

SOLVOMET Group Laboratory of Metallurgical Chemistry





SOLVOMET Group Mission

The dual mission of the SOLVOMET Group 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 and RTD) partners in the conceptual and practical development of more sustainable solvometallurgical (and hydrometallurgical) separation processes and new mining chemicals, which are subsequently tested using state-of-the-art lab-scale and mini-pilot-scale experimental facilities.

SOLVOMET Group Vision

The vision of the SOLVOMET Group is that chemistry can help to provide in a sustainable way the metals needed by future generations.

In this sense, the SOLVOMET Group takes advantage of the unique properties of organic molecules for developing more efficient and eco-friendly solvo-metallurgical and hydrometallurgical processes.









- Full professor at the Department of Chemistry, specialised in solvometallurgy and hydrometallurgy
- Core expertise in critical metals and solvent extraction (SX)
- Author of more than 500 papers, **H-index = 74**, > 25,000 citations
- 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 Solvometallurgy
- Steercom Member of KU Leuven Institute for Sustainable Metals and Minerals (SIM² KU Leuven)
- Was a Steercom Member of the European Rare Earth Competency Network (ERECON)
- Elected member of the Royal Flemish Academy of Belgium for Science and the Arts (KVAB)







SOLVOMET Group Mission



Curiosity-driven fundamental and strategic basic research

- Design (in-silico) and synthesis of new extractants
- Coordination chemistry of extractants and speciation studies
- Extraction behaviour of extractants and extraction mechanisms
- Modeling of SX equilibria

Problem-driven applied and competitive research

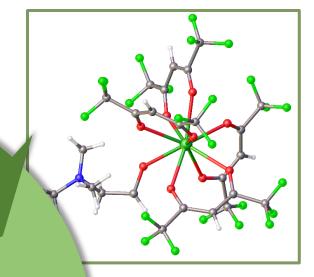
- Development of solvometallurgical and hydrometallurgical processes
- Modeling of solvometallurgical and hydrometallurgical processes
- Solvent formulation for SX processes
- Validation of separation processes on mini-pilot scale

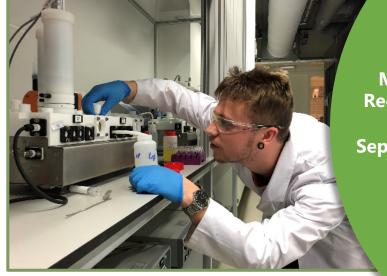


SOLVOMET Group Research domains









Metal Recovery and Separation



Extractant Design and Solvent Formulation







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



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 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) or precipitation

3. Solvent extraction processes (SX)

- ✓ SX processes studied from chemical point of view: mechanistic studies and kinetic studies + longterm 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...



SOLVOMET Industrial Service Centre:

Mini-pilot plant facilities for (solvo)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)



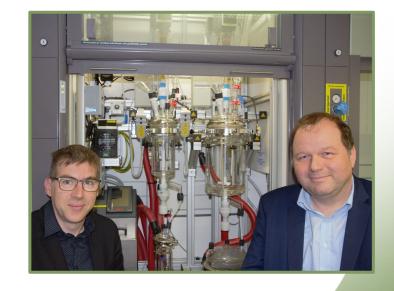
Multiple reactor system

- 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





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







3 SX mixer-settler set-ups





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



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

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



	Process	Equipment	Collaboration
PLATIRUS project	Pt, Pd and Rh recovery from spent autocatalysts	MEAB MS	JM Johnson Matthey Inspiring science, enhancing life
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	







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)





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





SOLVOMET Industrial Service Centre:

Key analytical facilities & services















SOLVOMET Industrial Service Centre:

Key analytical facilities & services











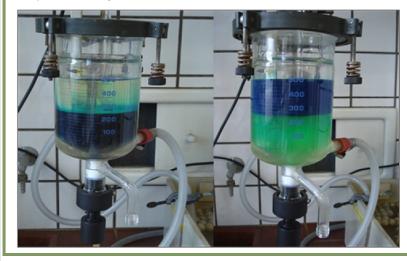


Some key achievements by SOLVOMET



Ionic liquid process for Co/Ni separation

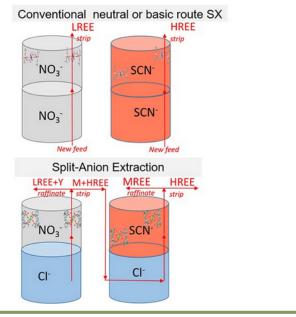
In collaboration with Umicore Research (Olen, Belgium), Prof. Koen Binnemans has developed a new ionic liquid solvent extraction process for separation of cobalt and nickel. Cobalt is efficiently extracted to the ionic liquid phase, while nickel is left behind in the raffinate. The advantages of the new process are: (1) the unprecedented high separation factor (> 50,000), which is nearly an order of magnitude larger than the industrial benchmark processes, and (2) the very easy stripping of cobalt from the ionic liquid phase. The process was demonstrated on a mini-pilot scale in batch reactors and in a continuous process with mixer-settlers. In fact, this study provided the proof-of-principle that ionic liquids can be used on an industrial scale for separation of metals by solvent extraction.





Separation of rare earths by split-anion extraction with ionic liquids

There are two main challenges in the field of separation of rare earths by solvent extraction: (1) the development of a highly selective extraction agent that can remove one rare-earth element from a mixture without co-extraction of the other elements; (2) the development of a process that allows extraction of rare earths from a chloride solution to an organic phase by a solvating extractant. Extraction from chloride solution is preferred by industry because hydrochloric acid is much cheaper than nitric acid, and because waste water treatment is simpler in case of chlorides compared to nitrates. Solvating extractants are the preferred type of extractants, because of the possibility of loading a high concentration of rare earths in the organic phase and the easy back extraction of the rare earths from the organic phase by water instead of strong acids. Within the framework of the FP7 project EURARE on the exploitation of European rare-earth deposits, Prof. Binnemans invented a new process, called "split anion extraction" with makes the extraction of rare earths from a chloride solution possible by extraction with a nitrate ionic liquid via a solvating mechanism.







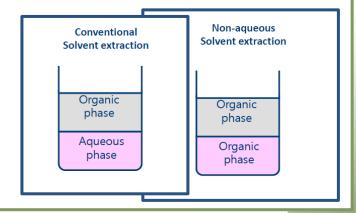
Separation of rare-earth ions from ethylene glycol (+LiCl) solutions by non-aqueous solvent extraction with Cyanex 923†

Nagaphani Kumar Batchu, ^a Tom Vander Hoogerstraete, ¹

^a Dipanjan Banerjee^b and Koen Binnemans ¹

^a

The separation of a mixture of rare earths by non-aqueous solvent extraction with two immiscible organic phases has been studied. The more polar organic phase was ethylene glycol with dissolved lithium chloride and the less polar organic phase was the extractant diluted in n-dodecane. Cyanex 923 was found to be the most performant extractant amongst the investigated acidic, basic and solvating extractants: Cyanex 272, Cyphos IL 101, Aliquat 336, bis(2-ethylhexyl)amine, trioctylphosphine oxide (TOPO) and Cyanex 923. The replacement of the aqueous chloride feed solutions by non-aqueous ethylene glycol feed solutions had a profound effect on the distribution ratios and separation factors. The separation factors for extraction of pairs of rare earths from aqueous chloride solutions by Cyanex 923 are too low to be of practical use. On the contrary, a mixture of rare earths can be separated conveniently in four different groups by extraction with Cyanex 923 from ethylene glycol (+LiCl) solutions. The influence of several parameters such as the chloride concentration, the type of chloride salt, the addition of other polar solvents to the of complexing agents to the ethylene glycol phase has been studied. The extraction mechanism for extraction of ytterbium(iii) was studied by slope analysis experiments. The ytterbium(iii) ethylene glycol phase and the extracted species in the n-dodecane phase were determined by EXAFS. Furthermore, a conceptual flow sheet for the fractionation of rare earths from an ethylene glycol (+LiCl) feed solution into different groups by extraction with Cyanex 923 has been proposed. The new extraction system is useful for extraction of scandium and for separation of scandium from the other REEs.











Some key achievements by SOLVOMET



Methanesulfonic acid: a sustainable acidic solvent

Recovery of rare earths from the green lamp phosphor LaPO₄:Ce³⁺,Tb³⁺ (LAP) by dissolution in concentrated methanesulphonic acid







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



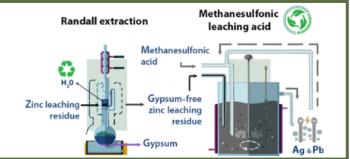




Recovery of Lead and Silver from Zinc Leaching Residue Using Methanesulfonic Acid



From the journal ACS Sustainable Chemistry and Engineering







Publications deriving from bilateral projects with industry – on solvoleaching



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 10 · Joris Roosen 10 · Sofía Riaño 10 · Peter Tom Jones 20 · Koen Binnemans 10

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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

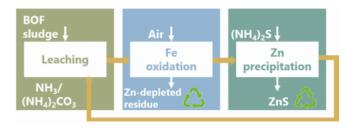
Nerea Rodriguez Rodriguez¹ · Lukas Gijsemans¹ · Jakob Bussé¹ · Joris Roosen¹ · Mehmet Ali Recai Önal¹ · Victoria Masaguer Torres² · Álvaro Manjón Fernández² · Peter Tom Jones³ · Koen Binnemans¹

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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.

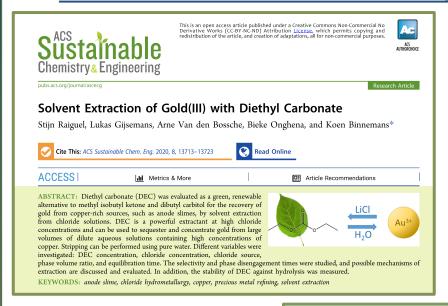
Graphical Abstract

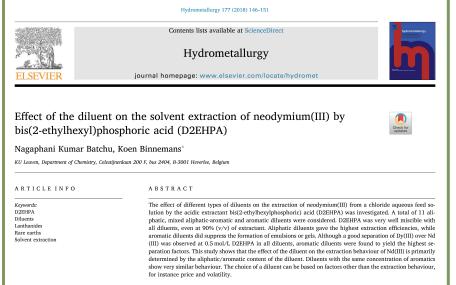




Publications deriving from bilateral projects with industry - on SX









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



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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 trin-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 $4.5\,\mathrm{mol}\,\mathrm{L}^{-1}$ 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. © 2020 Institution of Chemical Engineers. Published by Elsevier B.V. All rights reserved.



Broad industrial network



Bilateral projects

Previous collaborations / Active collaborations within H2020 projects

























umicore materials for a better life





















SOLVOMET's academic network



















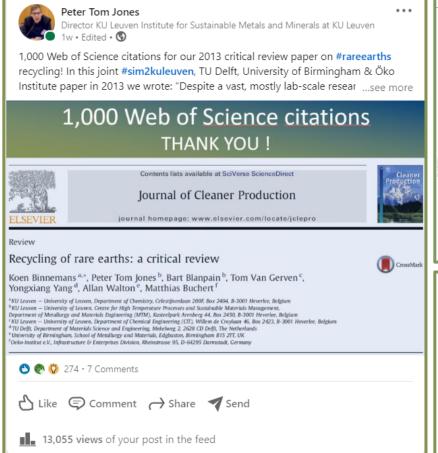






Scientific & societal impact







Om tot een milieuvriendelijkere wereld te komen, moeten we materialen en energie



De crux zit in houden wat ie

hebt, circulair

Dat maakt je werkeliik

onafhankelijk'



De investeringen moeten leiden tot een beleid dat import cor

bineert met eigen winning en recycling

we alleen al voor batterijen voor elektrische wagens en opslag achttien en zestig keer meer lithium nodig zullen

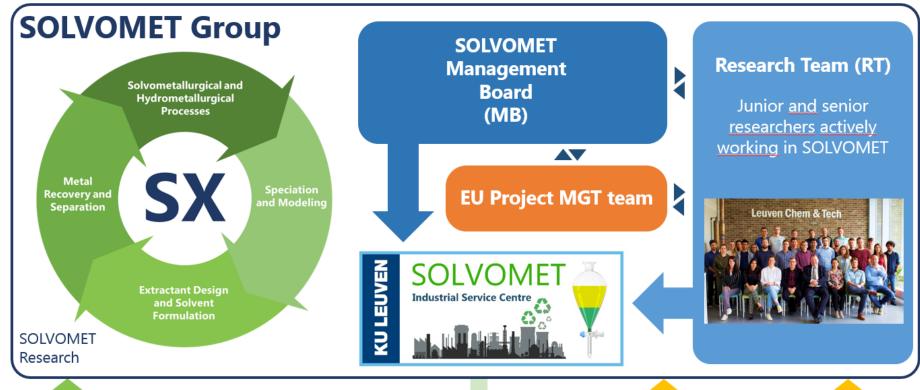
hebben tegen respectievelijk 2030 en

20 EOS

KU LEUVEN

SOLVOMET Group Governance







Department of Chemistry

for technical and administrative suppor



SOLVOMET Industrial Service Centre: Project types



Services Agreement

- Project description: 4-page document describing tasks, deliverables, milestones, budget, timing etc.
- **IP**: foreground IP transferred to company
- Duration: typically 6 to 12 months
- **Cost**: salaries of the involved researchers + 50% extra for working budget (chemicals, lab use, travel...)).
- Publications: not planned unless explicitly desired by company in terms of dissemination goals
- Ideal for fast delivery of (confidential) results by experienced, hands-on researchers

Industrial PhD project

- **Project description**: 4-page document with research programme, budget
- IP: foreground IP transferred to company
- **Duration**: 4 years
- Cost: ~95,000 euro/year [salary + 50% (overhead + working budget for chemicals, lab use, travel...)]
- Publication clause: Allowing to publish more generic parts of research while keeping the rest confidential
- Equitable remuneration principle: e.g. preferred partnership for follow-up projects
- Ideal for first-class training of PhD researcher that can go and work for the company later

Industrial postdoc project

- **Project description**: 4-page document research programme, budget
- **IP**: foreground IP transferred to company
- **Duration**: 1 to 2 years
- Cost: ~120,000 euro/year [salary + 50% (overhead + working budget for chemicals, lab use, travel...)]
- Publication clause: Allowing to publish more generic parts of research while keeping the rest confidential
- Equitable remuneration principle: e.g. preferred partnership for follow-up projects
- Ideal for advanced, first-class training of postdoc researcher that can go and work for the company later



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





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

simplement problemativen, 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.





All info on SOLVOMET: www.solvomet.eu





SOLVOMETALLURGY?

using non-aqueous solutions. Solvometallurgy discrete water phase.

testing. Concurrently. SOLVOMET offers of solvometallurgy. analytical services as well.

Read more >



Solvometallurgy is the extraction of metals from SOLVOMET is specialised in 1. solvometallurgical ores, extractive waste, industrial process recovery of metals from low-grade ores, residues, production scrap and urban waste extractive waste, industrial process residues and metal-rich urban waste, 2. removal of metals differs from hydrometallurgy by the absence of a from liquid streams, and 3, solvent extraction

SET UP A PROJECT?



SOLVOMET offers lab-scale and mini-pilot SOLVOMET is your experienced partner for both infrastructure for solvometallurgical leaching and bilateral (contract research & consultancy) solvent extraction process development and projects and EU/national projects in the domain

Welcome to SOLVOMET



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Mini-pilot scale equipment

The SOLVOMET centre is continuously investing in mini-pilot scale equipment for leaching and solvent extraction experiments, allowing to tackle upscaling issues (from the lab-scale to the mini-pilot scale level) and to assist in the development of novel, commercial solvometallurgical flow sheets. Upscaling questions that are investigated by the SOLVOMET centre include:

- 1. How can the solid-liquid separation be done after leaching, especially in case of viscous liquids?;
- 2. How can the solvent be recycled after leaching?;
- 3. How can loss of solvent on the solid particles be minimised?

1. Mini-pilot scale solvent extraction studies

Batch solvent extraction processes can be scaled up to a mini-pilot scale in a continuous counter current mode in our mixer-settler set-up, with the final goal to examine the possibility of scaling up the process and/or producing larger volumes of purified metal solution. First, a flow sheet is designed to determine the number of stages and optimal aqueous-to-organic phase ratio of extraction, scrubbing and stripping based on McCabe-Thiele diagrams. Furthermore, the residence time is stablished. Next, the process is run in the mixer-settlers. Samples of each stage are taken on a regular basis to evaluate the performance and make

SX Battery 1 (Rousselet)



The mixer settler set-up of Rousselet Robatel consists of 12 stages, that can be heated up to 80 °C using a circulating water bath. The stages are each isolated and made of PVDF. The volume of the mixer and settler chambers in each stage is 35 mL and 143 mL, respectively. The total flow rate can reach up to 6 L/h.

SX Battery 2 (MEAB)



The mixer settler set-up of MEAB consists of 27 stages in total, which can be combined into extraction scrubbing and stripping steps according to the objected flow sheet of the solvent extraction process. The mixer settlers are made of PVDF and can be used at room temperature (heating is not possible). The volume of the settler chamber is 480 mL and a max total flow rate up to 10 L/h can be achieved depending on the physical characteristics of both phases and the behavior of the mixture.

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