Purification and separation of rare-earth elements originating from sulphidic mining residue using nonaqueous solvent extraction

Brecht Dewulf

KU LEUVEN
SOLVOMET GROUP

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Industrial Service Centre

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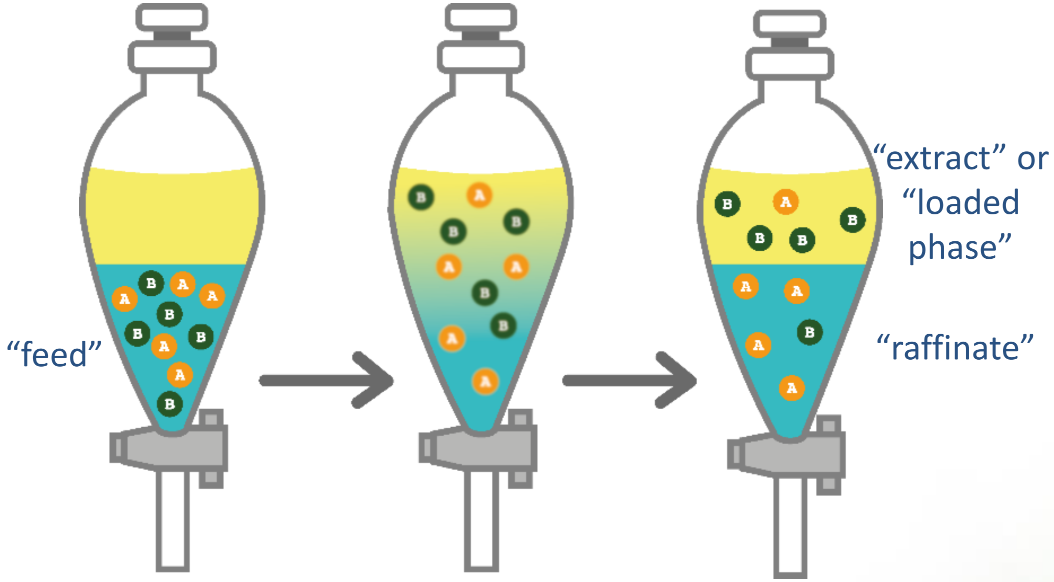
Solvometallurgy

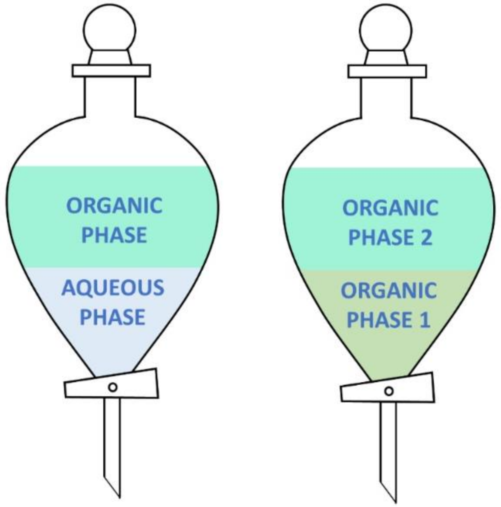
Non-aqueous solvent extraction (NASX)

- Distribution of a solute between two immiscible organic phases
- Expands scope of conventional SX
- Can offer enhanced selectivity and treat water-sensitive compounds



Requirements

- Low mutual solubility of two phases
- Fast phase separation
- Extractant is soluble in less polar (LP) phase, not in more polar (MP) phase
- Starting metal compound is soluble in the MP phase
- Metal compounds should not react with the organic solvents



Binnemans and Jones, **2017**, J. Sustain. Met. 3, 570–600

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Non-aqueous solvent extraction for rare-earth separation

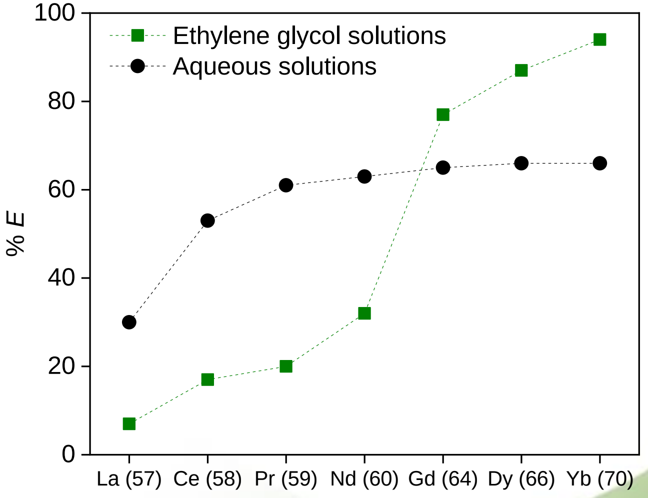
Rare-earth elements (REEs) and their application

- 15 lanthanides + Sc and Y
- Critical raw material: highest supply risk (EU: 98% imported from China)
- Economic importance: hard-disk drives, catalysts, abrasives, fluorescent phosphors, permanent magnets
- Importance in energy transition and green technologies

Group separation and purification of individual REE

- Conventional process: 1000s of stages comprising numerous extraction-scrubbing-stripping cycles
- Non-aqueous SX: higher separation factors → more efficient separation process

Batchu *et al.*, **2017**, Sep. Purif. Technol. 174, 544–553



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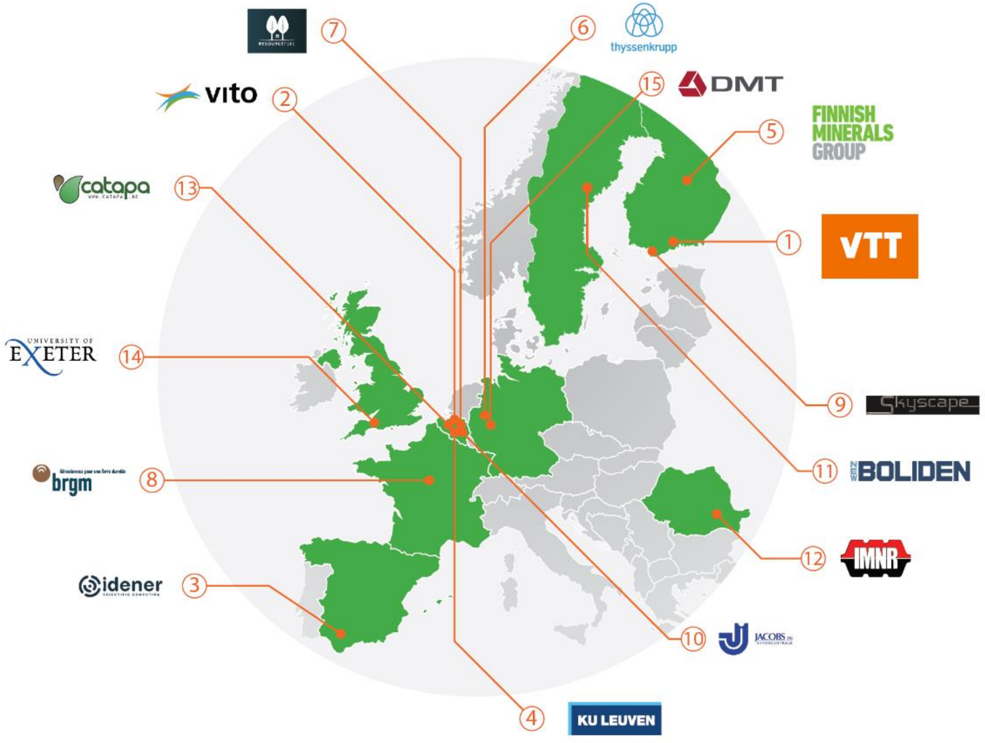
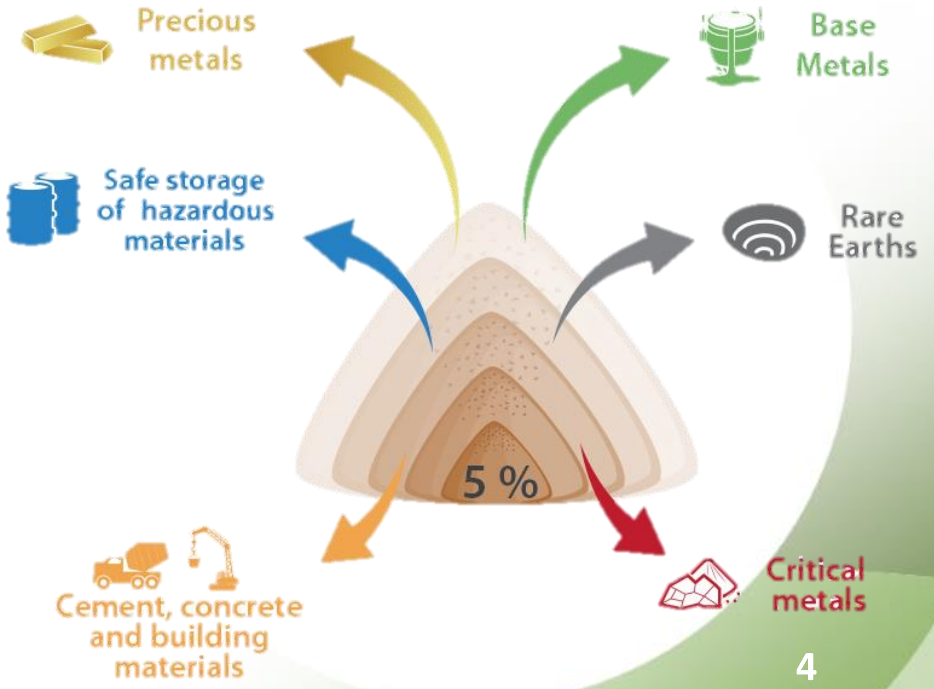
EU H2020 NEMO

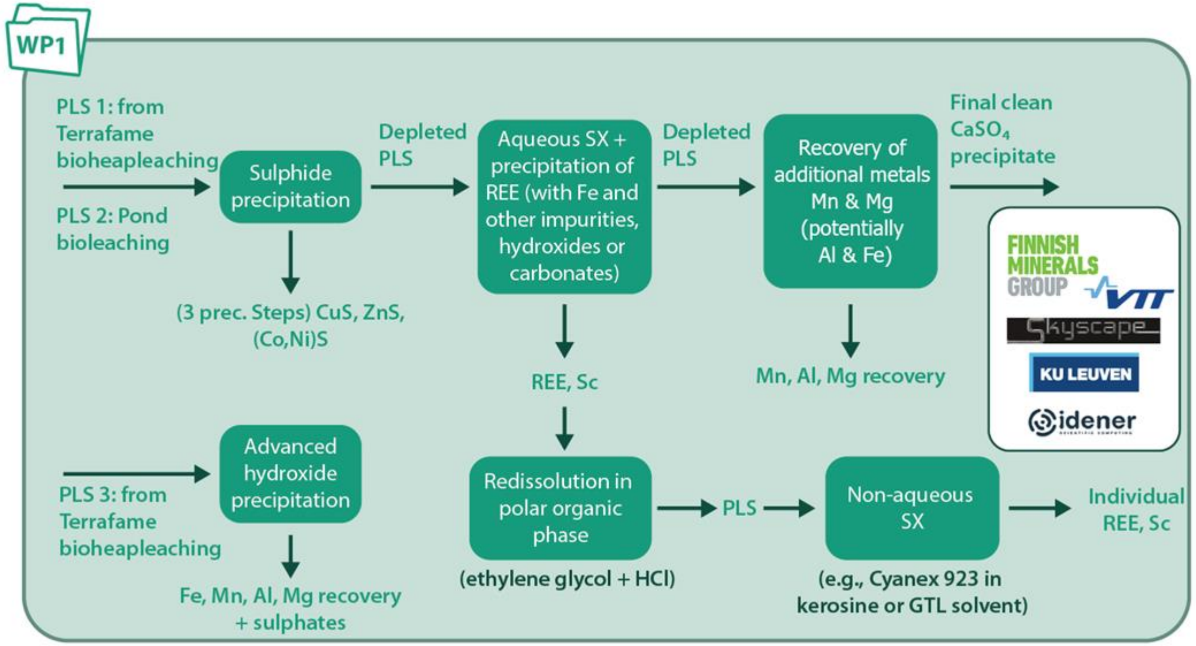
- Valorization of low-grade sulphide mining waste: 600 Mtonne/yr + historic stockpile 28,000 Mtonne in Europe
- 15 partners across Europe, 3 case studies, 4 pilots
- REE recovery from Sotkamo residue: study of solvometallurgical routes





https://h2020-nemo.eu/

EU H2020 NEMO



Impurities		Rare-earth element composition	
element	Concentration (wt% hydroxide mixture)	element	Concentration (wt% hydroxide mixture)
Al(III)	2.05	Dy(III)	0.42
Ca(II)	1.56	Ho(III)	0.27
Fe(III)	0.18	Er(III)	2.28
Mg(II)	0.12	Tm(III)	1.41
Mn(II)	0.93	Yb(III)	15.30
Ni(II)	0.30	Lu(III)	2.96
Zn(II)	0.81	Y(III)	29.88

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Experimental setup - overview

Solvometallurgy & rare earths

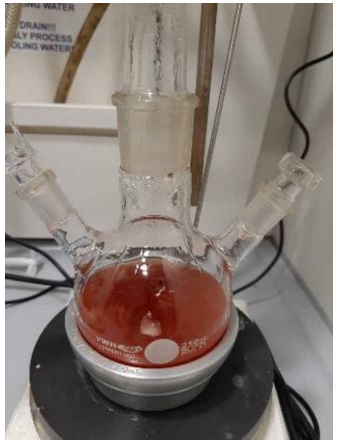
H2020 NEMO

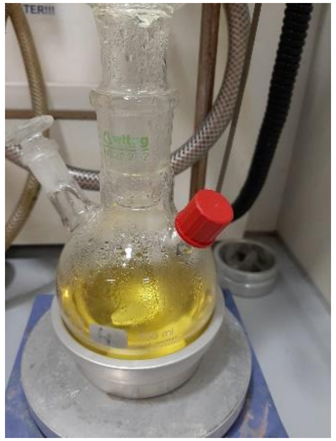
Materials & experimental conditions

Mixer-settler pilot

Conclusions

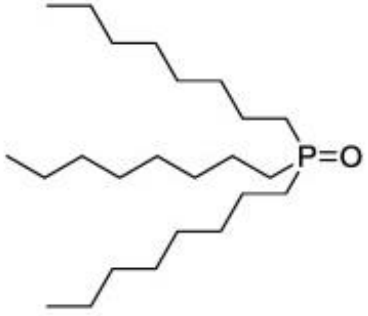
Dissolution → Lab-scale solvent extraction → Upscaling & piloting





LP: Cyanex 923 (C923) in petroleum ether

MP: 10 g/L REE, NaCl, 10 vol% water in ethylene glycol (EG)



Cyanex 923 (main component)

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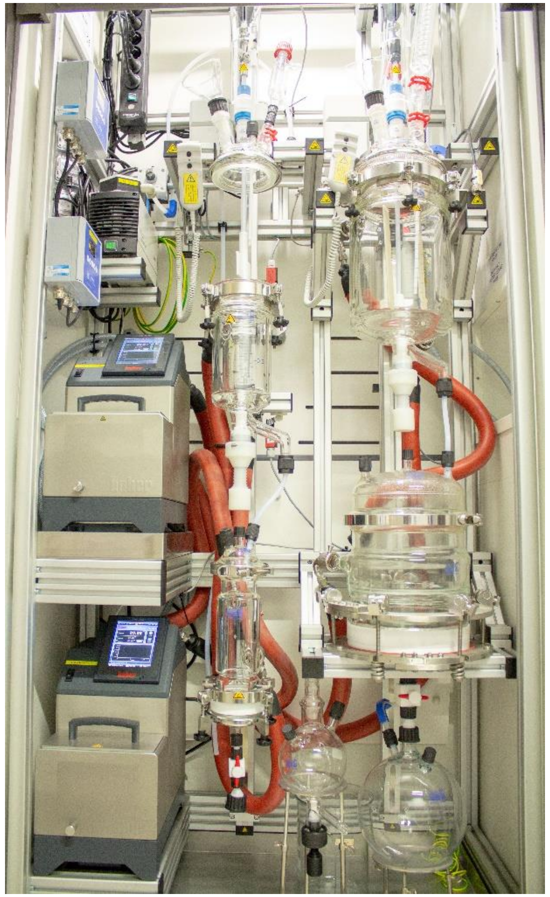
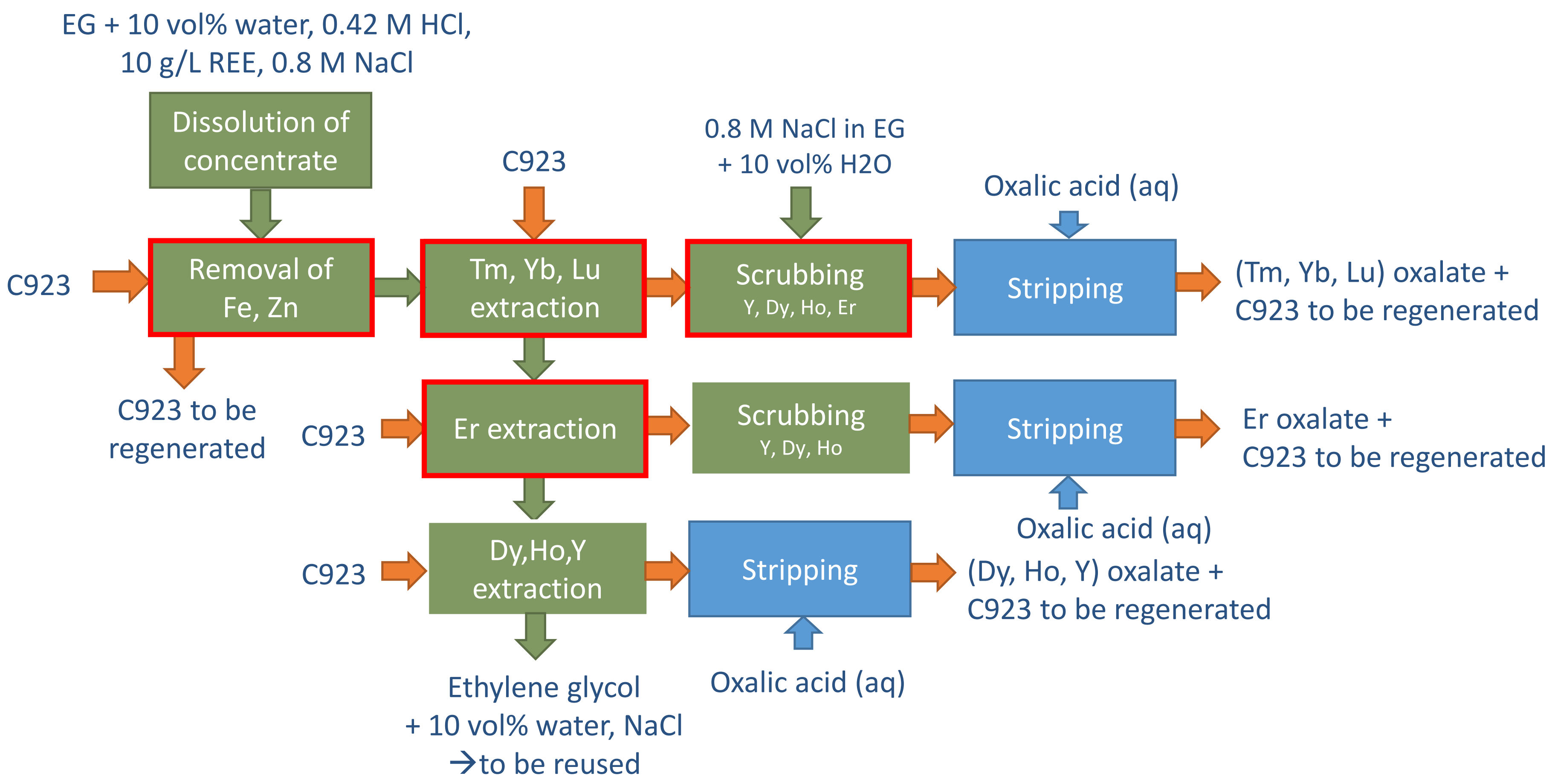
Experimental setup - equipment



Batch Leaching platform (HiTec-Zang)
- Jacketed 1 L and 5 L reactors
- Jacketed filtration units
- Borosilicate glass
- Optimized for viscous liquids

Mixer-settler setup (SX Kinetics)
- 16 stages
- Mixer: 0.270 L; settler: 1.050 L
- Pumps: Cole-Parmer Masterflex L/S: max 10 L/h

Flowsheet overview: Dy, Y, Ho // Er // Tm, Yb, Lu



Scale:

- 60 L of original MP feed solution → purification & separation
- 2* 30 L of scrub solution
- > 100 L of LP phase, with various [C923]

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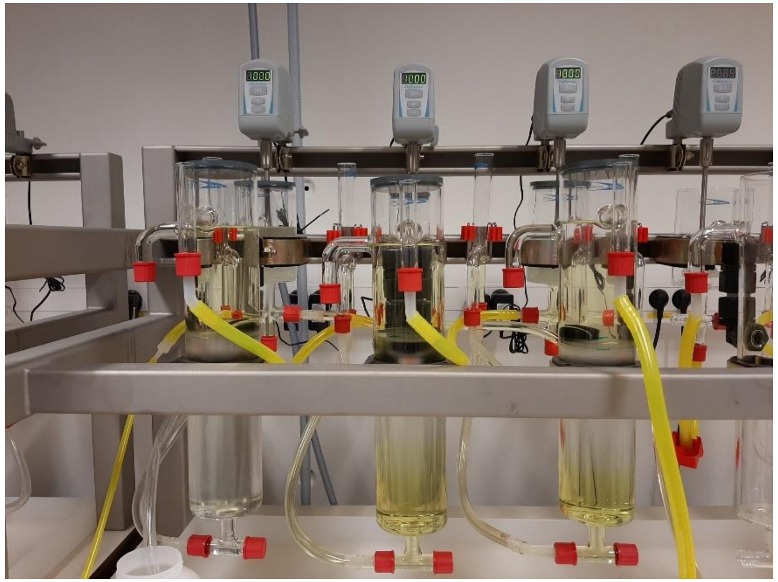
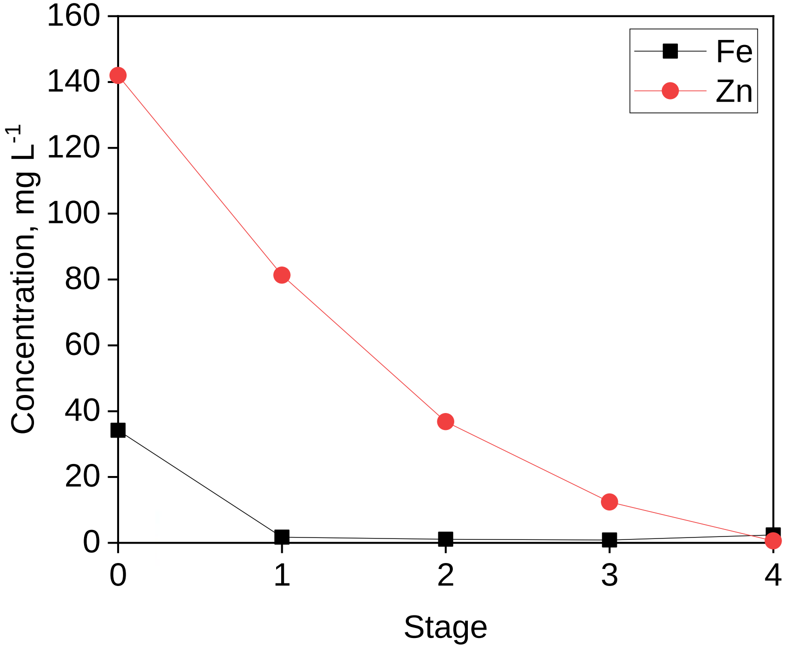
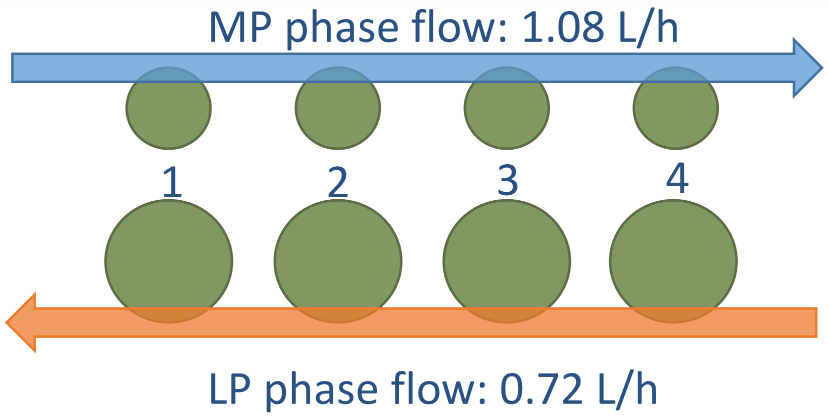
Removal of Fe, Zn







- Retention time 6 min
- Ni and Mg not extracted by C923; Ca, Mn limited extraction at higher C923 concentration
- Good phase separation, limited yet visible entrainment of LP in MP phase

3 stages:

- *%E* Fe 85% (6 ppm) - Zn 96% (6 ppm)
- No loss of REE

4 stages:

- *%E* Fe 95% (2 ppm) - Zn 99.95% (< 1 ppm)
- Limited loss of REE (1-3 %)

MP: 10 g L^{-1} REE, 0.14 g L^{-1} Zn, 0.4 g L^{-1} Fe, 0.8 M NaCl, 10 vol% H_2O, EG

LP: 0.1 M C923, petroleum ether

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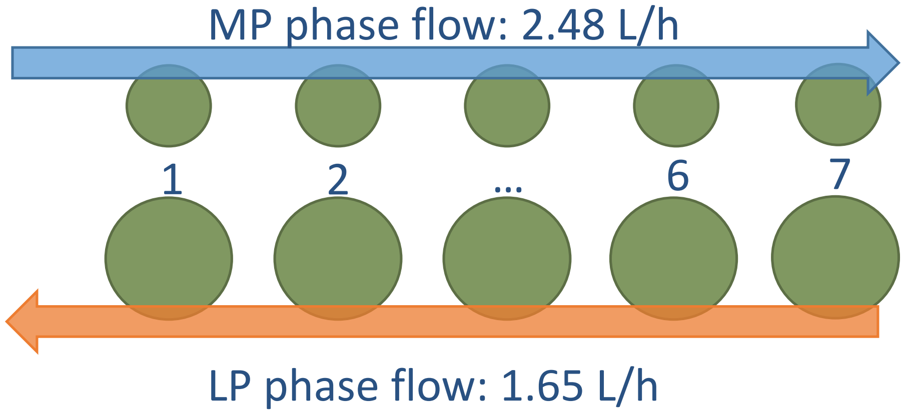
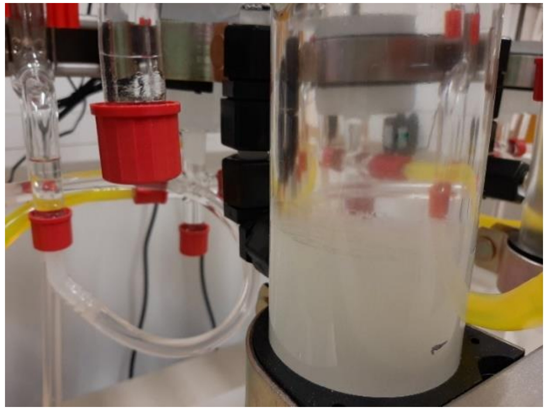
Separation Dy, Y, Ho, Er // Tm, Yb, Lu



element	%E	Conc MP (ppm)	Conc LP (ppm)
Y	24	3411	1743
Dy	11	59	11.6
Ho	25	26.5	16.0
Er	49	123	274
Tm	**95.4**	11.9	355
Yb	**99.4**	15.5	3905
Lu	**99.4**	3.1	919

- Retention time 4 min
- Quasi complete extraction of Tm, Yb, Lu
- Emulsion might pose a problem – adapt design of mixer-settlers



MP: raffinate ~10 g L^{-1} REE, 0.8 M NaCl, 10 vol% H_2O, EG

LP: 0.1 M C923, petroleum ether

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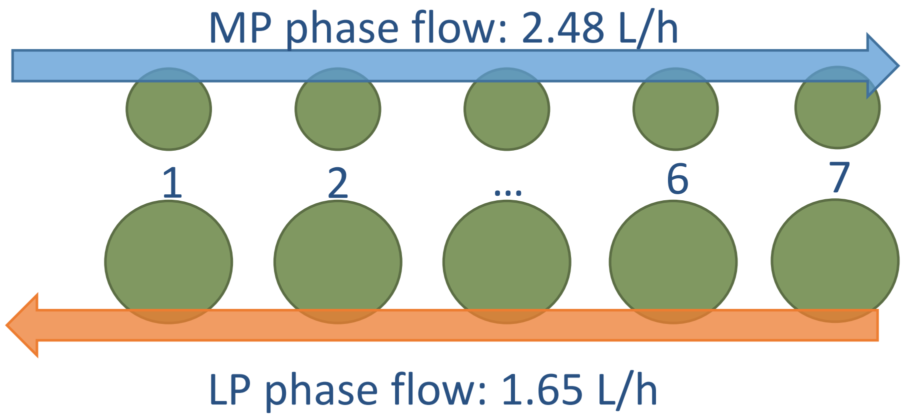
Scrubbing of Y, Dy, Ho, Er



element	*%Scr*	Conc MP (ppm)	Conc LP (ppm)
Y	**99.98**	1204	0.3
Dy	**100**	7.9	0
Ho	**98.5**	7.4	0.2
Er	**100**	184	0
Tm	78	173	77
Yb	44	1634	2285
Lu	42	296	566

- Retention time 4 min
- REE in MP scrub raffinate → recycled to extraction
- Scrubbing very efficient: purity Tm-group > 99.99%

MP: 0.8 M NaCl, 10 vol% H_2O, EG

LP: loaded 1 M C923, petroleum ether

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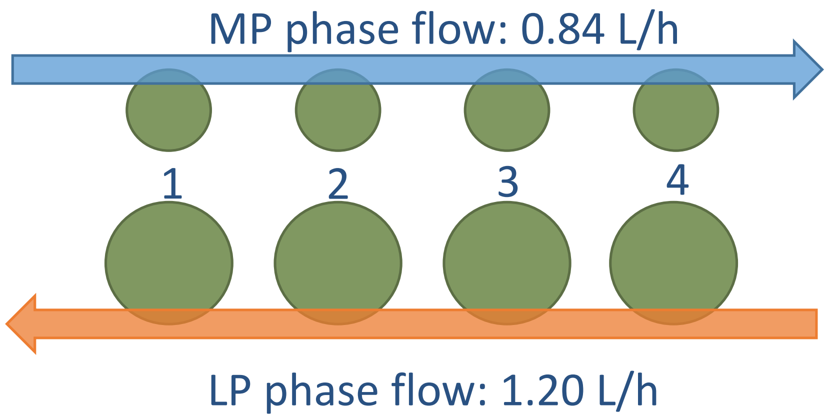
Separation Dy, Y, Ho // Er



element	%E	Conc MP (ppm)	Conc LP (ppm)
Y	90	766	2147
Dy	57	30	20
Ho	92	6	17
Er	**98.5**	8	112
Tm	100	0	15
Yb	100	0	18
Lu	100	0	3

- Retention time 7 min
- Significant co-extraction of Y, Ho
  → main issue will be scrubbing Y

MP: 59 ppm Dy, 32 ppm Ho, 130 ppm Er, 3927 ppm Y, 0.8 M NaCl, 10 vol% H_2O, EG

LP: 1 M C923, petroleum ether

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Main research conclusions

- Efficient group separation of HREE by non-aqueous solvent extraction (Dy, Y, Ho, Er // Tm, Yb, Lu)
- Er separation and purification challenging
- Feasibility of non-aqueous process demonstrated on lab-scale mixer-settler pilot
- Non-aqueous solvent extraction complementary to hydrometallurgical extraction techniques

Future outlook of non-aqueous solvent-extraction:

- Economic feasibility studies
- Further optimization of processes to overcome current issues
- Study the reuse of the MP and LP phases

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https://solvomet.eu
https://kuleuven.sim2.be/
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Contact: Brecht Dewulf
(brecht.Dewulf@kuleuven.be)

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