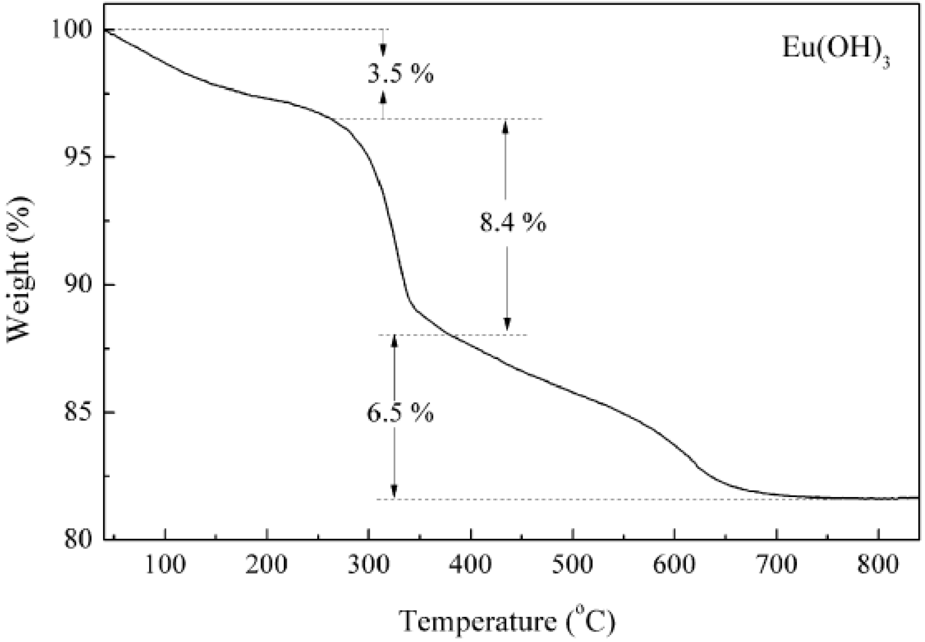
- Mixed according to approximate sample composition.

Possible issues:

- Non-homogenous powders: Nano-powders theoretically ideal, but does that reflect real sample?
- Possibly difficult to compact powders.
- Specifically, for RE_2O_3: often hygroscopic.







[1]

[1] Xin, Y. et al. (2010). J. Alloys Compd. 507, 105–111.

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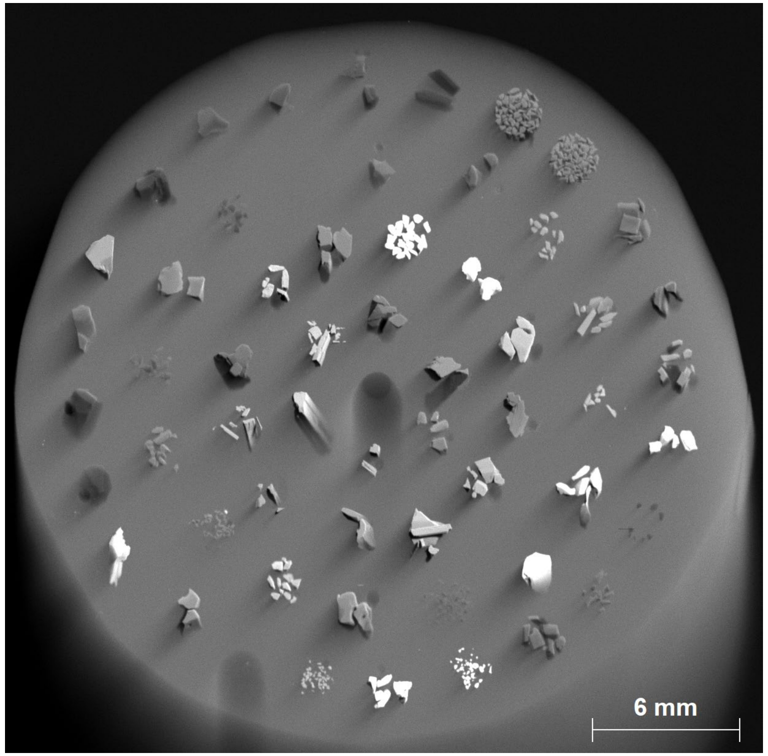
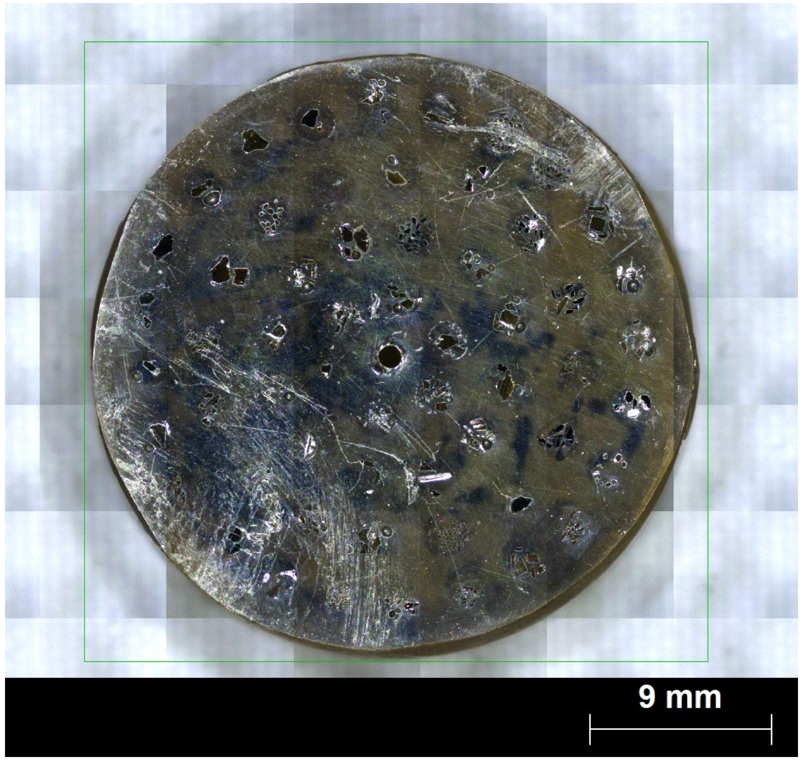
Smithsonian Microbeam Standards

Complete set of all *RE*PO_4 available as reference materials: Smithsonian reference samples for Microbeam Standards

Meant to be electron-microprobe standards!





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Smithsonian Microbeam Standards

Challenges:

- Meant for electron excitation.

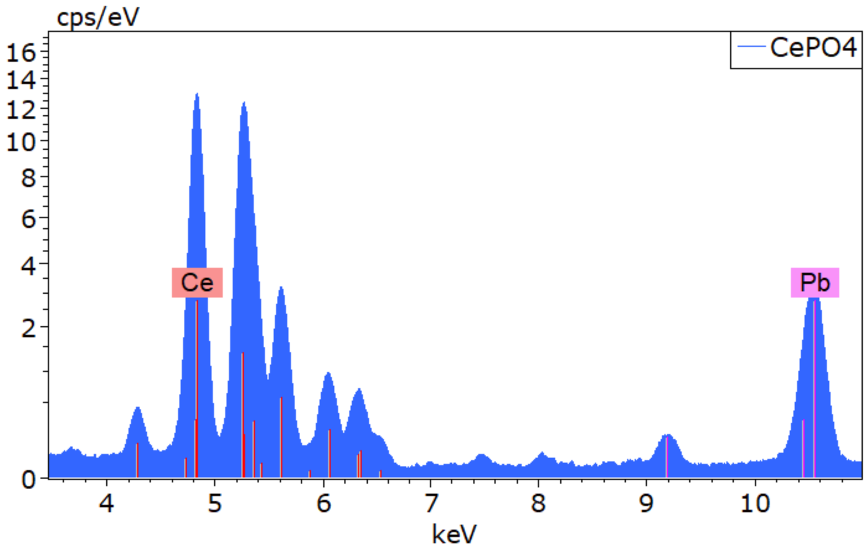
⟶ Low thickness.

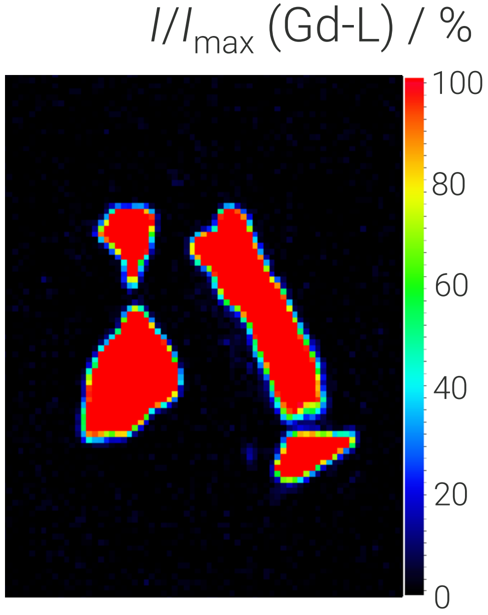
⟶ Area needs to be chosen carefully.

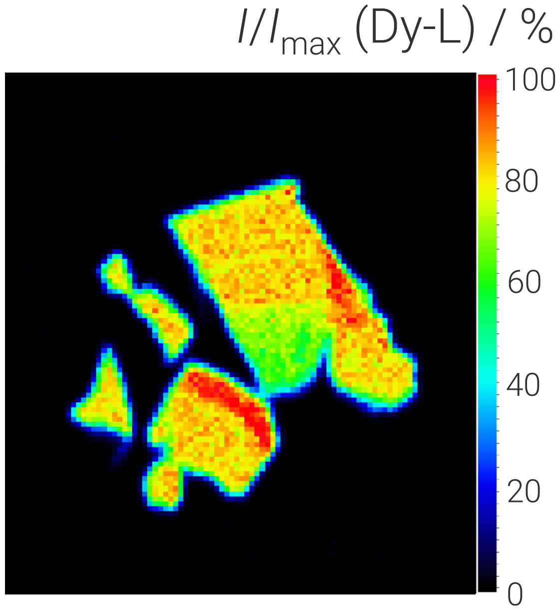
⟶ Sample may need to be rotated.

- Synthetic crystals grown from a $Pb_2P_2O_7$ flux.

⟶ varying Pb contamination.







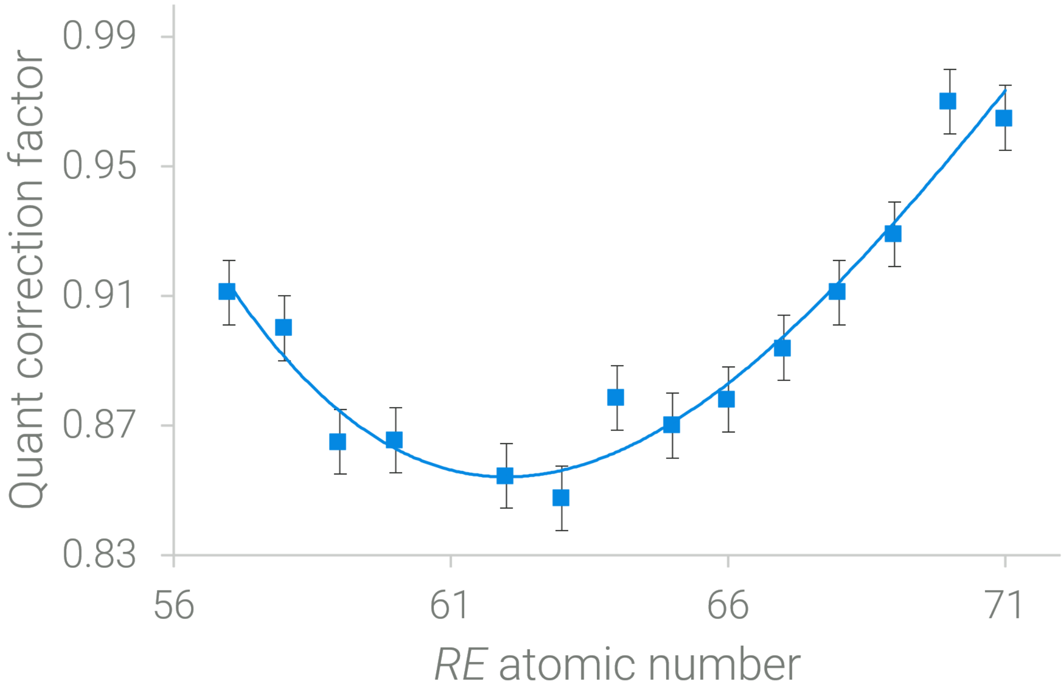
Quant correction factors: Trends within the *REE*

Based on Smithsonian Microbeam standards:

- Quant correction factors are similar for elements with similar cationic number.
- A clear trend for the Quant correction factor is observed.
- Reference materials have to be analyzed carefully.

→ Calibration factors for elements without standards may be estimated based on materials where there is no standard?

How valid is this approach?



57 La	58 Ce	59 Pr	60 Nd	61 Pm	62 Sm	63 Eu	64 Gd	65 Tb	66 Dy	67 Ho	68 Er	69 Tm	70 Yb	71 Lu

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Quant correction factors: Trends within the *REE*

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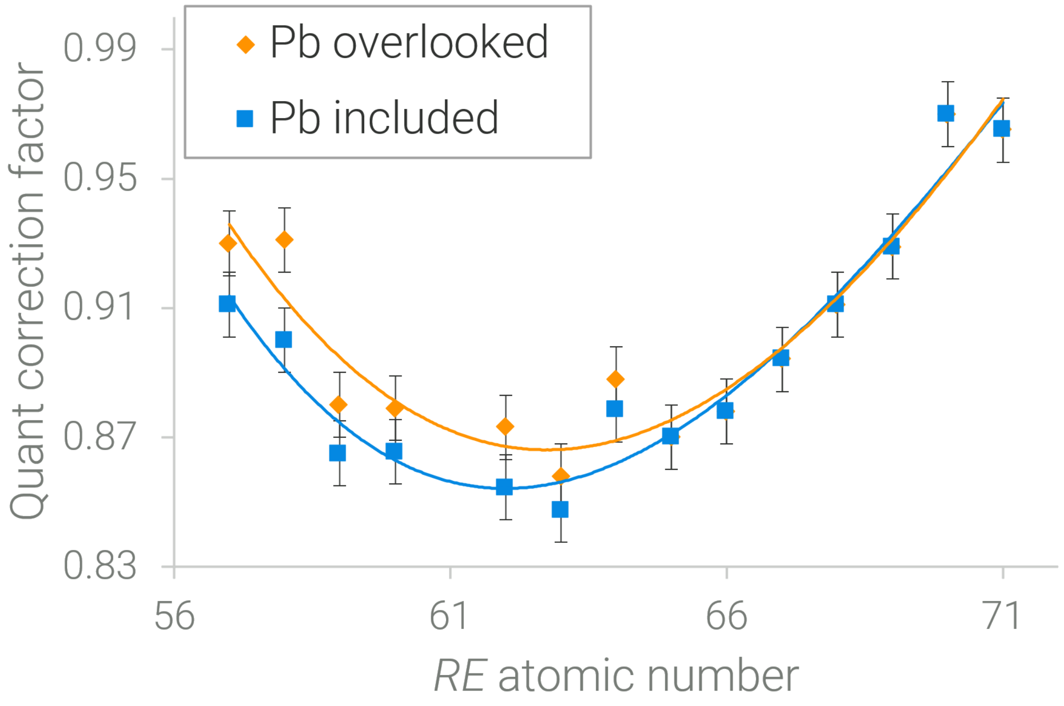

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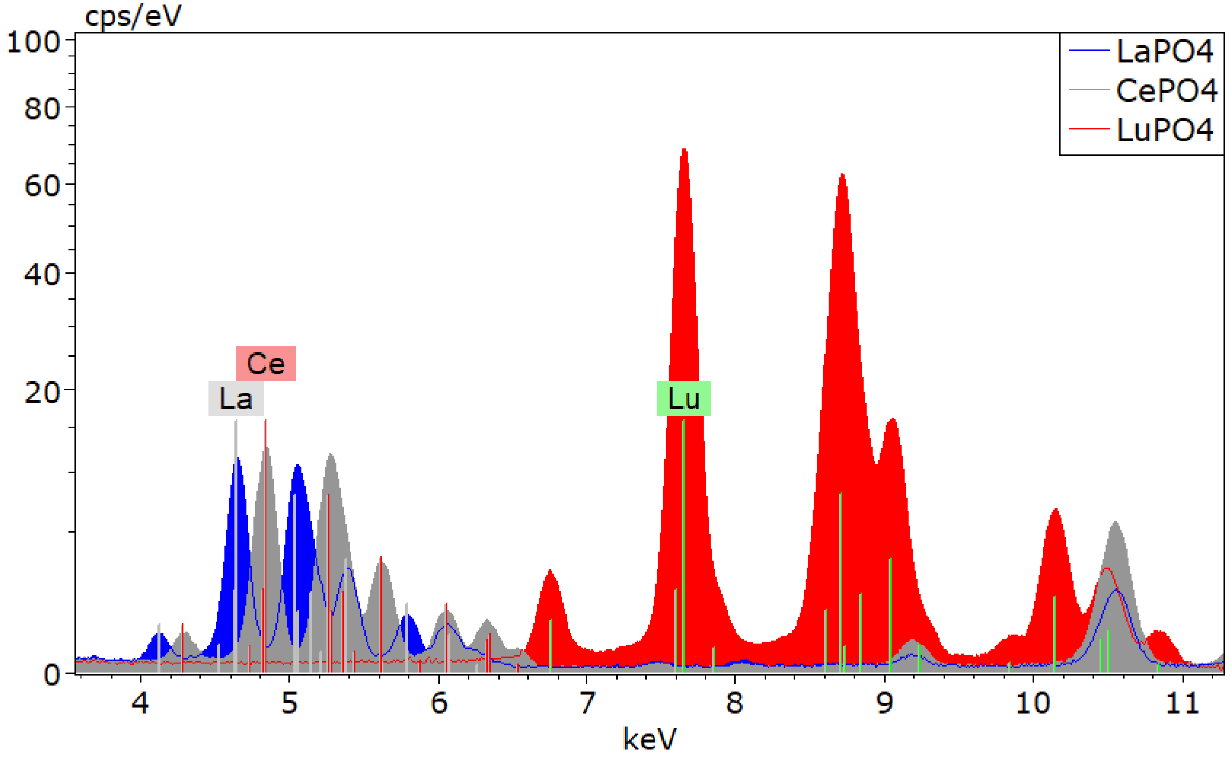
Quant correction factors: Trends within the *REE*

- Quantification of all 14 *RE* via L lines that fall into limited energy range.
- Absorption edges of neighboring *RE* are similar.

→ Good chances that physical effects that affect quantification have similar impact and can be similarly compensated for.

However: Entire spectra need to be considered!



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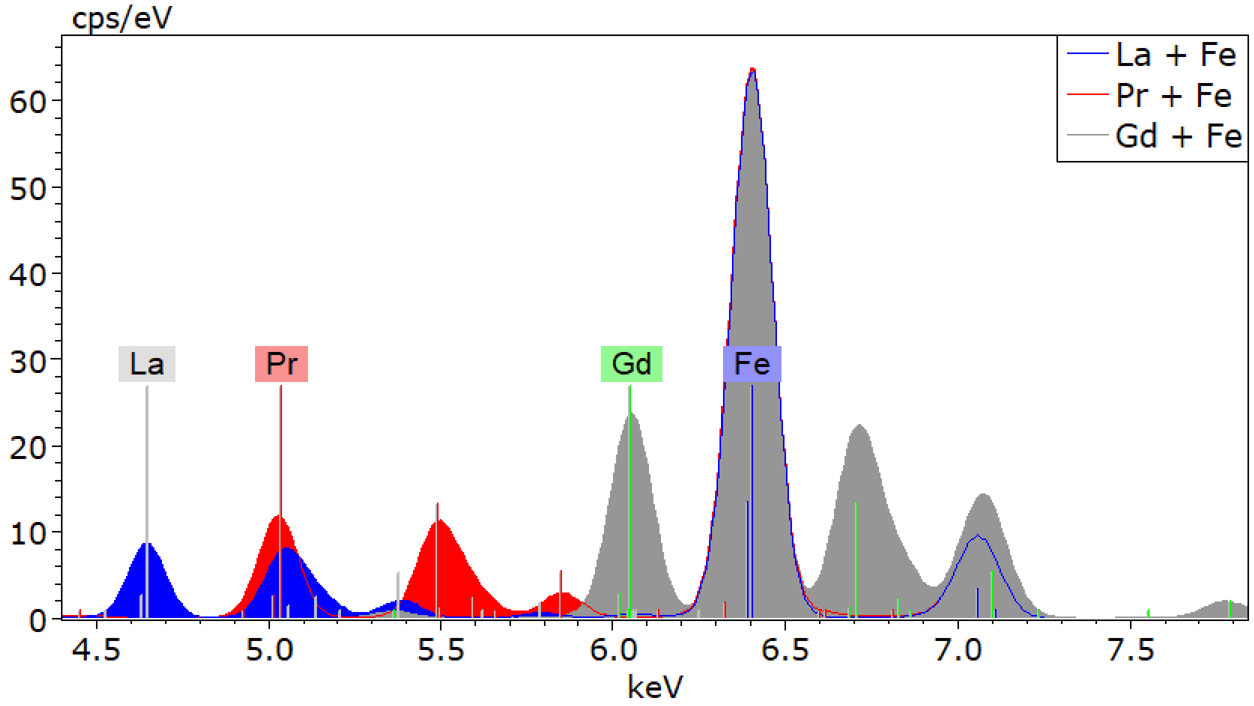
Quant correction factors: Trends within the *REE*

Samples with high Fe concentration

Fe X-ray energies:

- Fe-Kα: ~ 6.397 keV
- Fe-Kβ: ~ 7.058 keV

	L_I edge / keV	L_{II} edge / keV	L_{III} edge / keV
La	6.266	5.891	5.483
Pr	6.835	6.440	5.964
Gd	8.376	7.930	7.243



→ Both Fe lines can excite La via all three absorption edges, Pr via some edges and Gd via none.

→ Different inter-element effects in these systems; quant correction factors expected to be different.

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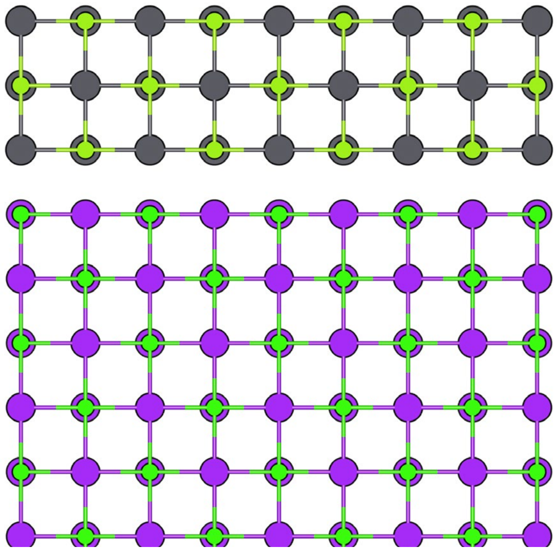
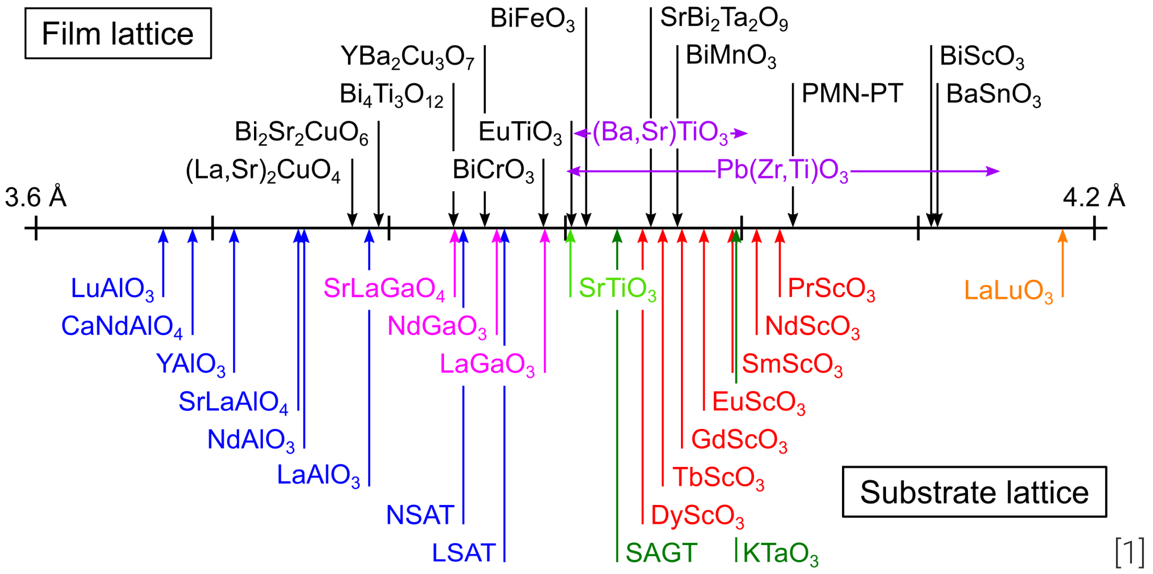
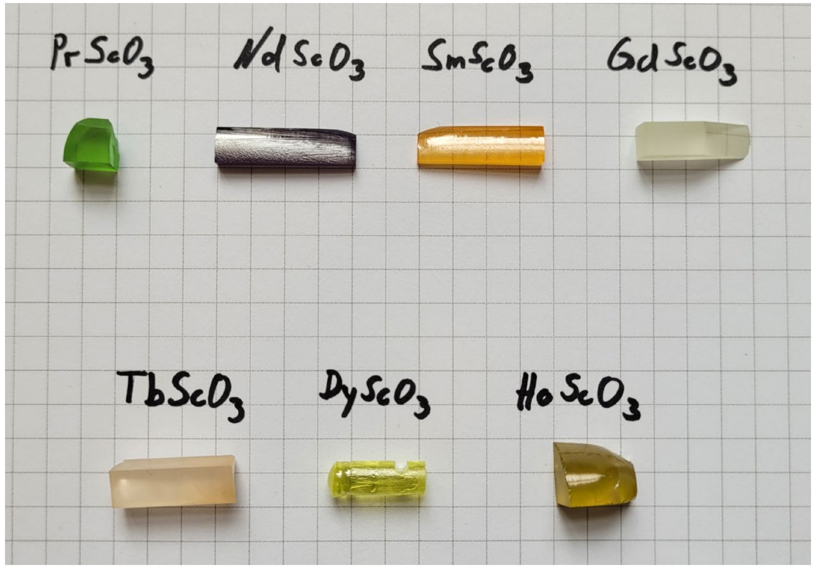
*RE*ScO_3: An alternative to *RE*PO4

- (Commercially) available as single crystals with perovskite-type structure for *RE* = Pr—Dy.
- Use as substrate material for (epitaxial) crystal growth for, e.g., ferroelectric/multiferroic materials.
- Exchanging the *RE* facilitates strain engineering.







[1] Hirschle (2021). PhD thesis. DOI: 10.13154/294-7970.

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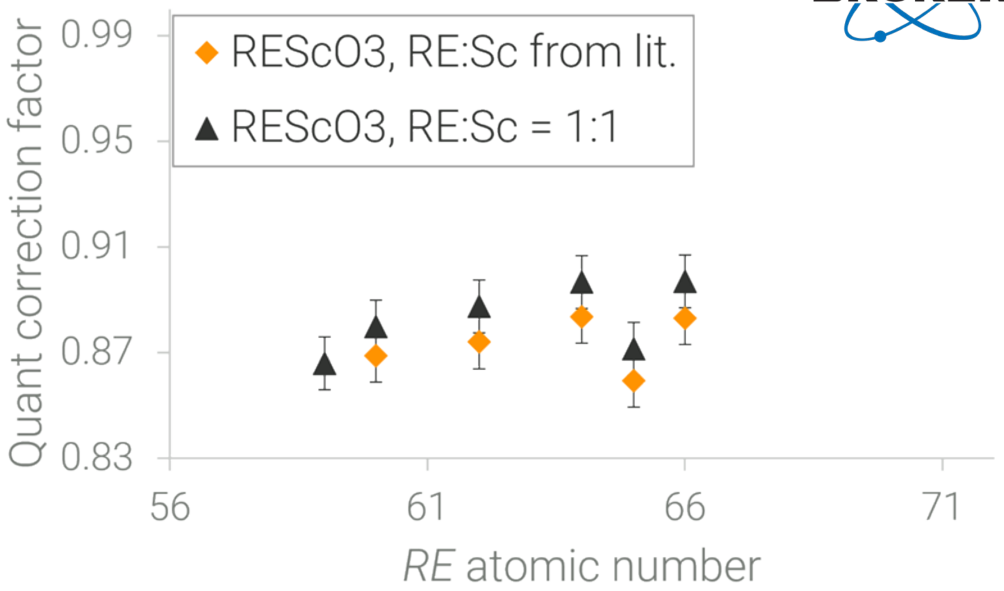
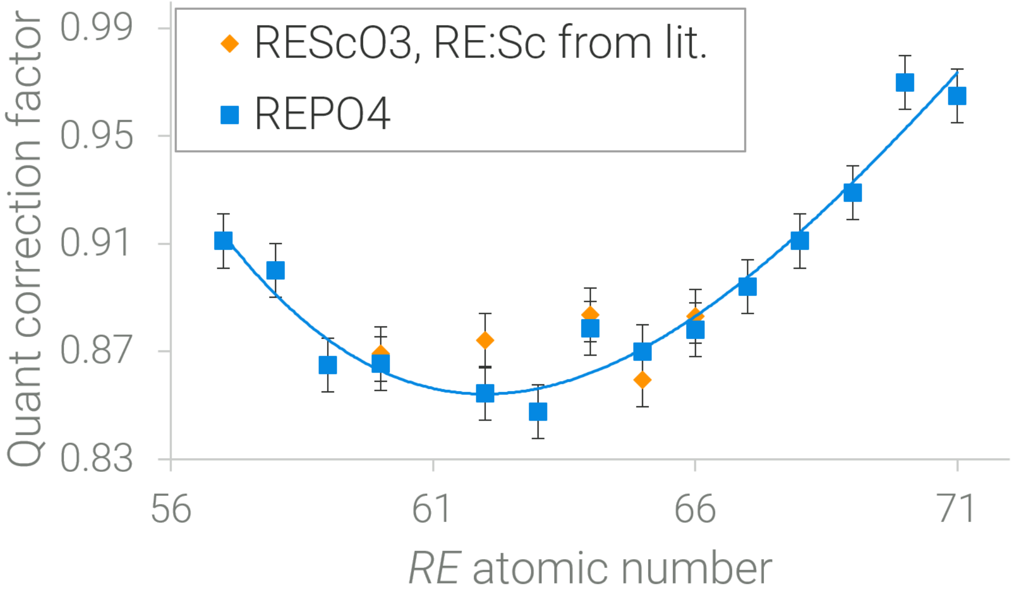
REScO_3: An alternative to *RE*PO4

- Single crystals: Deviations from *RE*:Sc = 1:1 reported in literature [1,2], e.g., $(\square_{0.045}Sm_{0.955})ScO_{2.933}$.

→ Non-Stoichiometry affects data in appreciable way.
→ Conclusions concerning correction factors of neighboring elements hold.
→ Correction factors from phosphates and scandates similar, as primary inter-element effects are already corrected for both systems.



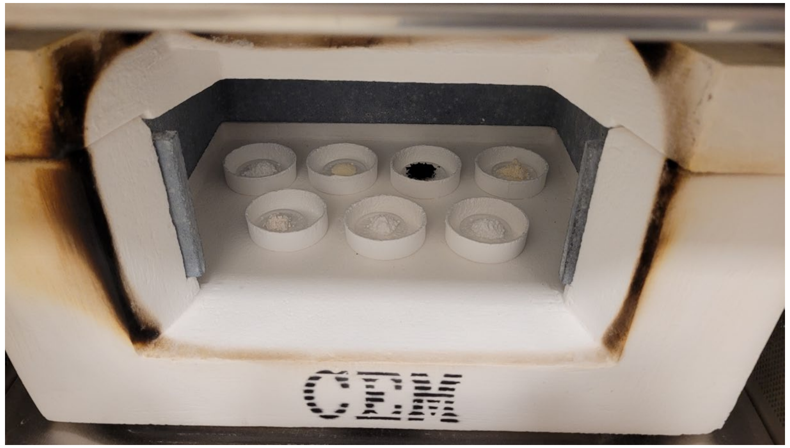


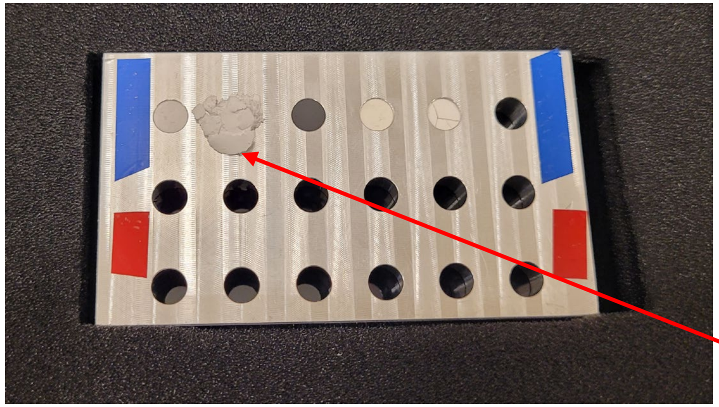
[1] Velickov, B. et al. (2007). Z. Kristallogr. 222, 466–473.
[2] Uecker, R. et al. (2008). J. Cryst. Growth 310, 2649–2658.

REScO_3: Self-made reference materials

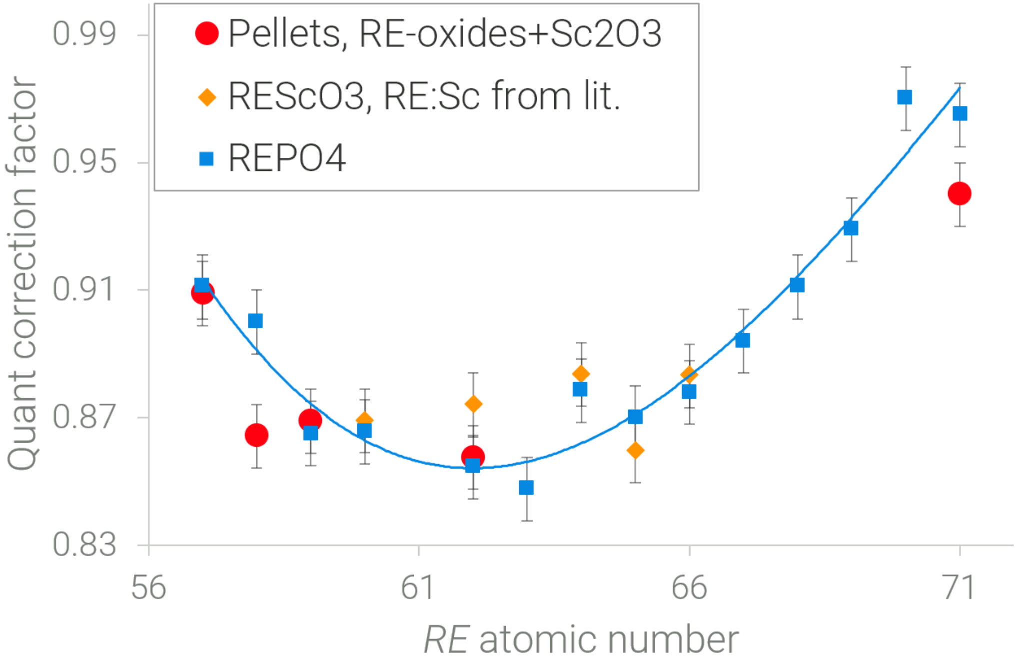
- In-house reference materials prepared: Pressed pellets of dehydrated *RE*-oxides and Sc_2O_3.
- Storage in N_2 due to hygroscopic nature.
- However: Powders not ground – no Nanopowders!

→ Results follow expectations, despite imperfect sample preparation.



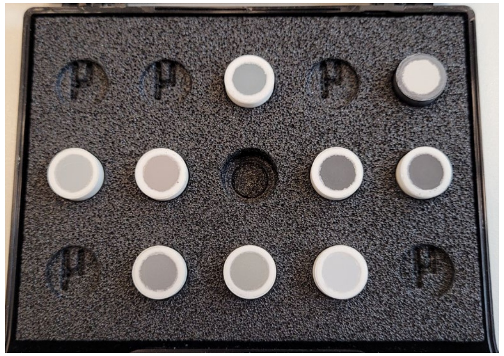


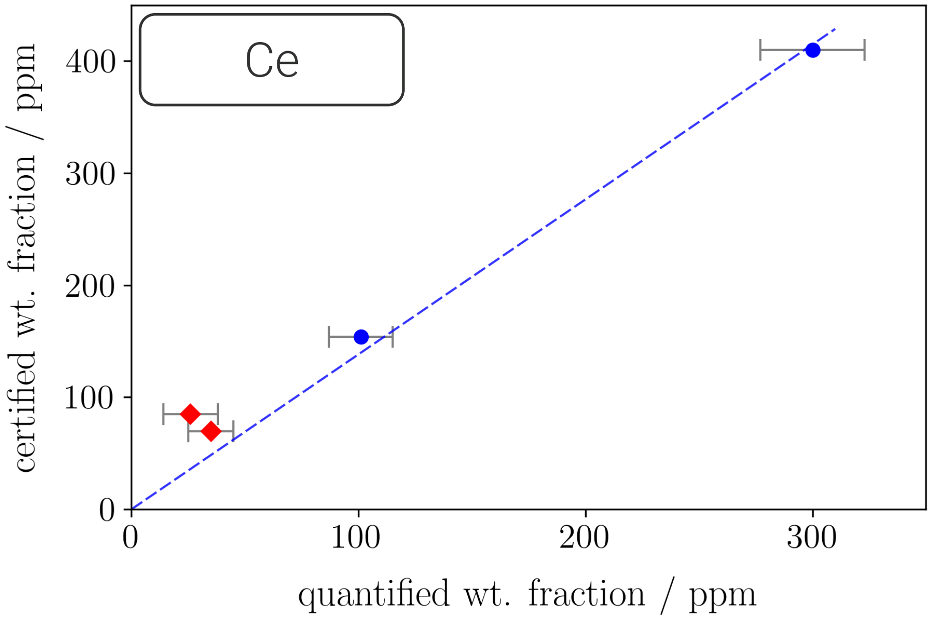
La_2O_3 is the most hygroscopic chemical here.



Samples with low *RE* concentrations and complex makeup
Nanopowders

- Nanopowders: Samples with complex composition.
- *RE*-concentrations comparatively miniscule.
- Four samples contain (enough) Ce.





→ Quantification can be calibrated based on multiple reference materials.

→ Correction factor derived by linear fitting of quantified and certified weight fractions; high-Ce data only: 1.38(5)

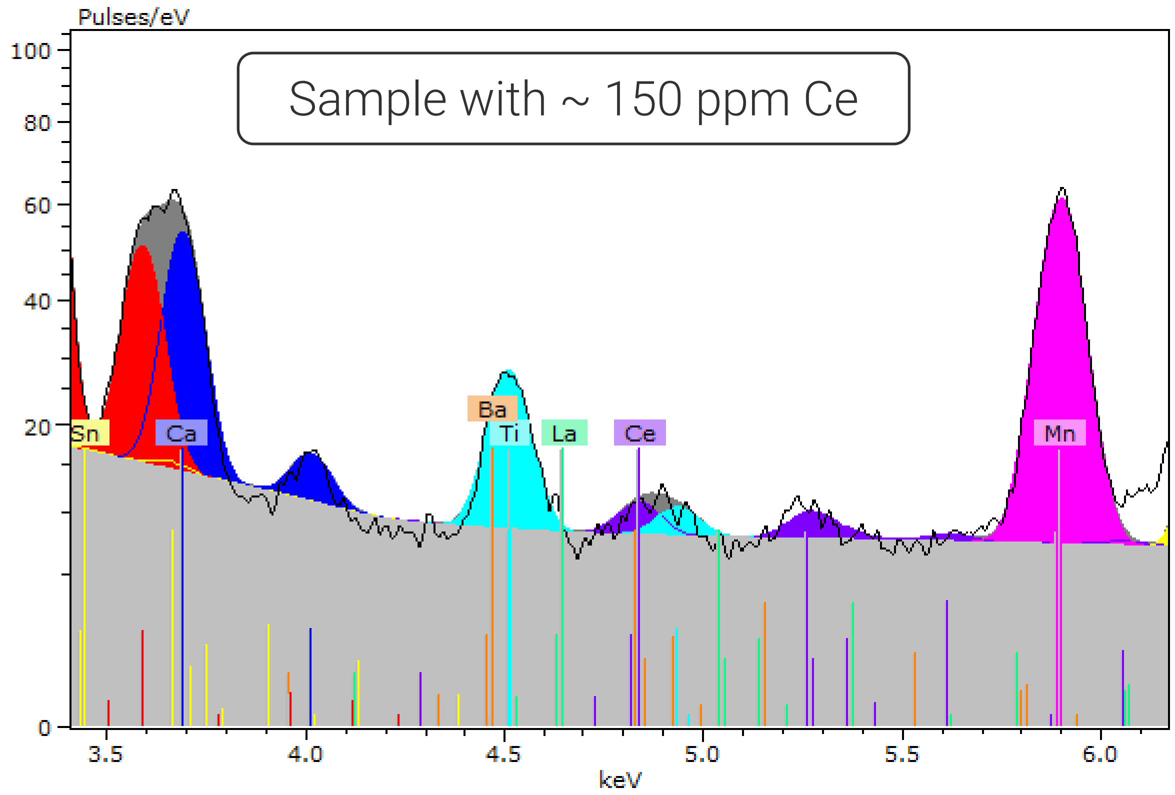
Why so (comparatively) imprecise?
Why exclude low-Ce data?

- Average of 10 measurements each.
- 20 minutes real time due to strong Al-200µm Ti-200µm filter, low concentrations and pronounced overlap.

Samples with low *RE* concentrations and complex makeup
Nanopowders

Deconvolution unreliable due to:
- Ce-peak very small due to low concentrations and strong filter.
- Overlap with multiple elements: Ba, Ti, La, V, Cr depending on the sample.

→ Calibration based on samples, where Ce signal is more distinct.



Lower precision due to:
- Lower *RE*-signal.
- Mixing of more varied reference materials: *RE* subject to varying interactions.

→ Could be improved by more conscious selection of reference materials/adjustment of measurement times.

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Calibrating FP based quantification in the M4 TORNADO
Glass reference materials

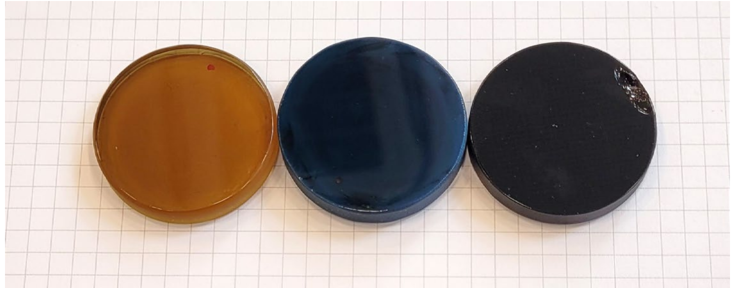
Glass reference materials: strongly varying primary components:

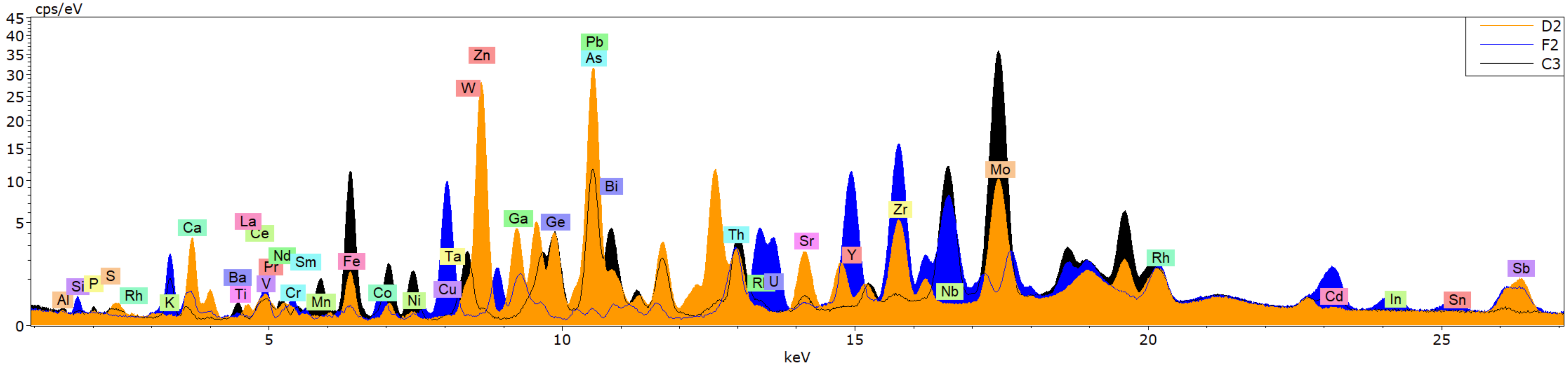
D2: 22% B_2O_3, 21% Al_2O_3, 14% CaO, 10% Na_2O

F2: 57% SiO_2, 18% K_2O

C3: 27% Al_2O_3, 19% B_2O_3, 16% P_2O_5, 10% SiO_2

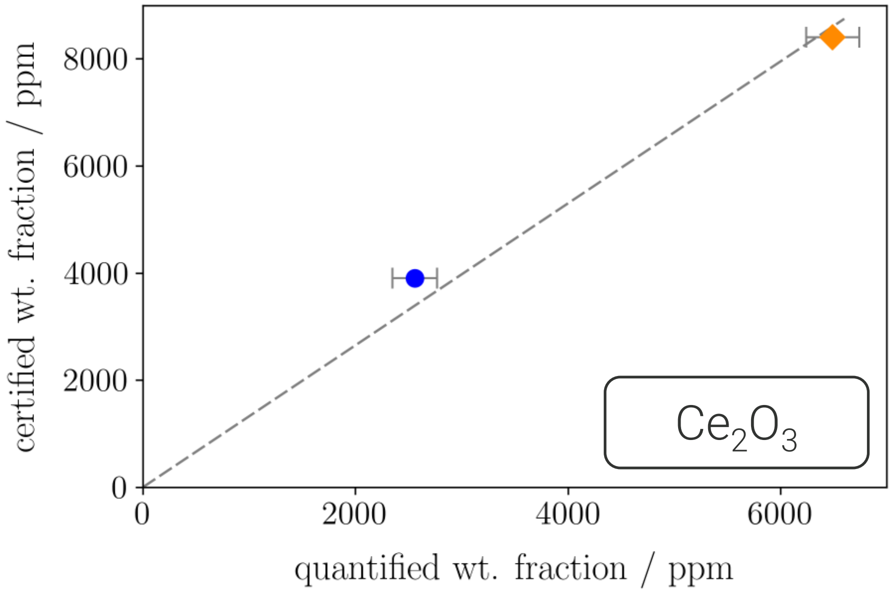
But some *RE* are present!

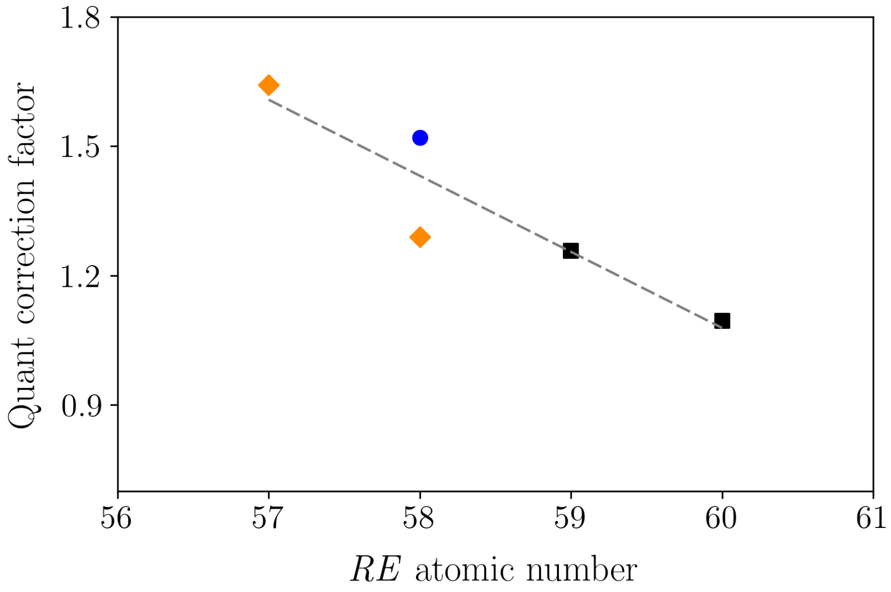




Calibrating FP based quantification in the M4 TORNADO
Glass reference materials

High variation in chemistry:
→ High scatter for correction factors expected.
→ Correction factor for Ce is **1.33(8)**
Nanopowders was **1.38(5)**!





- Trends within *RE* still observed.
- If no reference materials available: Correction factor from neighboring element may be applicable.

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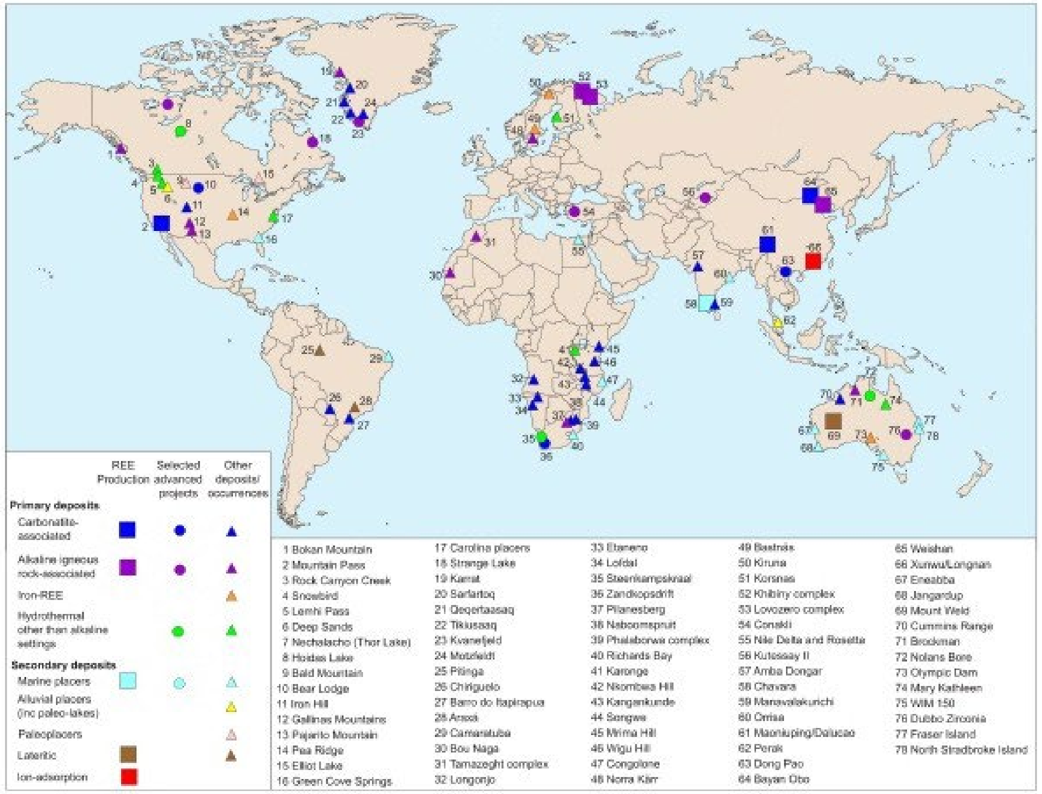
WEBINAR: MICRO-XRF – REE QUANTIFICATION

Examples

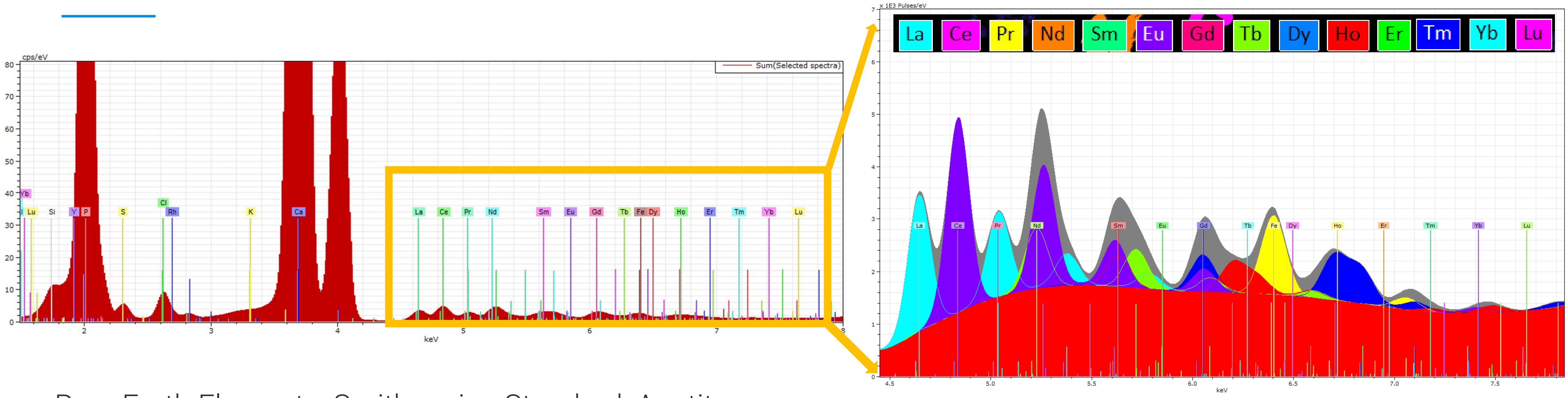
Rare Earth Elements:
Geology – major deposits



Mineral	Formula	Approximate REO %
Aeschynite-(Ce)	$(Ce,Ca,Fe,Th)(Ti,Nb)_2(O,OH)_6$.	32
Allanite-(Ce)	$(Ce,Ca,Y)_2(Al,Fe^{3+})_3(SiO_4)_3OH$.	38
Apatite	$Ca_5(PO_4)_3(F,Cl,OH)$	19
Bastnäsite-(Ce)	$(Ce,La)(CO_3)F$	75
Brannerite	$(U,Ca,Y,Ce)(Ti,Fe)_2O_6$	9
Britholite-(Ce)	$(Ce,Ca)_5(SiO_4,PO_4)_3(OH,F)$	32
Eudialyte	$Na_4(Ca,Ce)_2(Fe^{2+},Mn,Y)ZrSi_8O_{22}(OH,Cl)_2$(?).	9
Euxenite-(Y)	$(Y.Ca.Ce,U,Th)(Nb,Ta,Ti)_2O_6$	24
Fergusonite-(Ce)	$(Ce,La,Nd)NbO_4$	53
Gadolinite-(Ce)	$(Ce,La,Nd,Y)_2Fe^{2+}Be_2Si_2O_{10}$.	60
Kainosite-(Y)	$Ca_2(Y,Ce)_2Si_4O_{12}CO_3.H_2O$.	38
Loparite	$(Ce,La,Na,Ca,Sr)(Ti,Nb)O_3$	30
Monazite-(Ce)	$(Ce,La,Nd,Th)PO_4$	65
Parisite-(Ce)	$Ca(Ce,La)_2(CO_3)_3F_2$.	61
Xenotime	YPO_4.	61
Yttrocerite	$(Ca,Ce,Y,La)F_3.nH_2O$.	53
Huanghoite-(Ce)	$BaCe(CO_3)_2F$.	39
Cebaite-(Ce)	$Ba_3Ce_2(CO_3)_5F_2$.	32
Florencite-(Ce)	$CeAl_3(PO_4)_2(OH)_6$.	32
Synchysite-(Ce)	$Ca(Ce,LA)(CO_3)_2F$.	51
Samarskite-(Y)	$(Y,Ce,U,Fe^{3+})_3(Nb,Ta,Ti)_5O_{16}$.	24
Knopite	$(CaTi,Ce_2)O_3$	na

REE Minerals occur as:
silicates,
carbonates,
oxides,
phosphates…

Micro-XRF Analysis of Rare Earth Elements (REE´s):
Apatite – Low concentrations



Rare Earth Elements: Smithsonian Standard: Apatite
Total REEs approx. 1.43 wt%

La2O3	Ce2O3	Pr2O3	Nd2O3	Sm2O3	EuO	Gd2O3	Tb2O3	Dy2O3	Ho2O3	Er2O3	Tm2O3	Yb2O3	Lu2O3	Sum
0.509	0.655	0.000	0.165	0.073	0.000	0.081	0.000	0.001	0.000	0.001	0.000	0.008	0.000	1.493

Jarosewich, E., Gooley, R., and Husler, J. 1987. Chromium Augite – A new microprobe reference sample, Geostandards Newsletter, Vol. 11., No. 2, p197-198.

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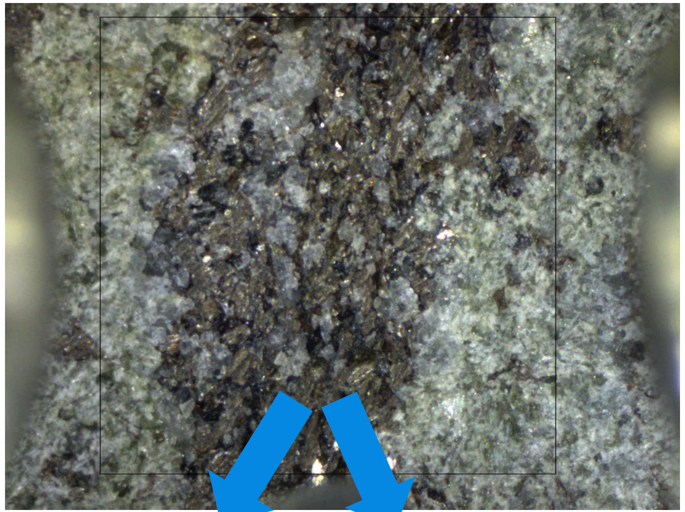
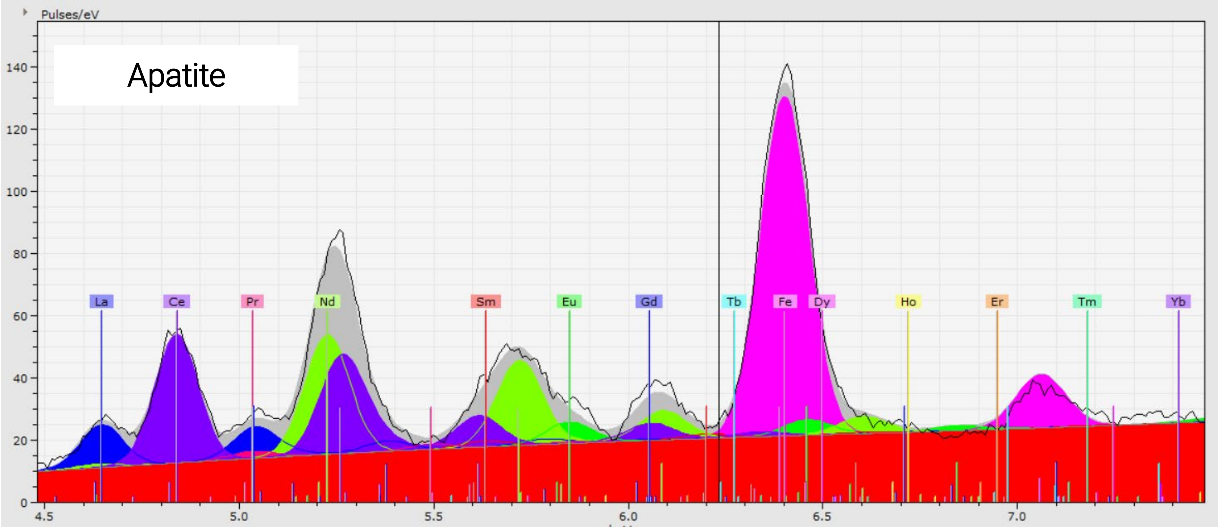
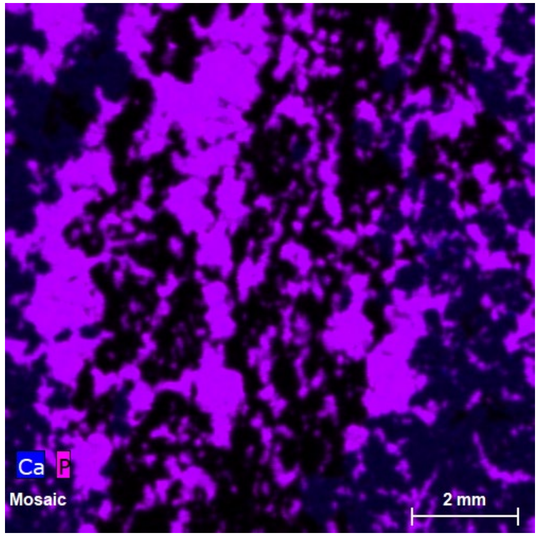
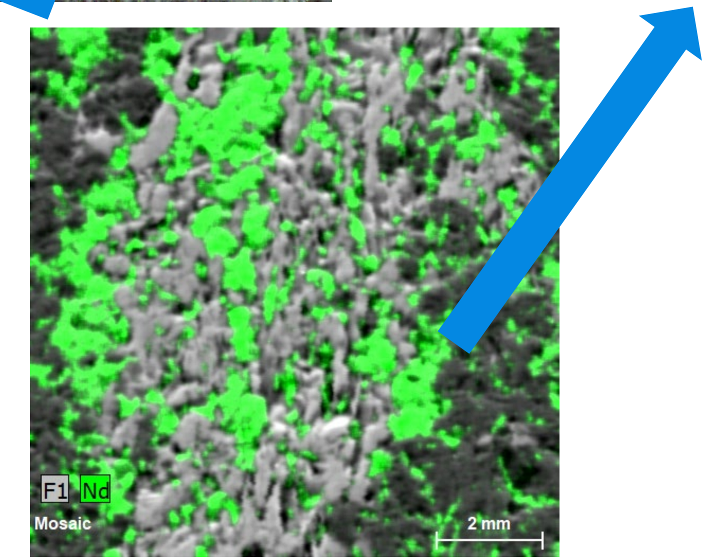
Micro-XRF Analysis of Rare Earth Elements (REE´s): Phalaborwa - Apatite









	Lanthanum	57	1089
	Cerium	58	2573
	Praseodymium	59	144
	Neodymium	60	1600
	Samarium	62	44
	Europium	63	228
	Gadolinium	64	10
	Terbium	65	0
	Dysprosium	66	0
	Holmium	67	54
	Erbium	68	0
	Thulium	69	0
	Ytterbium	70	0
	Lutetium	71	19

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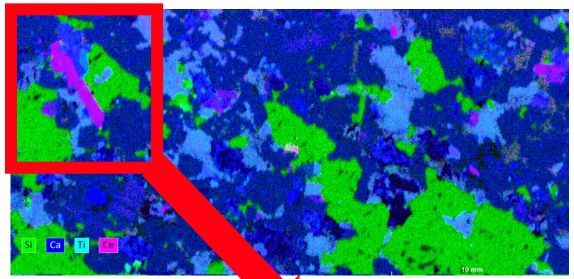
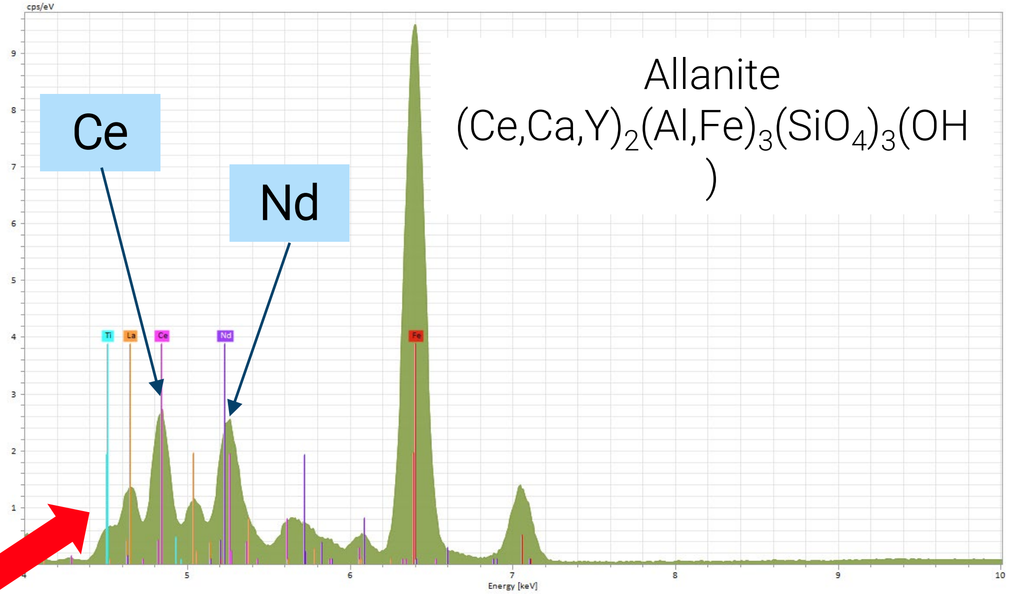
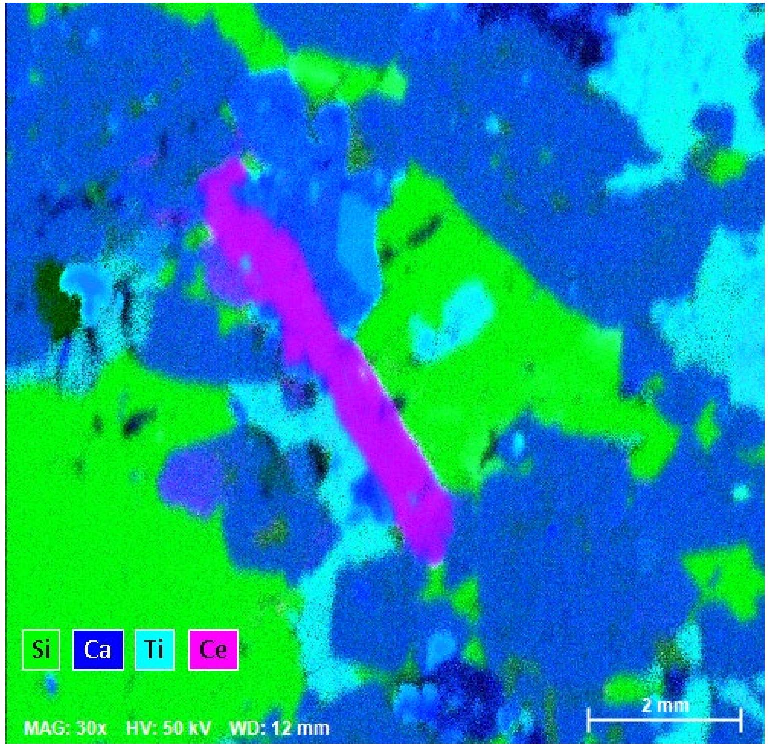
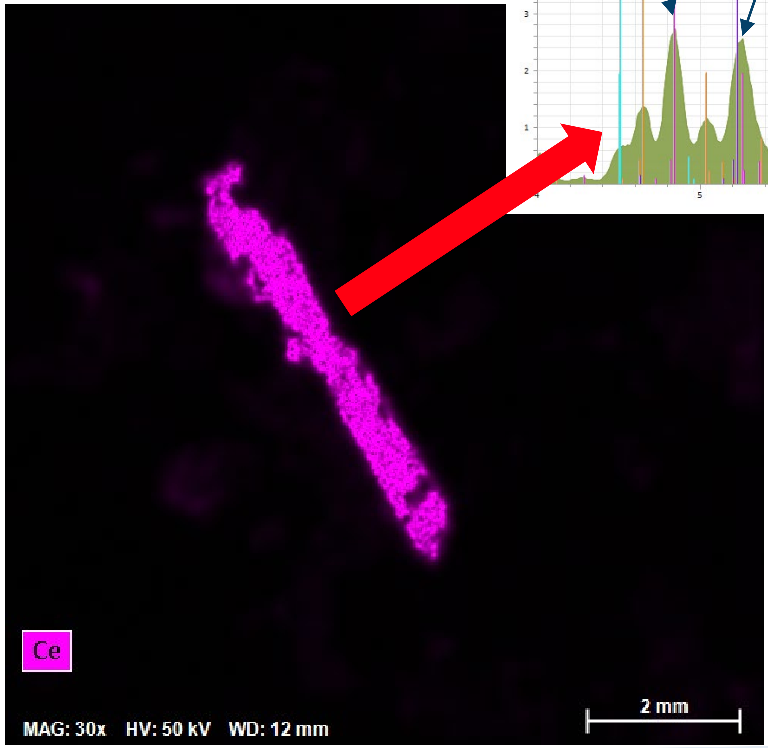
Micro-XRF Analysis of Rare Earth Elements (REE´s): Chile - Granite with Allanite



Analysis of Thin Section and of Rock Offcut







Element	Concentration (wt%)
CaO	5.3
Ce_2O_3	32.2
Y_2O_3	5.4
Al_2O_3	18.8
Fe_2O_3	9.7
SiO_2	29.4

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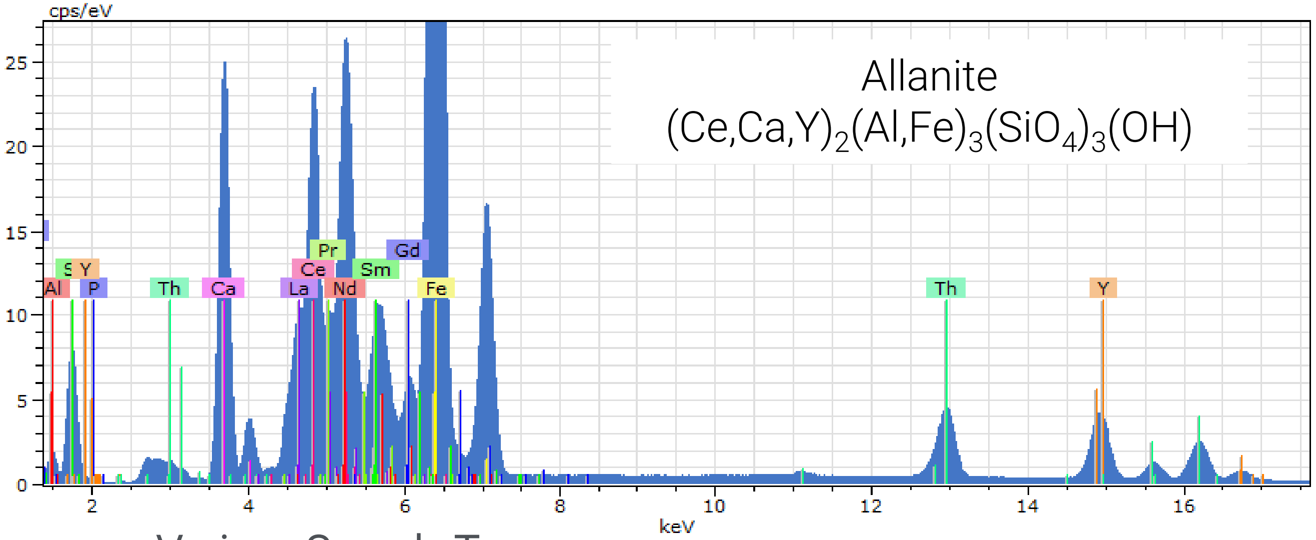
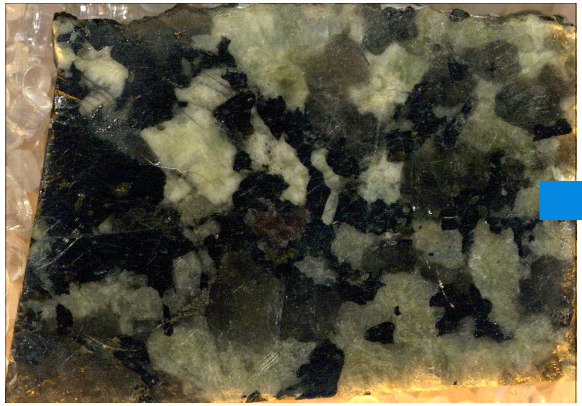
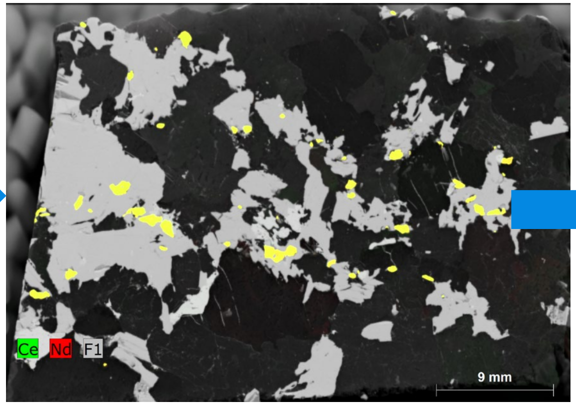
Micro-XRF Analysis of Rare Earth Elements (REE´s):
Sample Types: Geology

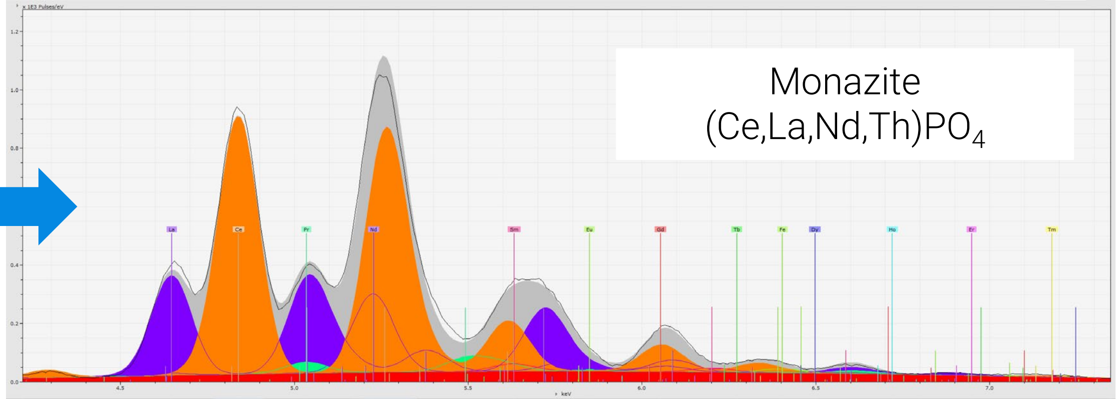


Various Sample Types:

Above: Loose Large Mineral Grains; **Below:** Cut Rock Sample Surface







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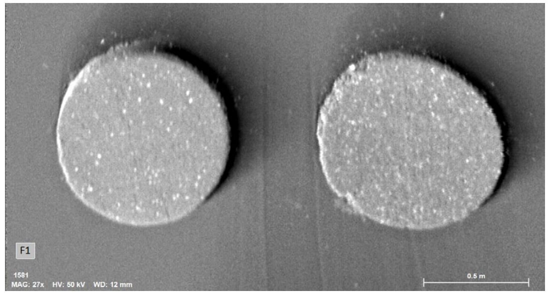
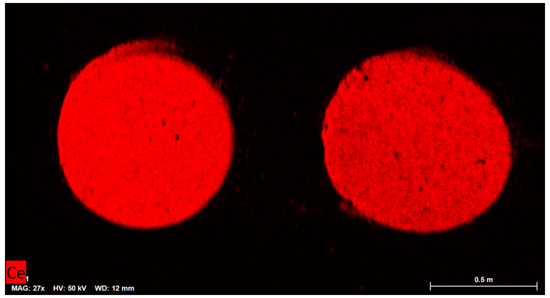
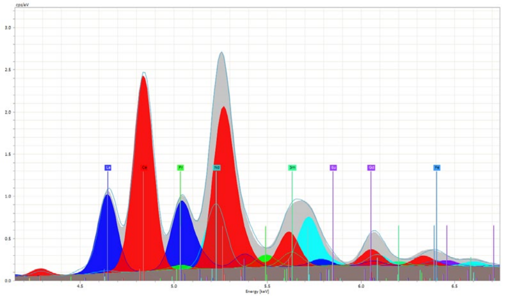
Micro-XRF Analysis of Rare Earth Elements (REE´s): Sample Types: Industry

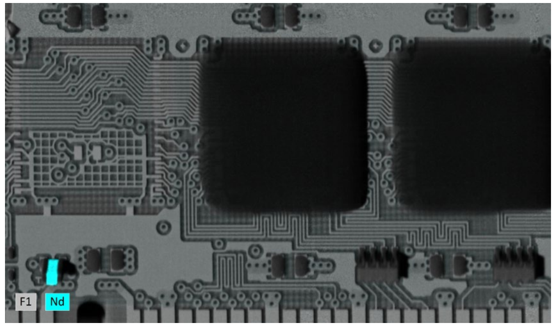
Crushed Powders

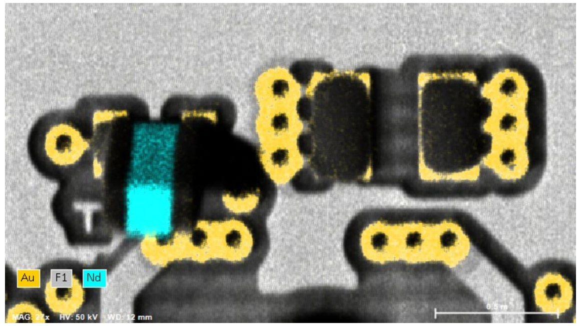


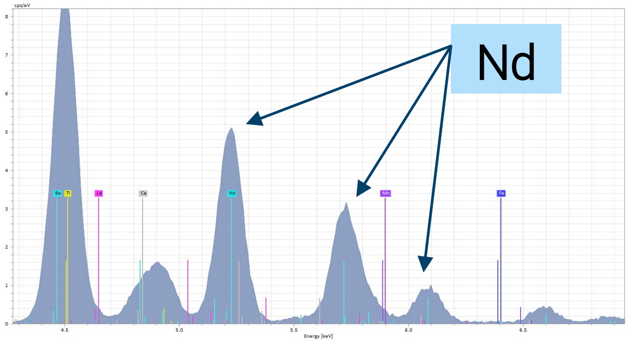




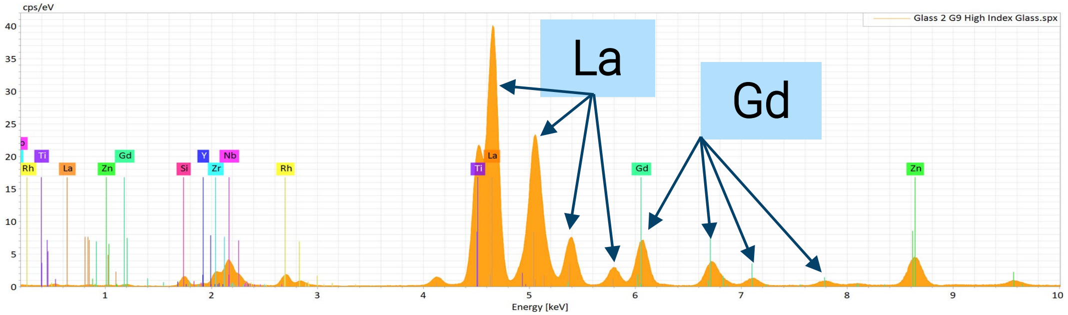
Electronics

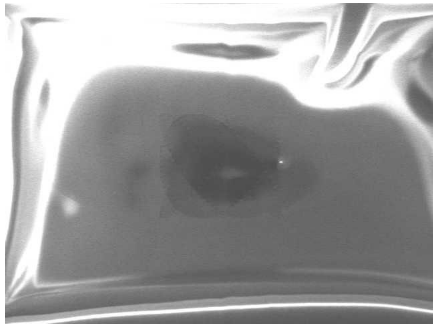






High Index Glasses





SEM Image showing charging under 20 kV e-beam:

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Summary and Conclusions:
Critical Metals and Micro-XRF REE Quantification

- The analysis of Critical Metals is important for our transition to the Green Technology Future
- Good quantification necessitates carefully chosen measurement conditions: Filter selection for crystalline materials, Measurement time…
- Surface roughness impacts the precision of quantification
- Reference materials are required for highest accuracy: Commercial/in house/self-made
- Depending on desired accuracy, quantification of a Rare Earth Element may be calibrated based on another Rare Earth Element standard with similar cationic number
- Micro-XRF is a flexible technique that can analyze a variety of sample types

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Micro-XRF
Further Information

www.bruker.com/micro-xrf

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Introducing micro-XRF as an Analytical Technique

The first webinar in our "Back to the Roots" Series, focusing on the fundamentals of micro-XRF.

This webinar introduces micro-XRF as a technique, highlights the analytical relevance of multiple instrument components, and discusses their significance from an analytical point of view.

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XRF Data Processing in Art and Conservation - Advanced Features of ESPRIT Reveal

X-ray fluorescence (XRF) measurements are widely considered as a key step for a diagnosis in the process of conservation, restoration, authentication, or when addressing historic questions around an art object.

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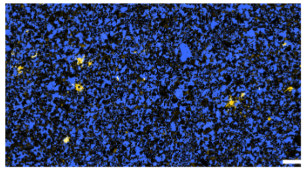

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Trace Elements and Mineralization: The benefits of combining micro-XRF and SEM-EDS/WDS

In many economic deposits the element or mineral of interest is a trace component. However, the ability to identify these elements and minerals depends on how they occur. Such information is important to understand the genesis of the deposit as well as the mineral and metallurgical processes to yield the maximum recovery.

VIEW

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Bruker's ELIO portable micro-XRF scanner supports studies in Art & Conservation worldwide, taking XRF measurements to the object of interest, wherever it may be. Join us to learn about how ELIO is helping our partners in Art & Conservation to delve deeper and reveal new insights with their research.

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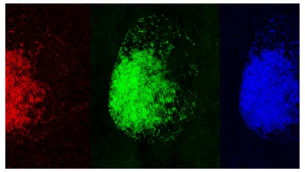

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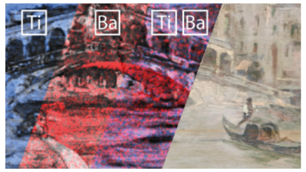
The M4 TORNADO micro-XRF Spectrometer - a Flexible Tool in Forensic Science

Micro-XRF is an established technique in forensic analysis, having been present in many laboratories for more than a decade. The advantages of this technique were immediately recognized- micro-XRF provides a unique ensemble that integrates the multi-element sensitivity of XRF with detection down to trace element levels, large sample accessibility, and spatial resolution down to the micrometer range.

VIEW



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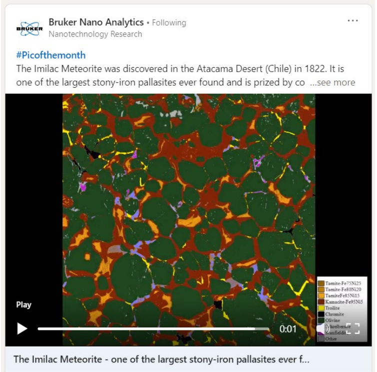
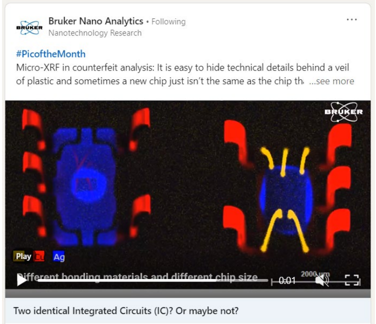
XRF Data Processing in Art and Conservation with ESPRIT Reveal

Bruker offers best in class solutions for the field of art studies from super portable handheld systems to highly sophisticated mapping instruments.

VIEW





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