# Presentation KU Leuven SOLVOMET Group:

*Laboratory of Metallurgical Chemistry & Industrial Service Centre*

*P.T. Jones & K. Binnemans*

June 21, 2022

# SOLVOMET Group
# Laboratory of Metallurgical Chemistry

SOLVOMET's vision is that metallurgical chemistry expertise allows to develop more efficient, eco-friendly hydrometallurgical and solvometallurgical processes to provide the critical metals that are needed for the transition to a climate-neutral society.

SOLVOMET's mission is (1) to perform excellent research in metallurgical chemistry and to educate and train young researchers in this domain and (2) to support its Industrial Service Centre partners in the conceptual and practical development of more sustainable (circular, low-energy input) hydrometallurgical (and solvometallurgical) processes, which are subsequently tested using state-of-the-art lab-scale and mini-pilot-scale experimental facilities.

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# Background info on Prof. Koen Binnemans & SOLVOMET Group

- **Full professor** at the Department of Chemistry, specialised in circular hydrometallurgy and solvometallurgy
- Core expertise in **critical metals and solvent extraction** (SX)
- Author of more than 560 papers, **H-index = 81**, > 29,000 citations
- **Former ERC Advanced Grant holder** (SOLCRIMET: Solvometallurgy for critical metals)
- **ERC Proof of Concept holder** (SOLVOLi: Solvometallurgy for battery-grade refining of lithium)
- Co-founder **SOLVOMET Industrial Service Centre** for Hydro/solvometallurgy
- Steercom Member KU Leuven Institute for Sustainable Metals and Minerals (**SIM² KU Leuven**)
- Former Steercom Member European Rare Earth Competency Network (**ERECON**)
- Elected member **Royal Flemish Academy of Belgium for Science and the Arts** (KVAB)
- For research domains “hydrometallurgy” & “solvent extraction”, according to Google Scholar (data retrieved 2021-07-06), the “**world’s most cited author**”.



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# SOLVOMET's take on the future of hydrometallurgy

Genuine breakthroughs in hydrometallurgy will not come from the use of neoteric solvents like ionic liquids or deep-eutectic solvents, but rather from a **deep understanding of hydro-processes at a molecular level**. Hydrometallurgy needs to evolve to low-energy-input **circular hydrometallurgy.**

(Prof. Koen Binnemans, August 2021)

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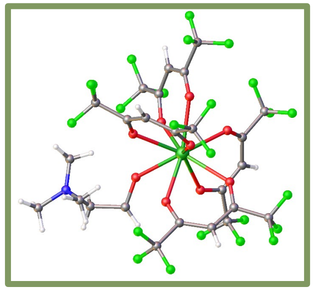
# SOLVOMET Group Research Topics

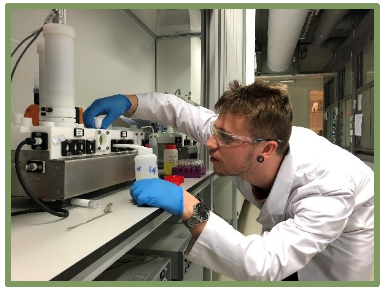
### Curiosity-driven fundamental and strategic basic research

- Solvent-assisted metal recovery (SX, direct reduction or hydrolysis in organic phase after SX, antisolvent precipitation, salt crystallisation, SX-assisted metal carbonation)
- Circular hydrometallurgy
- Molecular thermodynamics (incl. thermodynamic modelling)

### Problem-driven applied and competitive research

- Development of (near-circular, low-energy-input) hydro/solvometallurgical processes (incl. leaching, SX)
- Modelling of hydro/solvometallurgical processes
- Solvent formulation for SX processes
- Validation of leaching and separation processes on mini-pilot scale





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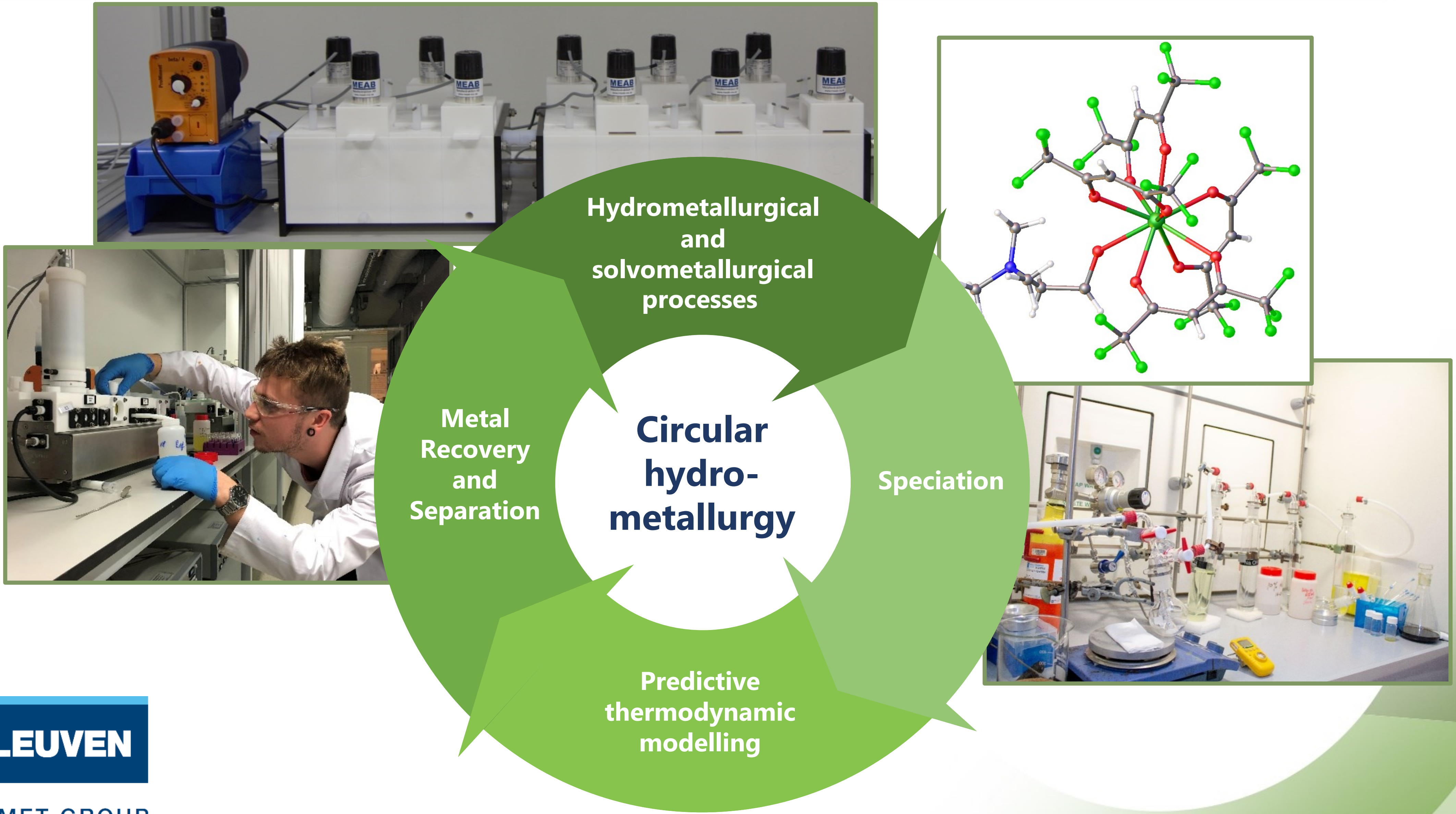
# SOLVOMET Group Research domains



# SOLVOMET Industrial Service Centre: Flagship services offered (general overview)

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**1. Recovery of metals from ores, solid industrial process residues and urban waste**

- ✓ Leaching at lab scale in batch reactors (up to 5 L), mortar grinder or in columns
- ✓ High-pressure leaching in autoclave reactor

**2. Removal of metals from liquid (aqueous or organic) process streams**

- ✓ After leaching, metal ions are recovered from pregnant leach solutions by (non-)aqueous SX (see next section), (non-) aqueous ion exchange (IX), cementation or precipitation

**3. Solvent extraction processes (SX)**

- ✓ SX processes studied from chemical point of view: mechanistic studies and kinetic studies + long-term stability studies of extractants and diluents
- ✓ SX tests in lab-scale mixer-settler batteries, batch extractors, pulsed columns or in series of separatory funnels

**4. Analytical services**

- ✓ QXRD, WDXRF, TXRF, Raman, ICP-OES, ICP-MS, NMR, UV-VIS, SEM-EDS, (EPMA)…

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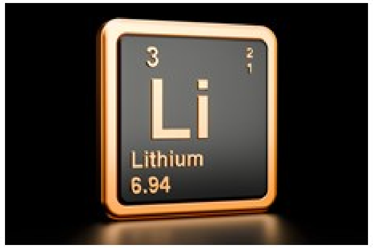
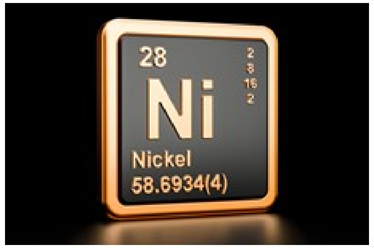
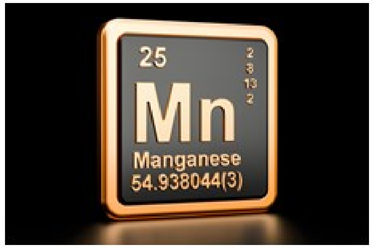
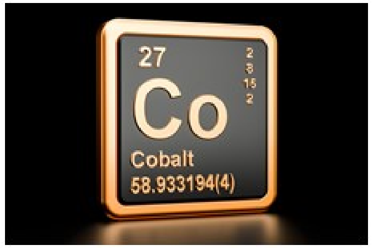
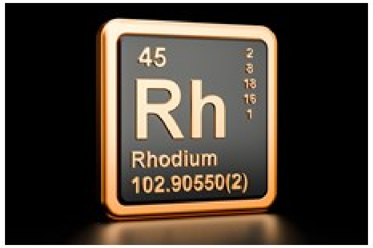
# SOLVOMET ISC's key metals of interest

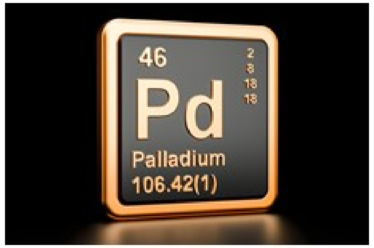


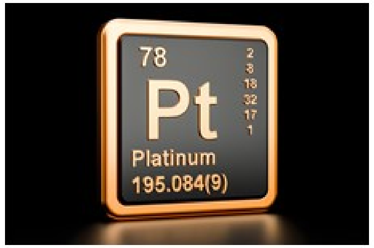



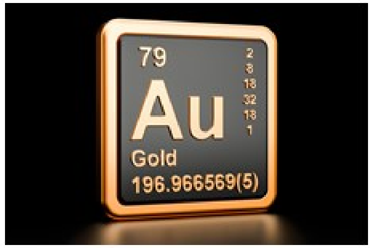



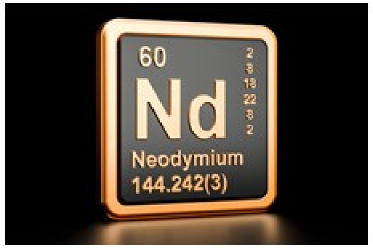



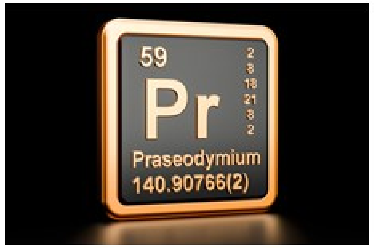



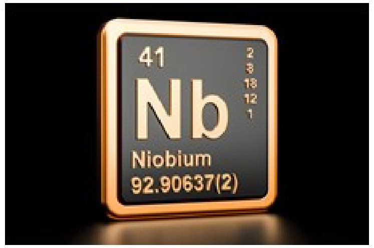



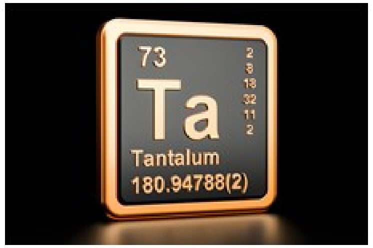



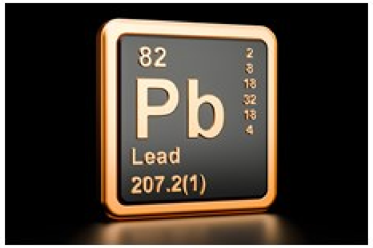



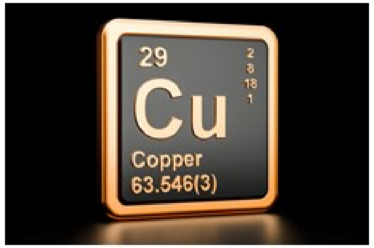



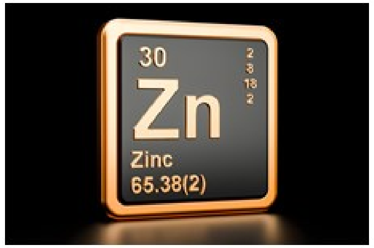



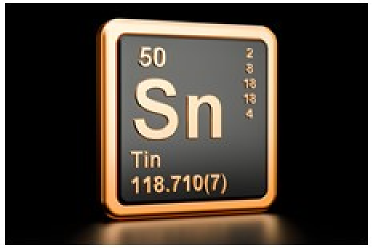

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# SOLVOMET Industrial Service Centre: Mini-pilot plant facilities for leaching

## High pressure reactor

- Effective capacity of 800 mL
- Made from stainless steel with PTFE liner
- Max. pressure = 200 bar
- Max. T = 230 °C with PTFE liner (and 300 °C without)


## Multiple reactor system

- 6 reactors ($V_{max}$ = 40 mL) with internal stirring
- Individual T & p control
- Max. p = 200 bar
- Max. T = 300 °C, heating rates up to 15 °C/min


## Batch leaching reactors

- Two jacketed reactors (1 & 5 L)
- pH and T control
- Digital overhead stirrer
- Filtration system included

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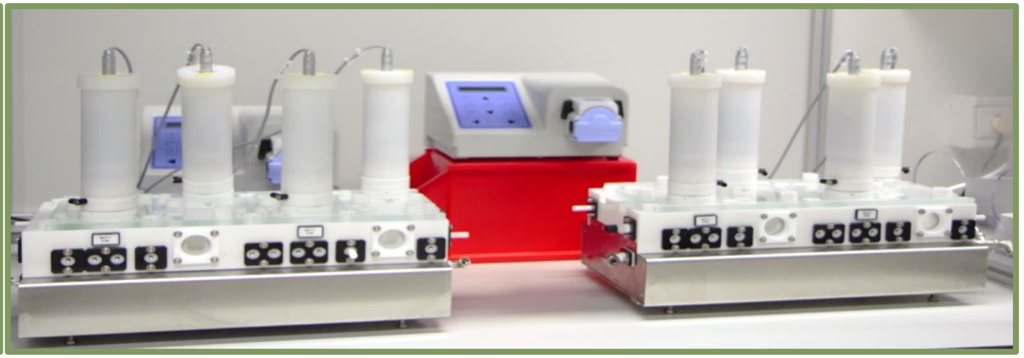
# SOLVOMET Industrial Service Centre: Mini-pilot facilities for continuous, countercurrent Solvent Extraction (SX) - Mixer-settlers

3 SX mixer-settler set-ups

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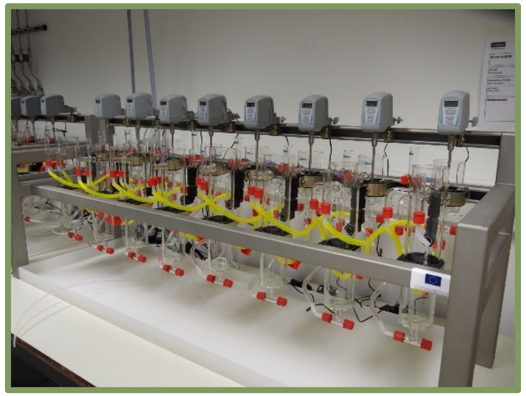
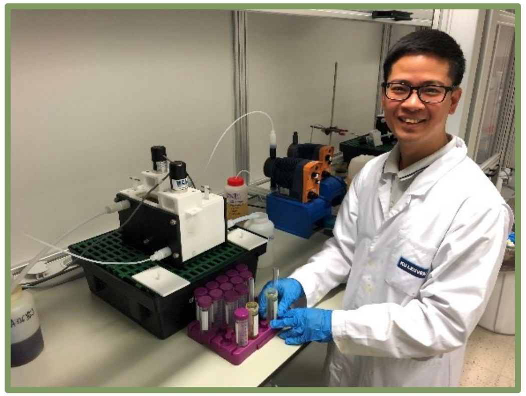
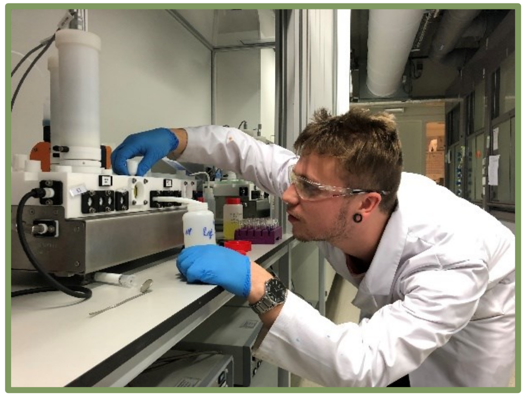
# SOLVOMET Industrial Service Centre: Mini-pilot facilities for continuous, countercurrent Solvent Extraction (SX) - Mixer-settlers

| Characteristics | SX Kinetics | MEAB | Rousselet Robatel |
|---|---|---|---|
| |  |  |  |
| Temperature | Only room temperature | Only room temperature | Heatable (water or oil) |
| Operability | Robust, easy to operate. Easy to assemble. | Robust, easy to operate. | Robust. Easy to assemble. The operation requires more attention. Mainly for research. |
| Visibility | Transparent | Opaque | Opaque but with a window in the settling chamber |
| Capacity | Mixer: 0.270 L, Settler: 1.050 L | Mixer: 0.12 L, Settler: 0.48 L | Mixer 0.035 L, Settler 0.143 L |
| Flows (depends on settling velocity) | Max flow: 10 L/h* | Max flow: 10 L/h | Max flow: 2-4 L/h |

*the limitation is for the pump, not the mixer-settler

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# SOLVOMET Industrial Service Centre: Mini-pilot facilities for continuous, countercurrent Solvent Extraction (SX) - Mixer-settlers

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| | Process | Equipment | Collaboration |
|---|---|---|---|
| **PLATIRUS project** | **Pt, Pd and Rh recovery from spent autocatalysts** | **MEAB MS** | JM Johnson Matthey Inspiring science, enhancing life; vito |
| **Bilateral project with industry** | **Cu recovery from high-grade Chrysocolla** | **1 L Hitec Zang leaching reactor and Rousselet MS** | Shell |
| **Fundamental research** | Li and Mg separation using binary extractants | Rousselet MS | |
| | Nd and Dy separation using ionic liquids | MEAB MS | |
| | Fe, Pb and Zn separation from DES | Rousselet MS | |
| | Y and Eu separation using non-aqueous solvent extraction | Rousselet MS | |

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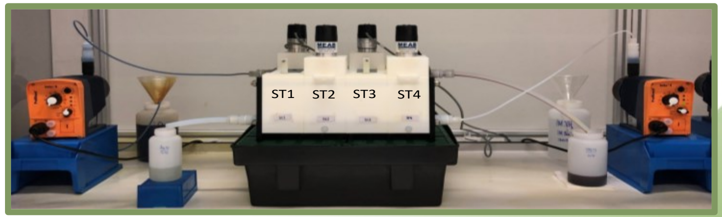

# SOLVOMET Industrial Service Centre: Mini-pilot facilities for continuous, countercurrent SX – (Agitated column SX)

## Kühni-type agitated column

For processes with low mass transfer, average residence time and high number of stages.

- Jacketed column made from glass with internals made from PEEK
- Max active volume: 0.9 L
- Active height: 1.2 m
- Total Flow: 5-25 L/h (both phases)

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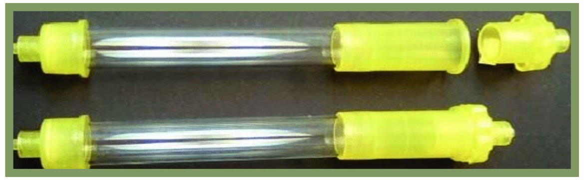
# SOLVOMET Industrial Service Centre: Lab facilities for Ion exchange work (lab-scale column IX set-up)

## Econo-chromatography columns

- Low-pressure (<1 bar) or gravity flow separations
- Used in various dimensions (*e.g.* 0.7 x 30 cm)



## CF-2 Fraction collector

- Equipped with drop sensor
- Capacity of 174 tubes
- Coupled with a peristaltic

## Ismatec IPC Peristaltic pump

- High-precision 8-channel dispenser
- Flow rates 0.002 – 44 ml/min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# SOLVOMET Industrial Service Centre: Key analytical facilities & services

TXRF


WDXRF


XRD

ICP-OES

ICP-MS (Multi Quadruple)

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# SOLVOMET Industrial Service Centre: Key analytical facilities & services

UV-VIS-NIR

FT-IR/Raman


NMR

Raman microscopy

# Publications deriving from bilateral projects with industry – on (solvo)leaching

SOLVOMET Industrial Service Centre

Journal of Sustainable Metallurgy
https://doi.org/10.1007/s40831-020-00294-3

RESEARCH ARTICLE

## Ammoniacal Solvoleaching of Copper from High-Grade Chrysocolla

Lukas Gijsemans[1] · Joris Roosen[1] · Sofía Riaño[1] · Peter Tom Jones[2] · Koen Binnemans[1]

Received: 22 June 2020 / Accepted: 14 September 2020


**Abstract**
The copper silicate ore chrysocolla forms a large potential copper resource, which has not yet been fully exploited, due to difficulties associated with its beneficiation by flotation and metallurgical processing. Direct acid leaching of chrysocolla causes silica gel formation. Therefore, in this work, the feasibility of solvometallurgical methods to leach copper from high-grade chrysocolla while avoiding issues with silica gel formation was assessed. Ammoniacal solvoleaching was performed with a solvent comprising the chelating extractant LIX 984 N or the acidic extractant Versatic acid 10 in an aliphatic diluent (ShellSol D70 or GTL Fluid G70), combined with a small volume of aqueous ammonia. In the three-phase system, aqueous ammonia dissolves copper from milled and sieved chrysocolla, while copper is simultaneously extracted to the organic phase, releasing ammonia that can be reused for further extraction. The best results were obtained with LIX 984 N as extractant: using a 50 vol% LIX 984 N solution, about 75% of copper could be extracted after 60 min of leaching at 25 °C. The stripping of copper from the pregnant leach solution was optimized. Quantitative stripping of copper was achieved with 1.89 M sulfuric acid and the final aqueous solution of copper sulfate had a concentration of 33 g $L^{-1}$. Experiments in a leaching reactor (1 L) and small battery of mixer-settlers (3 stages, 35 and 143 mL effective volume in the mixer and the settler, respectively, per stage) were successfully conducted and allowed to recover copper with a purity of 99.9%. A conceptual flow sheet has been developed.

Journal of Sustainable Metallurgy
https://doi.org/10.1007/s40831-020-00305-3

RESEARCH ARTICLE

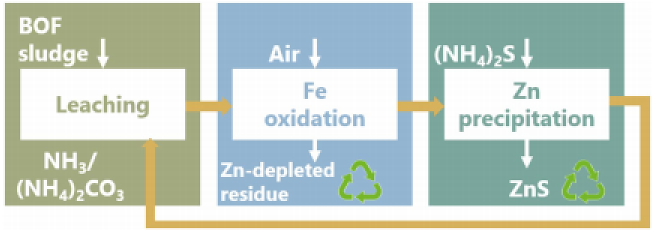
## Selective Removal of Zinc from BOF Sludge by Leaching with Mixtures of Ammonia and Ammonium Carbonate

Nerea Rodriguez Rodriguez[1] · Lukas Gijsemans[1] · Jakob Bussé[1] · Joris Roosen[1] · Mehmet Ali Recai Önal[1] · Victoria Masaguer Torres[2] · Álvaro Manjón Fernández[2] · Peter Tom Jones[3] · Koen Binnemans[1]

Received: 18 July 2020 / Accepted: 13 October 2020


**Abstract**
The zinc content of basic oxygen furnace (BOF) sludges is too high for direct recycling into the blast furnace via the sinter plant, as excessive zinc concentrations are detrimental for the refractory lining of the blast furnace. However, by partial and selective removal of zinc from the BOF sludge, the residual sludge can be used as a secondary iron resource in the blast furnace. In this paper, BOF sludge was leached with aqueous ammonia, aqueous solutions of ammonium salts (chloride, carbonate, and sulfate), and aqueous mixtures of ammonia and ammonium salt. The mixtures of ammonia and ammonium salt could leach more zinc with respect to either the aqueous ammonia or the aqueous ammonium salt solution. The ammonia–ammonium carbonate (AAC) mixture was selected as the most suitable lixiviant due to the high zinc leaching efficiency in combination with a high selectivity towards iron; furthermore, this combination does not introduce unwanted chloride or sulfate impurities in the residue. The leaching process was optimized in terms of the liquid-to-solid ratio, total ammonia concentration, ammonium:ammonia molar ratio, temperature, and leaching time. The co-dissolved iron was precipitated as a hydroxide after oxidation of ferrous to ferric ions by an air stream, without co-precipitation of zinc, while the dissolved zinc could be easily recovered as zinc sulfide by precipitation with ammonium sulfide. The (almost) closed-loop process was successfully up-scaled from 10 mL to 1 L scale.

**Graphical Abstract**

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# Publications deriving from bilateral projects with industry – on SX

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ACS Sustainable Chemistry & Engineering
pubs.acs.org/journal/ascecg
Research Article

## Solvent Extraction of Gold(III) with Diethyl Carbonate

Stijn Raiguel, Lukas Gijsemans, Arne Van den Bossche, Bieke Onghena, and Koen Binnemans*

Cite This: ACS Sustainable Chem. Eng. 2020, 8, 13713–13723 Read Online

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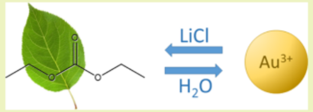
**ABSTRACT:** Diethyl carbonate (DEC) was evaluated as a green, renewable alternative to methyl isobutyl ketone and dibutyl carbitol for the recovery of gold from copper-rich sources, such as anode slimes, by solvent extraction from chloride solutions. DEC is a powerful extractant at high chloride concentrations and can be used to sequester and concentrate gold from large volumes of dilute aqueous solutions containing high concentrations of copper. Stripping can be performed using pure water. Different variables were investigated: DEC concentration, chloride concentration, chloride source, phase volume ratio, and equilibration time. The selectivity and phase disengagement times were studied, and possible mechanisms of extraction are discussed and evaluated. In addition, the stability of DEC against hydrolysis was measured.




**KEYWORDS:** *anode slime, chloride hydrometallurgy, copper, precious metal refining, solvent extraction*

Chemical Engineering Research and Design 161 (2020) 304–311
Contents lists available at ScienceDirect
Chemical Engineering Research and Design
journal homepage: www.elsevier.com/locate/cherd
ELSEVIER IChemE

## Selection criteria of diluents of tri-*n*-butyl phosphate for recovering neodymium(III) from nitrate solutions

*Mercedes Regadío**, Nagaphani Kumar Batchu, Koen Binnemans*
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ARTICLE INFO

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*Keywords:*
Diluents
Neutral extractant
Rare earths
TBP
Solvent extraction

ABSTRACT

The selection of a proper diluent should be based on several criteria such as the distribution ratio, phase disengagement time, cost, safety and environmental impact of the process. The effect of different diluents on the solvent extraction of Nd(III) by the neutral extractant tri-*n*-butylphosphate (TBP) from nitrate feed solutions was studied. The nature of the diluent had little effect on the extraction kinetics of Nd(III) by TBP above 2.5 min. In general, phase disengagement times were relatively shorter for aromatic diluents compared to aliphatic diluents. Conversely, extraction efficiencies were the highest for aliphatic diluents, slightly lower for mixed aliphatic-aromatic diluents and much lower for aromatic diluents. The poorer extraction efficiencies of aromatic diluents maybe due to the lower concentration of free extractant as a result of the stronger interactions of the diluent with water and/or of the diluent with the extractant. Under the experimental conditions, the differences in extraction between aliphatic and aromatic diluents decreased with increasing the salting-out effect of nitrate ions in the feed. At nitrate concentrations of 4.5 mol $L^{-1}$ or more, the different diluents had a limited influence on the metal extraction with 1 mol $L^{-1}$ TBP from feed solutions of 1 g $L^{-1}$ Nd(III). Thus, under these conditions, the selection of the diluent can be preferably based on its cost, safety and biodegradability rather than on its physico-chemical properties.


Hydrometallurgy 177 (2018) 146–151
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Hydrometallurgy
journal homepage: www.elsevier.com/locate/hydromet
ELSEVIER

## Effect of the diluent on the solvent extraction of neodymium(III) by bis(2-ethylhexyl)phosphoric acid (D2EHPA)

Nagaphani Kumar Batchu, Koen Binnemans*
*KU Leuven, Department of Chemistry, Celestijnenlaan 200 F, bus 2404, B-3001 Heverlee, Belgium*

ARTICLE INFO

*Keywords:*
D2EHPA
Diluents
Lanthanides
Rare earths
Solvent extraction

ABSTRACT

The effect of different types of diluents on the extraction of neodymium(III) from a chloride aqueous feed solution by the acidic extractant bis(2-ethylhexylphosphoric) acid (D2EHPA) was investigated. A total of 11 aliphatic, mixed aliphatic-aromatic and aromatic diluents were considered. D2EHPA was very well miscible with all diluents, even at 90% (v/v) of extractant. Aliphatic diluents gave the highest extraction efficiencies, while aromatic diluents did suppress the formation of emulsions or gels. Although a good separation of Dy(III) over Nd(III) was observed at 0.5 mol/L D2EHPA in all diluents, aromatic diluents were found to yield the highest separation factors. This study shows that the effect of the diluent on the extraction behaviour of Nd(III) is primarily determined by the aliphatic/aromatic content of the diluent. Diluents with the same concentration of aromatics show very similar behaviour. The choice of a diluent can be based on factors other than the extraction behaviour, for instance price and volatility.

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# Some key achievements by SOLVOMET

## Methanesulfonic acid: a sustainable acidic solvent

Recovery of rare earths from the green lamp phosphor $LaPO_4:Ce^{3+},Tb^{3+}$ (LAP) by dissolution in concentrated methanesulphonic acid

From the journal: RSC Advances


Remaghic

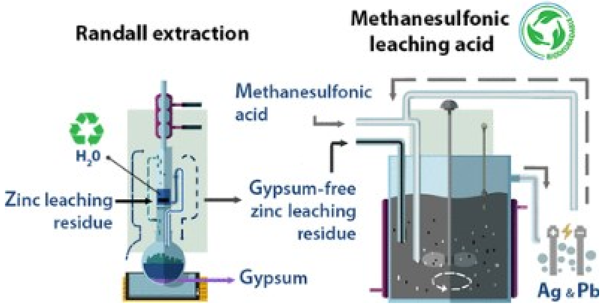
Methanesulfonic acid: a sustainable acidic solvent for recovering metals from the jarosite residue of the zinc industry†

From the journal Green Chemistry


SOCRATES

Recovery of Lead and Silver from Zinc Leaching Residue Using Methanesulfonic Acid

From the journal ACS Sustainable Chemistry and Engineering




nyrstar

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# Broad industrial network

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## Bilateral projects

umicore materials for a better life
Aurubis Beerse
Shell
ArcelorMittal
TOYOTA
MICHELIN
Lithium Americas
ICL
nyrstar
Meclex

## Previous collaborations / Active collaborations within H2020 projects

JM Johnson Matthey Inspiring science, enhancing life
TREIBACHER INDUSTRIE AG
TOYOTA
LCM less common metals
CLC Cobre Las Cruces, S.A.
METALLO AURUBIS GROUP
MEAB
INDAVER
nyrstar
NEW BOLIDEN
ALUMINIUM OF GREECE
umicore materials for a better life
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# SOLVOMET's academic network

## Some key Projects

SOLCRIMET – erc Advanced Grant

SAMEX

MSCA-ETN SULTAN

SOCRATES EU MSCA-ETN

tarantula

METGrow+

NEMO

CROCoDILE

Platirus

HE ENiCoN

HE HEPHAESTUS

## Key Partners

RWTH AACHEN UNIVERSITY

University of Leicester

vito

tecnalia

LiU

CHALMERS UNIVERSITY OF TECHNOLOGY

Aalto University

TU Delft – Delft University of Technology

brgm

GTK

UNIVERSITY OF HELSINKI

National Technical University of Athens

LIÈGE université

UNIVERSITY OF BIRMINGHAM

Universiteit Utrecht

universität bonn

VTT

UNIVERSITEIT GENT

ETH zürich

Universität Zürich

HZDR

TECHNISCHE UNIVERSITÄT BERGAKADEMIE FREIBERG – Die Ressourcenuniversität. Seit 1765.

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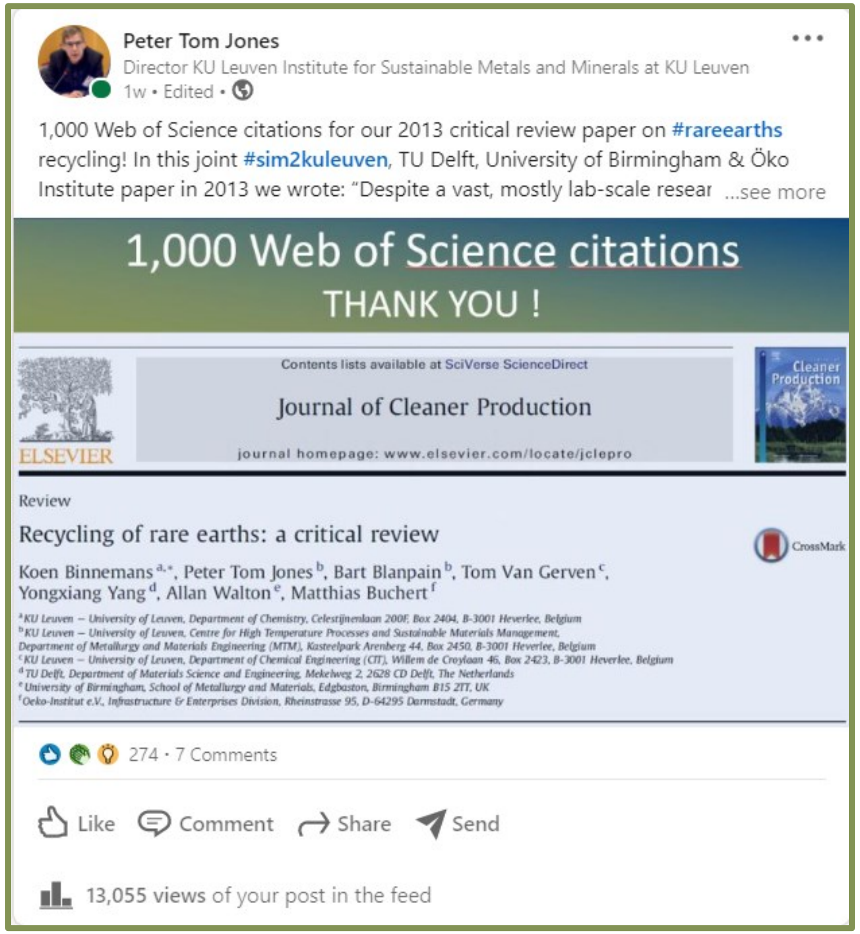
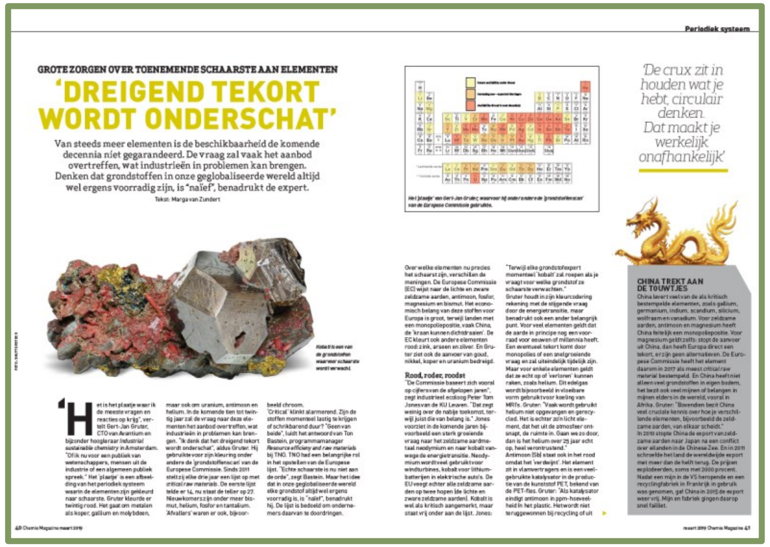
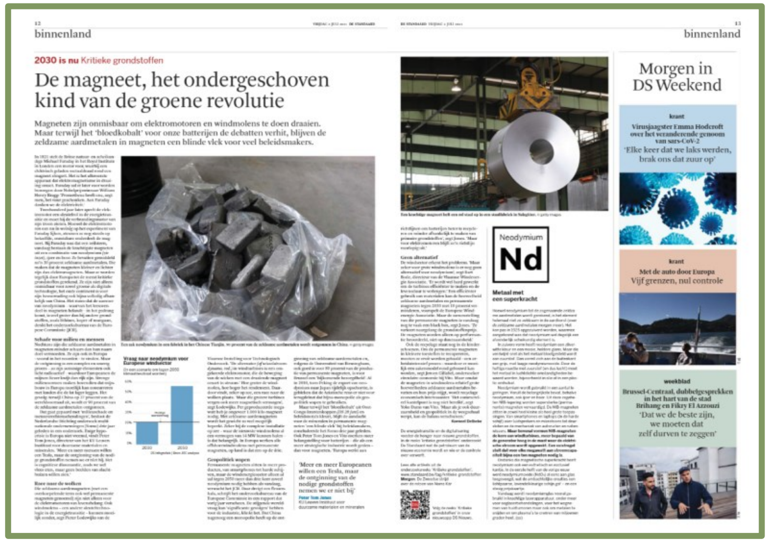
# Scientific & societal impact





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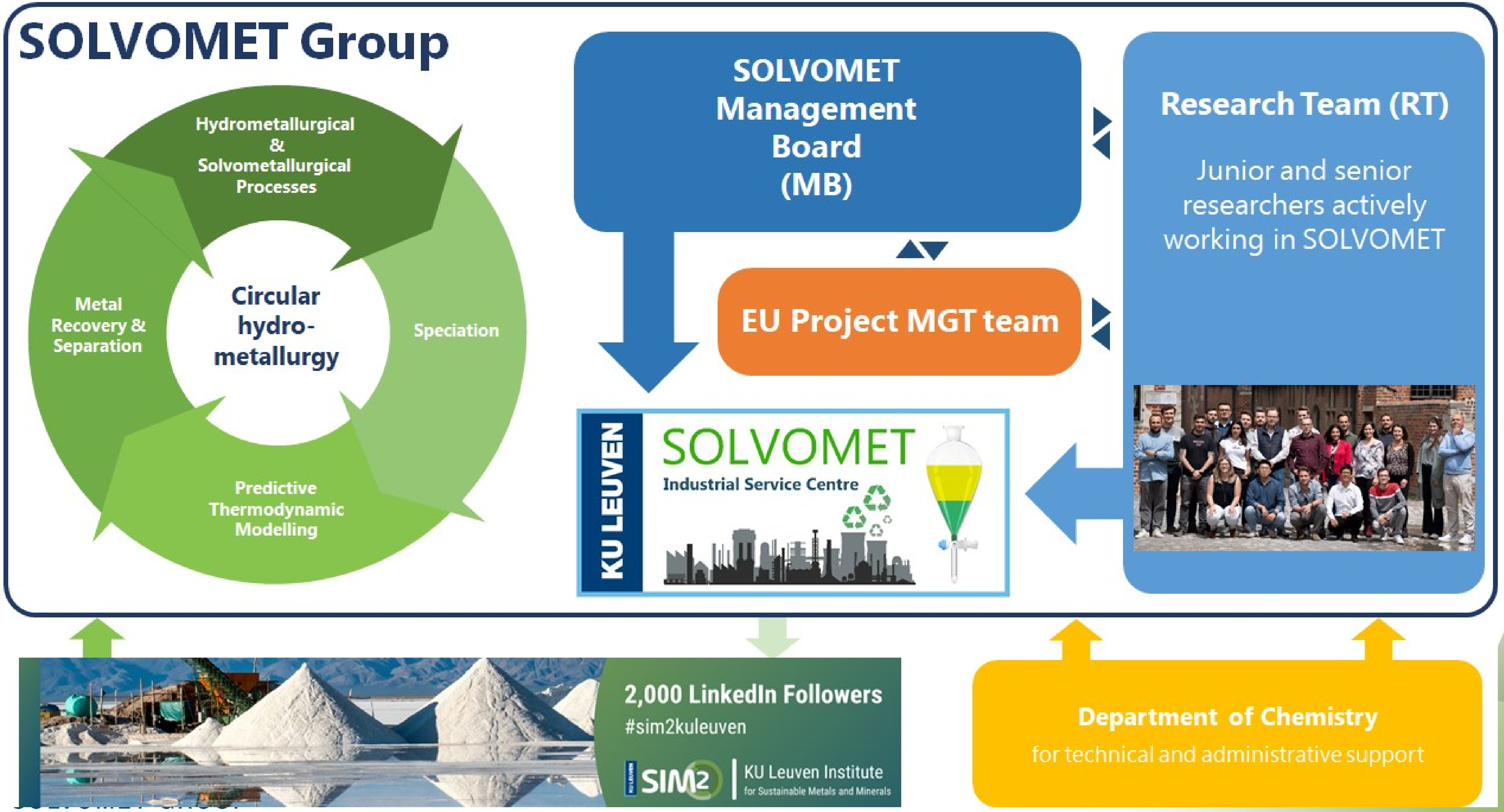
# SOLVOMET Group Governance



# SOLVOMET Industrial Service Centre: Project types

| Services Agreement (short-term) | Long-term framework agreement | Industrial PhD project |
|---|---|---|
| • **Project description**: 4-page document describing tasks, deliverables, milestones, budget, timing etc.<br>• **IP**: foreground IP transferred to company<br>• **Duration**: typically 6 to 12 months<br>• **Cost**: salaries of the involved researchers + 50% extra for working budget (chemicals, lab use, travel…)).<br>• **Publications:** not planned unless explicitly desired by company in terms of dissemination goals<br>• **Ideal for fast delivery of (confidential) results by experienced (permanent staff) research experts/managers & research associates** | • **Project description**: 4-page document research programme, budget<br>• **IP**: foreground IP transferred to company<br>• **Duration**: 2 to 5 years<br>• **Cost**: salaries of the involved researchers + 50% extra for working budget (chemicals, lab use, travel…)).<br>• **Publications:** not planned unless explicitly desired by company in terms of dissemination goals<br>• **Ideal for long-term, in-depth, research support provided by experienced (permanent staff) research experts/managers & research associates** | • **Project description**: 4-page document with research programme, budget<br>• **IP**: foreground IP transferred to company<br>• **Duration**: 4 years<br>• **Cost**: ~95,000 euro/year [salary + 50% (overhead + working budget for chemicals, lab use, travel…)]<br>• **Publication clause:** Allowing to publish more generic parts of research while keeping the rest confidential<br>• **Equitable remuneration principle**: e.g. preferred partnership for follow-up projects<br>• **Ideal for first-class training of PhD researcher that can go and work for the company later** |

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# SOLVOMET is embedded in the KU Leuven Institute for Sustainable Metals and Minerals (SIM² KU Leuven)

KU LEUVEN SOLVOMET Industrial Service Centre

**Research Line 1**

Geological exploration and advanced resource characterisation

**Research Line 2**

Remanufacturing and demanufacturing

**Research Line 3**

Sustainable metallurgical processes

**Research Line 4**

Upcycling processes for primary and secondary resources

**Research Line 5**

Sustainability assessment and policy research


**Research Line 6**

Process intensification and digitalisation

SIM² KU Leuven's mission is to develop, organise and implement problem-driven, science-deep research and future-oriented education, contributing to the environmentally friendly production and recycling of metals, minerals and engineered materials, supporting the transition to a climate-friendly, circular-economy.

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https://chem.kuleuven.be/solvomet
https://solvomet.eu
https://kuleuven.sim2.be/

Contact: Peter Tom Jones (LinkedIn profile)