

1-cycle SANEX process development studies performed at Forschungszentrum Jülich

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Abstract – *In the framework of our research activities related to the partitioning of spent nuclear fuel solutions, the direct selective extraction of trivalent actinides from a simulated PUREX raffinate solution (1-cycle SANEX) was studied using a mixture of CyMe₄BTBP and TODGA. The solvent showed a high selectivity for trivalent actinides with a high lanthanide separation factor. However the coextraction of some fission products, such as Cu, Ni, Zr, Mo, Pd, Ag and Cd was observed. The extraction of Zr and Mo could be suppressed using oxalic acid but the use of the well-known Pd complexant HEDTA was unsuccessful. During screening experiments with different amino acids, the sulphur-bearing amino acid L-Cysteine showed good complexation of Pd and prevented its extraction into the organic phase without influencing the extraction of trivalent actinides. A strategy for a single-cycle process is proposed within this paper.*

A. INTRODUCTION

The development of new and innovative processes for the processing of spent nuclear fuel solutions is a very intensively studied topic in nuclear research all over the world [1, 2]. As the liquid aqueous waste solution from reprocessing contains approx. 40 different elements in concentrations ranging from a few milligrams up to several grams per litre, the selective separation of trivalent actinides from this multi-element solution is one of the most challenging problems. The separation of the trivalent actinides from the lanthanides is a particularly difficult step, as the two groups of f-elements have very similar physical and chemical properties.

In Europe, the DIAMEX-SANEX (DIAmide EXtraction - SElective ActiNide EXtraction) partitioning process is one of the most promising strategies, which is foreseen to be converted from lab scale to industrial scale. The first step of this process (DIAMEX) uses a diamide extractant to coextract lanthanides and minor actinides from the highly acidic PUREX raffinate [3, 4]. In the subsequent step (SANEX), the trivalent actinides are separated from the lanthanides e.g. by the highly selective CyMe₄BTBP extractant [5, 6]. A drawback of such a process design is the need for two separate processes using two different ligands. Within the current European project ACSEPT (Actinide reCYcling by SEparation and Transmutation), a new process design is envisaged, the so-called “innovative SANEX” concept. In this strategy, the trivalent actinides and lanthanides are coextracted in a DIAMEX-type process. Then, the loaded solvent is subjected to several stripping steps. The first one concerns selectively stripping the trivalent actinides with selective water-soluble ligands followed by the subsequent stripping of trivalent lanthanides [7].

A more challenging route studied within this paper is the direct actinide (III) separation from the PUREX raffinate using a mixture of CyMe₄BTBP and TODGA (structures shown in Figure 1) as extractants, the so-called 1-cycle SANEX process. A single process directly using the PUREX raffinate would reduce the number of cycles, thus saving the DIAMEX process, making the complete advanced reprocessing process more economical and easier.

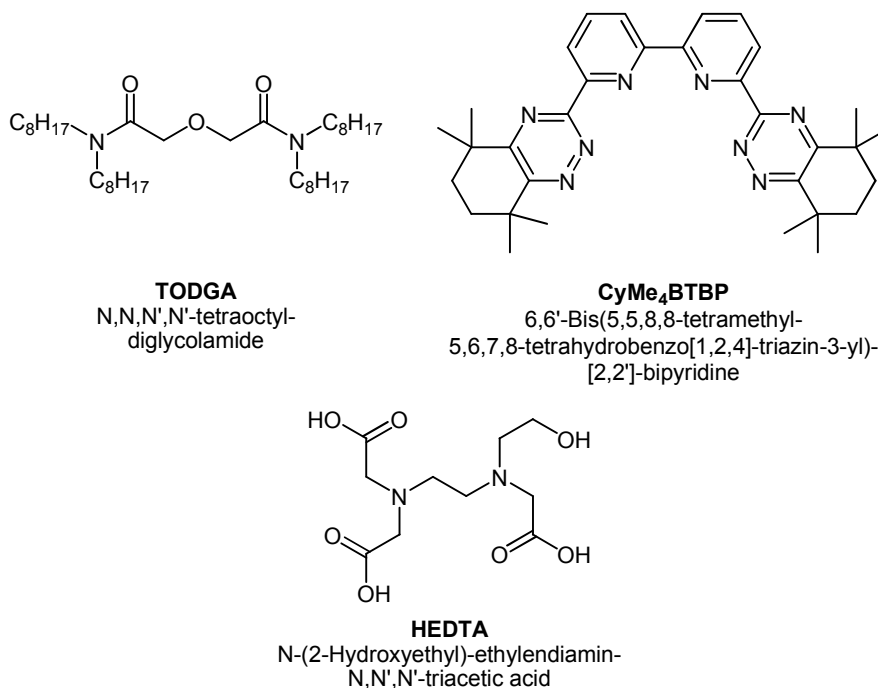


Figure 1: Structures of TODGA, CyMe₄BTBP and HEDTA

Geist et al. have shown the CyMe₄BTBP molecule to be a very selective extractant for the separation of actinides from lanthanides [8]. Magnusson et al. demonstrated the performance of CyMe₄BTBP in a hot SANEX test using 0.25 mol/L of the malonamide DMDOHEMA as phase transfer catalyst [5]. The use of a phase transfer catalyst is necessary owing to the slow kinetics of the CyMe₄BTBP-molecule which can be significantly improved by the use of DMDOHEMA. Modolo et al. recently proposed that 0.25 mol/L DMDOHEMA can be substituted with 0.005 mol/L TODGA [9]. This system shows comparably good extraction properties and kinetic behaviour compared to DMDOHEMA, and the performance of the system was demonstrated in a spiked test. Both experiments used a DIAMEX raffinate solution containing actinides and lanthanides as a SANEX feed solution for the experiments. After a 20-stage counter-current process, an actinide product fraction containing >99.9% of the actinides with less than 0.1% lanthanides was obtained. Despite these very successful tests, the question arose as to whether it would be possible to directly and selectively extract the actinides from a PUREX raffinate solution leaving the lanthanides and the other fission products in the aqueous phase.

B. RESULTS AND DISCUSSION

B.1. Extraction from simulated HAR solution

Batch extraction studies were carried out with a synthetic PUREX raffinate. An organic phase consisting of 0.015 mol/L CyMe₄BTBP and 0.005 mol/L TODGA diluted in a mixture of TPH and 1-Octanol (40/60 v/v) was used as extractant. The composition of the synthetic PUREX raffinate solution and the corresponding distribution ratios of the elements for the extraction without adding any complexants are given in Table 1 (see column 3).

These preliminary results show that the direct extraction of Am and Cm from a synthetic PUREX raffinate solution is possible with good extraction of the actinides and a high separation factor of Am/Eu of 68. However, some non-lanthanide fission products were coextracted with the actinides, namely Zr, Ag, Cd, Mo, Ni, Cu and Pd. Zirconium and molybdenum play a major role, because their concentration in the PUREX raffinate solution is very high (1071 and 678 mg/L, respectively). Even a relatively low distribution ratio in this case leads to a considerable loading of the organic phase, thereby reducing the free extractant concentration available for the actinide extraction. Palladium (168 mg/L) must also be considered due to the higher concentration, whereas Ag (12 mg/L), Cd (15 mg/L), Ni (40 mg/L) and Cu (19 mg/L) are contained in smaller amounts.

The coextraction of Zr, Mo and Pd is a problem that was often overcome by the use of complexing agents, namely oxalic acid and HEDTA (Figure 1) in experiments concerning the DIAMEX process [10].

Table 1 shows the results for the addition of oxalic acid. It shows that the distribution ratios of Zr and Mo are reduced significantly by oxalic acid, as expected. The extraction behaviour of Ag, Cd, Ni, Cu and Pd is not affected very much by oxalic acid and the results furthermore show that Y is extracted much better due to the lower overall loading of the organic phase.

Table 1 also shows the results for experiments with the addition of HEDTA alone, and those with a mixture of oxalic acid and HEDTA. The experiment with HEDTA alone shows that the Zr distribution ratio is approximately halved compared to the experiment without the addition of complexants, but there is no influence on the extraction of Pd. This was not expected and experiments with Pd single-element solution showed no influence of the HEDTA concentration on the extraction of Pd. The experiment with a mixture of oxalic acid and HEDTA shows that the addition of HEDTA is not advantageous compared to the experiment with oxalic acid alone and that the use of HEDTA can be omitted.

Table 1: The extraction of actinides and fission products from a simulated PUREX raffinate with CyMe₄BTBP/TODGA

Element	Concentration [mg/L or as shown]	Distribution ratio			
		Without complexant	C ₂ H ₂ O ₄	HEDTA	C ₂ H ₂ O ₄ + HEDTA
²⁴¹ Am	trace amounts	10.8	9.1	14.8	9.4
²⁴⁴ Cm	trace amounts	4.3	3.6	6.1	3.8
Y	90	0.04	0.85	0.15	0.73
La	239	<0.01	0.02	0.01	0.01
Ce	567	<0.01	0.03	0.01	0.02
Pr	223	0.01	0.04	0.02	0.03
Nd	718	0.02	0.06	0.04	0.05
Sm	149	0.07	0.14	0.10	0.13
Eu	34	0.16	0.24	0.14	0.25
¹⁵² Eu	trace amounts	0.06	0.22	0.10	0.20
Gd	51	0.08	0.17	0.08	0.15
Ni	40	30.0	18.4	32.1	37.3
Cu	19	4.88	15.7	19.1	5.60
Zr	1071	0.50	0.01	0.23	0.01
Mo	678	2.57	0.21	3.76	0.18
Pd	168	6.19	8.81	6.63	4.69
Ag	12	0.88	2.48	3.59	0.51
Cd	15	12.3	6.92	14.2	4.03
Cr	93	0.02	0.03	0.03	0.04
Sn	11	0.12	0.31	0.46	0.05
Sb	4.6	0.12	0.07	0.09	0.08
Rb	63	0.08	0.10	0.11	0.09
Ru	356	0.09	0.07	0.10	0.05
Rh	73	<0.01	<0.01	<0.01	<0.01
Te	165	0.03	<0.01	0.01	<0.01
Sr	177	<0.01	<0.01	0.01	<0.01
Ba	259	<0.01	<0.01	<0.01	<0.01
Cs	542	<0.01	<0.01	<0.01	<0.01
Al	2	n.d.	n.d.	n.d.	n.d.
Fe	1900	n.d.	n.d.	n.d.	n.d.
Se	10	n.d.	n.d.	n.d.	n.d.
Na	1600	n.d.	n.d.	n.d.	n.d.
HNO ₃	3.2 mol/L				

n.d.: not determined

Organic Phase: 0.015 mol/L CyMe₄BTBP, 0.005 mol/L TODGA in TPH/1-Octanol = 40/60 (not pre-equilibrated with nitric acid)

Aqueous Phase: 3.2 mol/L HNO₃, solution with HAR elements, complexants as given in the table + tracers (²⁴¹Am, ²⁴⁴Cm, ¹⁵²Eu), mixing time: 15 min.; T = 22 °C ± 1 °

B.2. Screening of different amino acids as masking agents for Pd

The aim of this work was to find a suitable masking agent for Pd. A number of amino acids and some amino acid derivatives were therefore tested for their influence on the Pd distribution ratio, together with ^{241}Am and ^{152}Eu . An overview of the tested complexants is shown in Figure 2. Amino acids were chosen because of their relatively complex coordination chemistry due to the presence of different donor atoms (O, N, S) in diverse structural constitutions thus allowing different chelate ring sizes, and because of their well solubility in aqueous solutions.

They were all tested in two concentrations, 0.1 and 0.2 mol/L. The results of the test with 0.1 mol/L amino acid are shown in Figure 3. The results of the test with 0.2 mol/L amino acid show a similar behaviour and therefore are not shown.

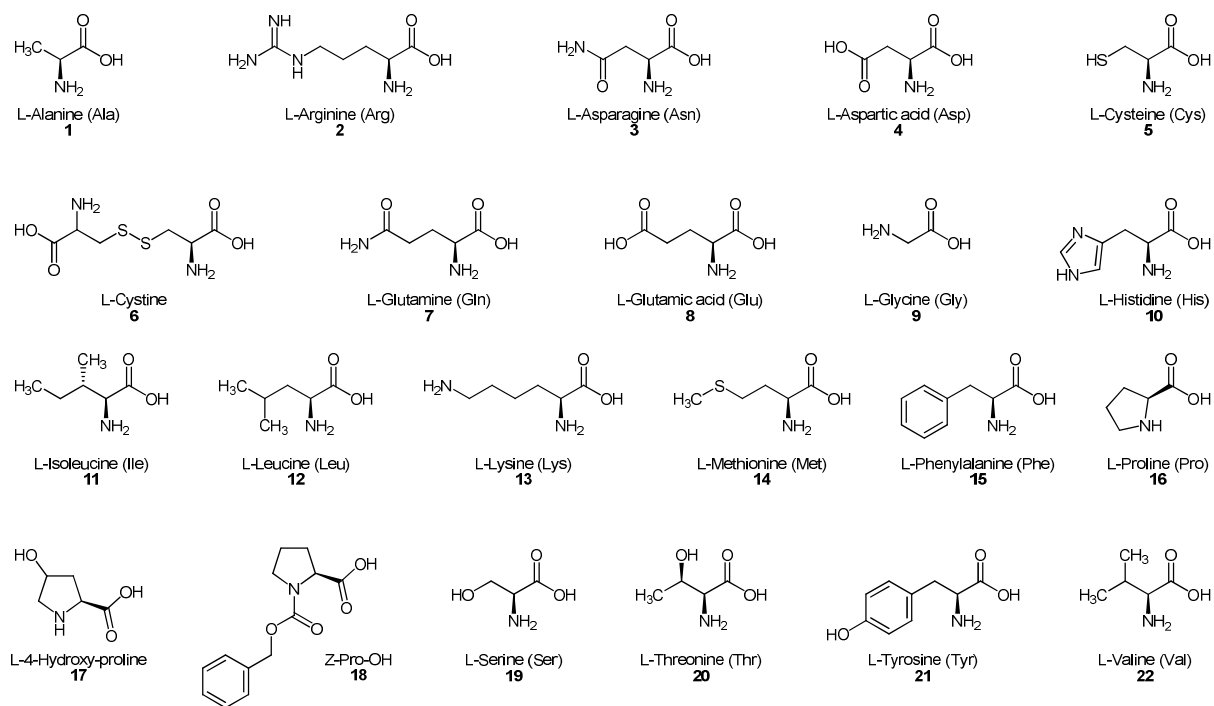
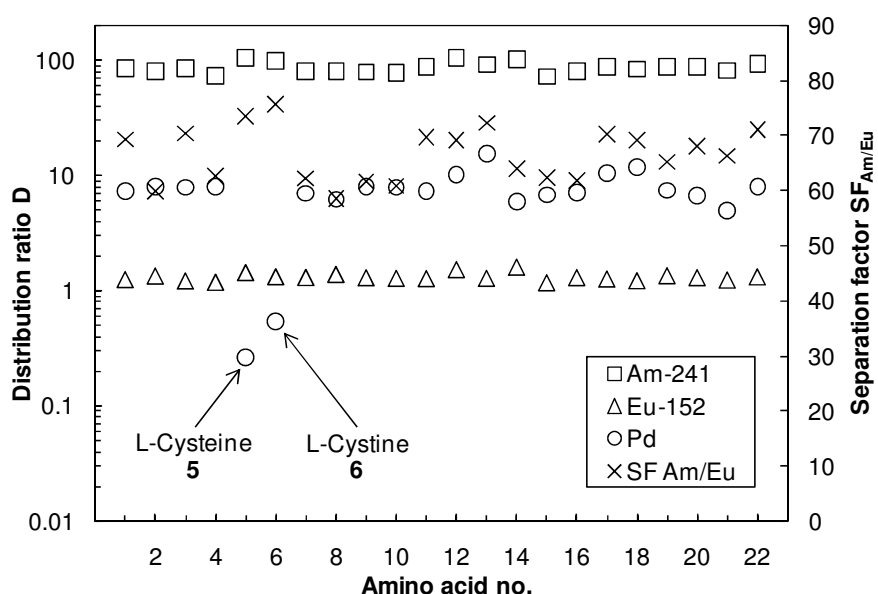


Figure 2: Overview of the tested amino acids and derivatives

Just two of the 22 tested amino acids showed a significant influence on the distribution ratio of Pd: L-Cysteine **5** and L-Cystine **6** (structures are shown in Figure 2). The Pd distribution ratios are reduced considerably and reach values below 1. Furthermore, they had no influence on the extraction of Am(III) and Eu(III). The distribution ratios of Am(III) and Eu(III), as well as the separation factor, remained unchanged. L-Cysteine was chosen for further studies, because of the structural similarity of the two molecules. L-Cystine is a dimer of two L-Cysteine molecules.



Organic Phase: 0.015 mol/L CyMe₄BTBP + 0.005 mol/L TODGA in TPH/1-Octanol = 40/60 (not pre-equilibrated with nitric acid)

Aqueous Phase: 3 mol/L HNO₃, 150 mg/L Pd (0.0015 mol/L), 0.1 mol/L of different amino acids + tracers (²⁴¹Am, ¹⁵²Eu, ²⁴⁴Cm, ²⁵²Cf), mixing time: 15 min.; T = 22 °C ± 1 °C

Figure 3: Amino acid screening

B.3. Experiments with simulated HAR solution

The 1-cycle SANEX process is intended to be used to separate the minor actinides from a PUREX-raffinate solution. To test the new complexant under process relevant conditions a simulated PUREX-raffinate solution had been prepared with the composition shown in Table 1.

In the following batch experiments, a process-like extraction series was used. The composition of the aqueous phases is shown in Table 2. In the first step, the extraction step, 4 mL of the simulated PUREX-raffinate solution were contacted for 15 min. with 4 mL of a freshly prepared organic phase with the same composition as in the previous experiments (0.015 mol/L CyMe₄BTBP + 0.005 mol/L TODGA in TPH/1-Octanol = 40/60). After phase separation, aliquots of each phase were taken for analysis and 3.0 mL of the remaining organic phase were contacted with 3.0 mL of a freshly prepared aqueous phase (Scrub I). Again after phase separation, aliquots of each phase were taken for analysis and 2.0 mL of the remaining organic phase were contacted with 2.0 mL of a freshly prepared aqueous phase (Scrub II). In the last step, called Strip, 1.0 mL of the separated organic phase was contacted with 1.0 mL of a freshly prepared aqueous phase.

Table 2: Composition of the aqueous phases used in the first experiment

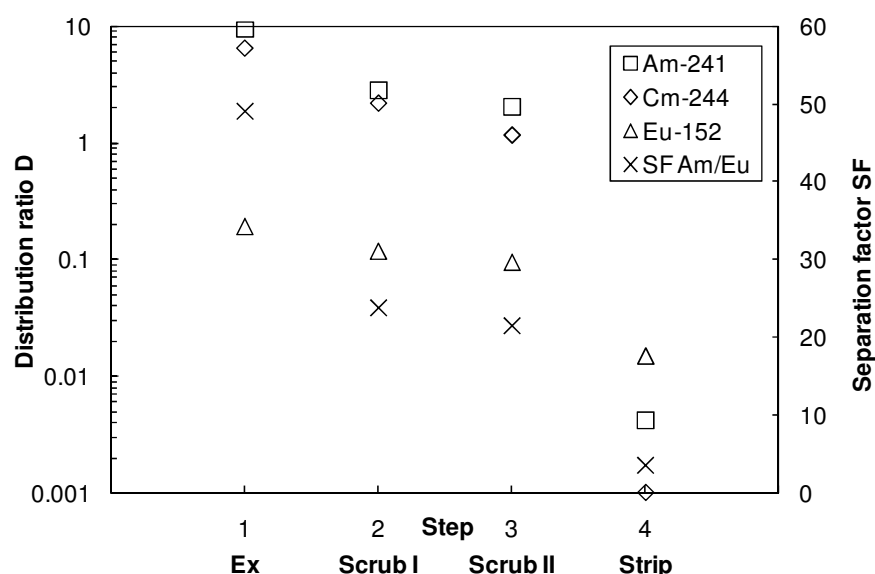
	Step 1: Ex	Step 2: Scrub I	Step 3: Scrub II	Step 4: Strip
c(HNO ₃) / mol/L	3.2	1.0	1.0	0.01
c(oxalic acid) / mol/L	0.3	0.2		
c(L-Cysteine) / mol/L	0.05	0.05		

During this first extraction experiment, L-Cysteine caused a voluminous precipitation, which is unwanted in a counter-current process. Therefore, the extraction series was altered as shown in Table 3. The experiment was conducted as described above.

Table 3: Composition of the aqueous phases used in the second experiment

	Step 1:	Step 2:	Step 3:	Step 4:
	Ex	Scrub I	Scrub II	Strip
c(HNO ₃) / mol/L	3.2	1.0	1.0	0.01
c(oxalic acid) / mol/L	0.3	0.2		
c(L-Cysteine) / mol/L			0.01	

During this experiment, no precipitate formed and no third phase formation was observed. Figure 4 shows that americium and curium are well extracted and that they stay in the organic phase during the washing steps. In the last step (Strip), they were back extracted into the aqueous phase. The trivalent lanthanides were not extracted well (with the highest distribution ratios for Europium) and therefore a high separation between the trivalent lanthanides and the actinides was achieved. The good back-extraction behaviour of the CyMe₄BTBP system is advantageous for the development of a reversible process with recycling of the organic phase which could be reused after a possible solvent treatment.

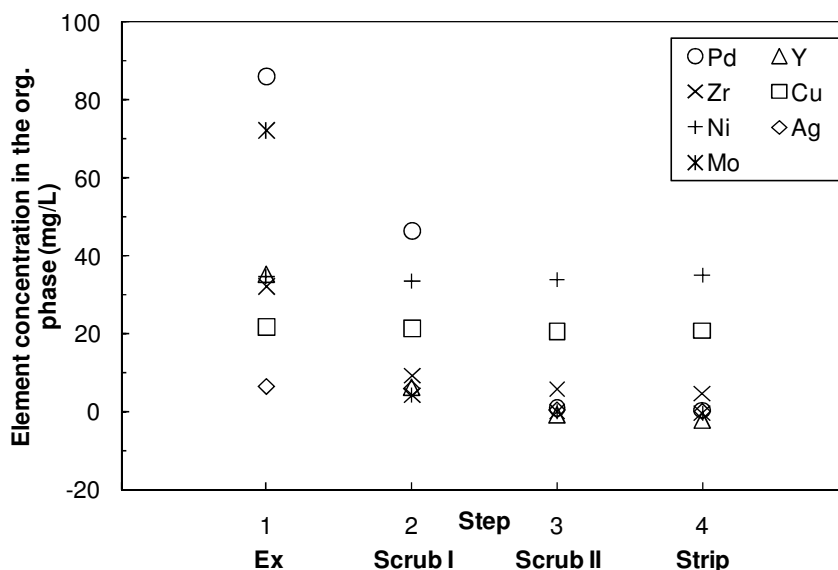


Organic Phase: Ex: 0.015 mol/L CyMe₄BTBP, 0.005 mol/L TODGA in TPH/1-Octanol = 40/60 (not pre-equilibrated with nitric acid; in the other steps, the loaded phase from the preceding step was used)

Aqueous Phase: as shown in Table 3.

Figure 4: Distribution ratios and separation factor of ²⁴¹Am, ²⁴⁴Cm and ¹⁵²Eu in a simulated four-step process using HAR simulate solution

Figure 5 shows the results from ICP-MS analysis for some selected inactive elements. In this figure, the concentration of the elements in the organic phase is depicted instead of the distribution ratio because the D-values of copper and nickel could not be determined due to their low concentration in the aqueous phase. The results show that copper and nickel are nearly completely extracted and that they stay in the organic phase during the whole experiment. They could possibly be scrubbed in an alkaline solvent treatment, but this topic was not investigated during this work. The extraction of zirconium and molybdenum was prevented by the use of oxalic acid and yttrium was scrubbed during the first two steps. Furthermore, the results show that palladium and silver were effectively scrubbed from the organic phase in the third step, the section with L-Cysteine addition.



Organic Phase: Ex: 0.015 mol/L CyMe₄BTBP, 0.005 mol/L TODGA in TPH/1-Octanol = 40/60 (not pre-equilibrated with nitric acid; in the other steps, the loaded phase from the preceding step was used)

Aqueous Phase: as shown in Table 3.

Figure 5: Organic phase concentration of some stable elements as determined by ICP-MS in a simulated four-step process using HAR simulate solution

C. CONCLUSION

In this work, it was shown that the direct and selective extraction of trivalent actinides from a synthetic PUREX raffinate solution as 1-cycle SANEX seems to be possible. Distribution ratios for trivalent actinides and the separation factor from the trivalent lanthanides were high. Nonetheless the reported system suffered from the coextraction of some of the non-lanthanide fission product elements. The extraction of the most abundant fission products Zr and Mo was suppressed satisfactorily by the use of oxalic acid. The extraction of the other elements was not influenced by the use of oxalic acid or HEDTA.

22 amino acids and derivatives were tested and L-Cysteine showed good complexation behaviour of Pd, without influencing the extraction of trivalent actinides. In a process-like extraction series, the practicability of the use of L-Cysteine as complexant for Pd and Ag was shown.

The high distribution ratios of Ni and Cu could be problematic for a continuous working industrial process. As the elements are not stripped back to the aqueous phase, they could possibly accumulate in the solvent and thereby disrupt the extraction process. More investigations have to be done to examine possible stripping conditions for the stripping of these elements. A possible solvent cleanup step has to be developed for the design of an industrial process.

Acknowledgements

Financial support for this research was provided by the European Commission (project ACSEPT – Contract No. FP7-CP-2007-211 267). M. R. S. Foreman, University of Reading, is greatly acknowledged for providing CyMe₄BTBP.

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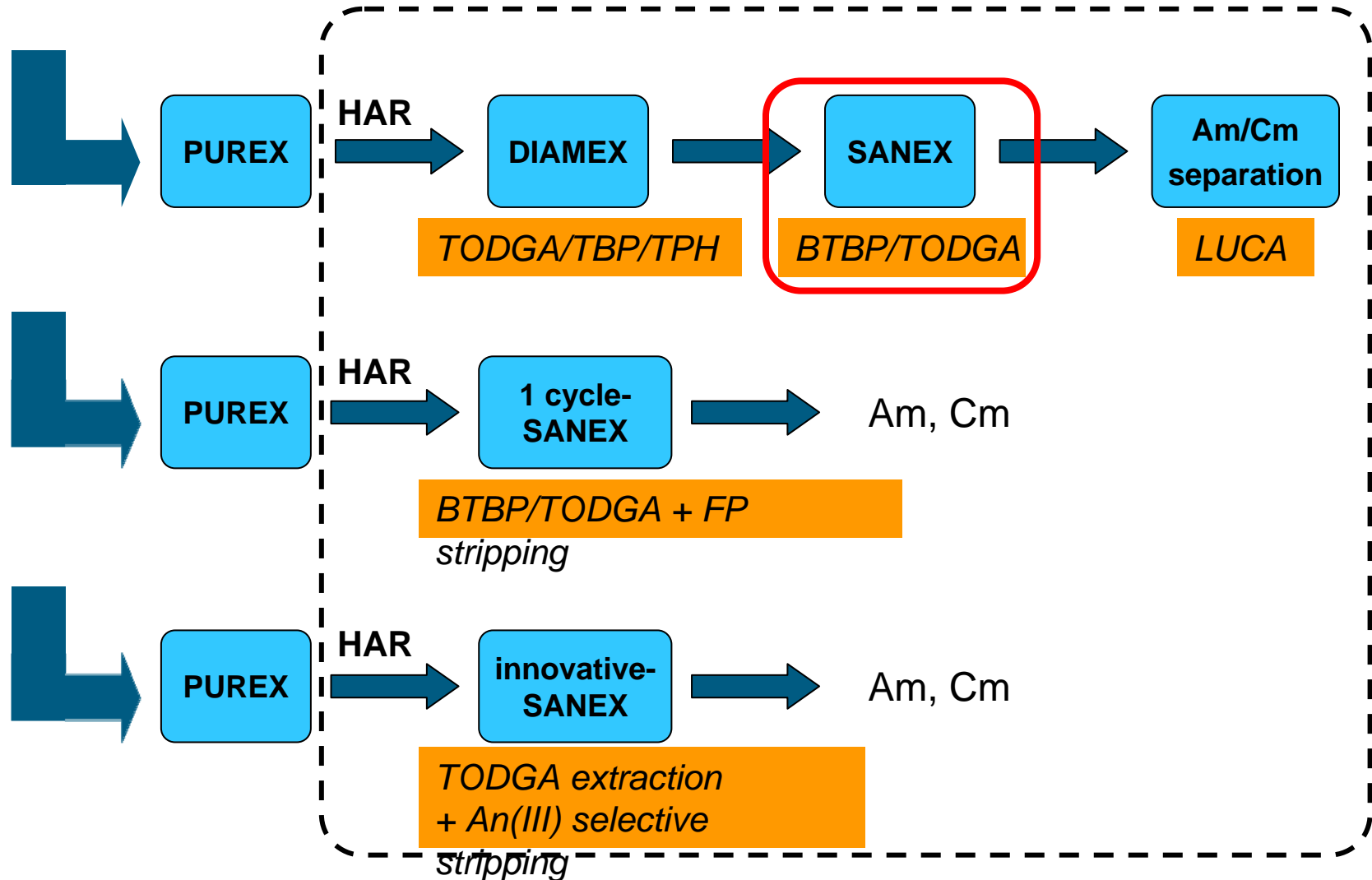
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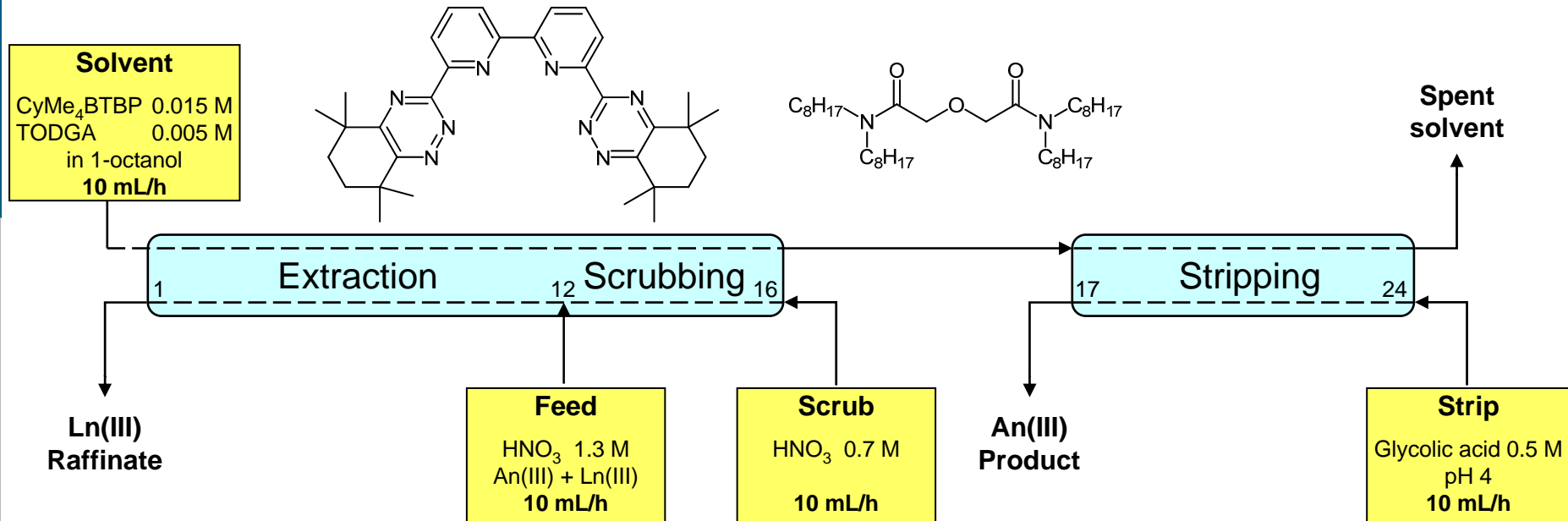
Overview of the processes

Dissolved spent fuel

Research areas at FZJ 

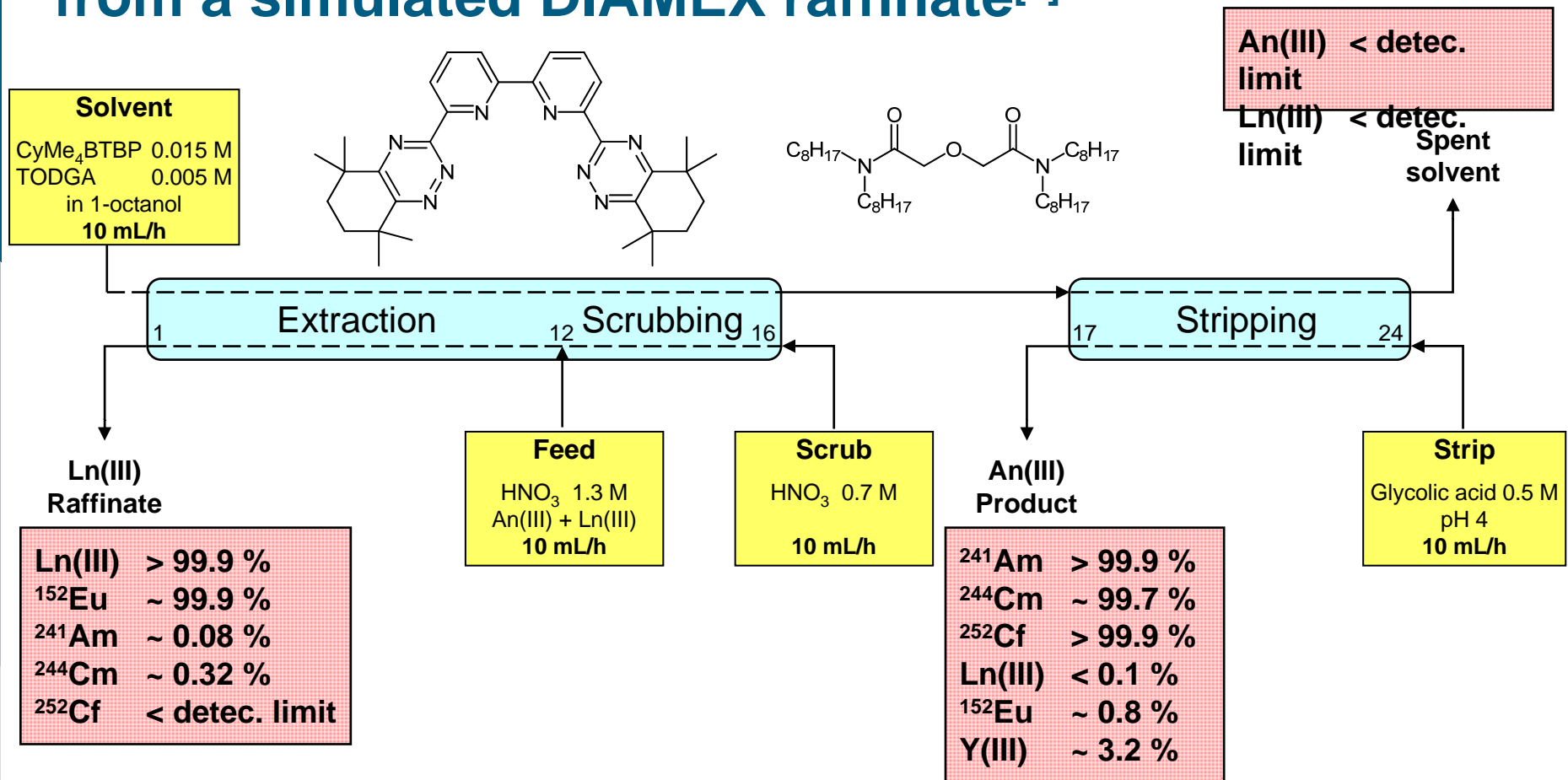


SANEX-process for An(III)/Ln(III) separation from a simulated DIAMEX raffinate^[1]



Element	Conc. [mg/L]	Nuclide	Activity [MBq/L]
Ce	383		
Eu	129	¹⁵² Eu	3.3
Gd	85	²⁴¹ Am	2.5
La	205	²⁴⁴ Cm	2.4
Nd	716	²⁵² Cf	1.6
Pr	192		
Sm	142		
Y	55		

SANEX-process for An(III)/Ln(III) separation from a simulated DIAMEX raffinate^[1]

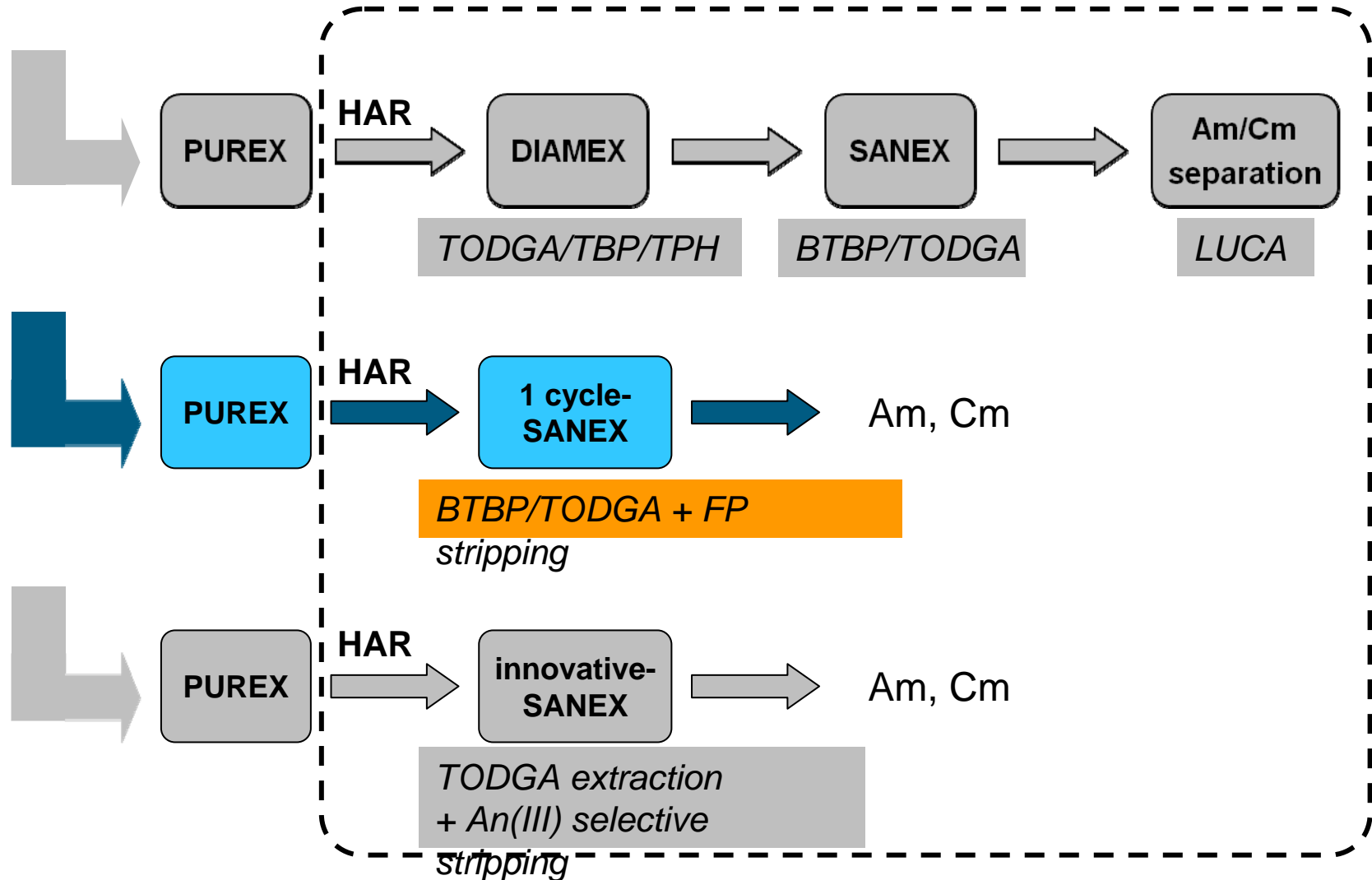


Is it possible to directly extract An(III) from a PUREX raffinate?

Overview of the processes

Dissolved spent fuel

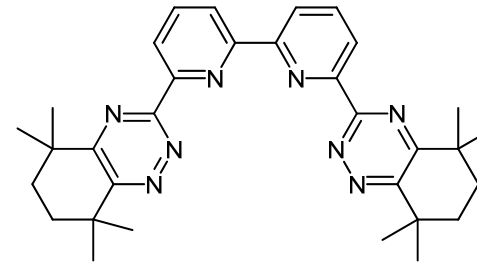
Research areas at FZJ 



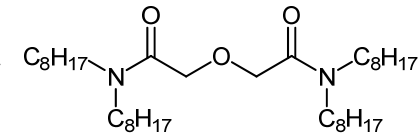
1-cycle SANEX – preliminary tests

- + High selectivity of the extractant (CyMe₄BTBP) for An(III) over Ln(III) and most FP
- + Hydrolytic- / radiolytic stability
- TODGA is used as phase transfer catalyst

- Coextraction of some fission products (Zr, Mo, Pd, Y, Cu, Ag, Cd, Ni)
- + Oxalic acid works for Zr, Mo
- HEDTA doesn't work for Pd



CyMe₄BTBP



TODGA

Task:

Find a selective masking agent for Pd.

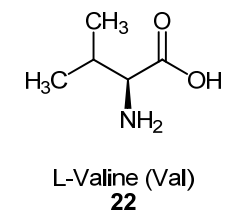
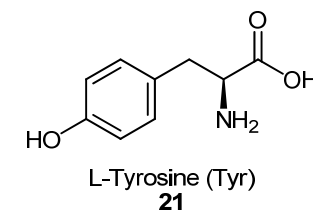
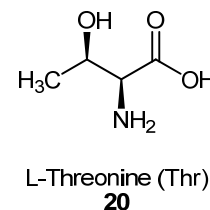
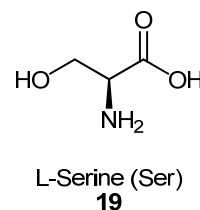
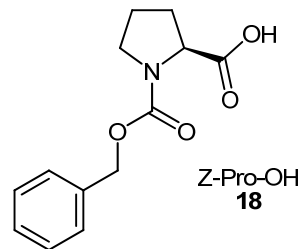
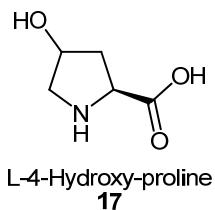
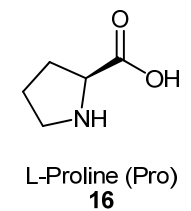
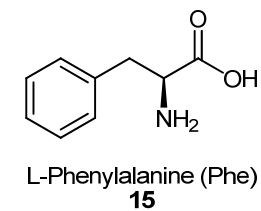
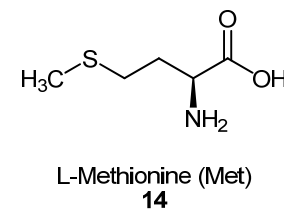
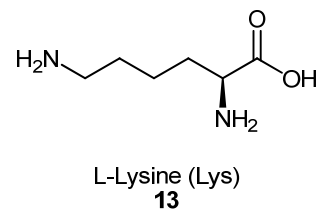
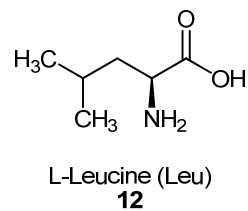
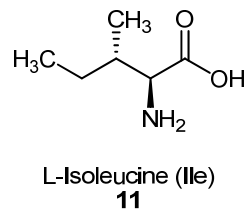
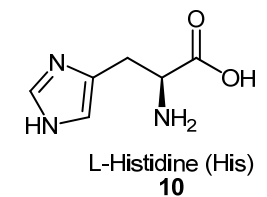
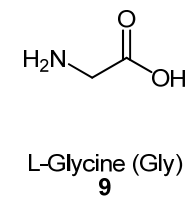
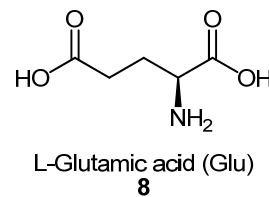
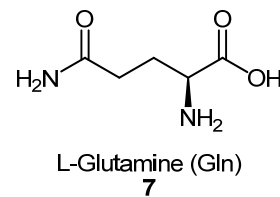
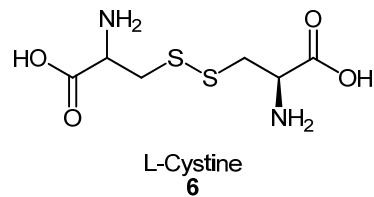
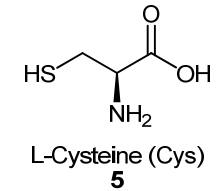
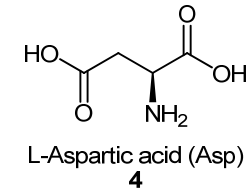
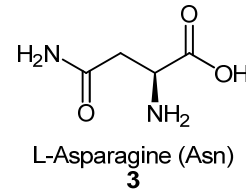
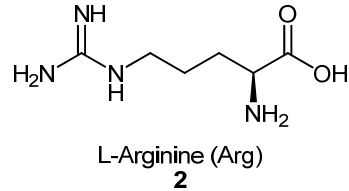
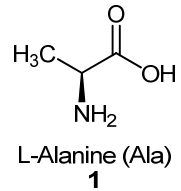
1 H																	2 He
3 Li	4 Be											5 B	6 C	7 N	8 O	9 F	10 Ne
11 Na	12 Mg											13 Al	14 Si	15 P	16 S	17 Cl	18 Ar
19 K	20 Ca	21 Sc	22 Ti	23 V	24 Cr	25 Mn	26 Fe	27 Co	28 Ni	29 Cu	30 Zn	31 Ga	32 Ge	33 As	34 Se	35 Br	36 Kr
37 Rb	38 Sr	39 Y	40 Zr	41 Nb	42 Mo	43 Tc	44 Ru	45 Rh	46 Pd	47 Ag	48 Cd	49 In	50 Sn	51 Sb	52 Te	53 I	54 Xe
55 Cs	56 Ba	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	
87 Fr	88 Ra	89 Ac	90 Th	91 Pa	92 U	93 Np	94 Pu	95 Am	96 Cm	97 Bk	98 Cf	99 Es	100 Fm	101 Md	102 No	103 Lr	

Lanthanides	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
Actinides	89 Ac	90 Th	91 Pa	92 U	93 Np	94 Pu	95 Am	96 Cm	97 Bk	98 Cf	99 Es	100 Fm	101 Md	102 No	103 Lr

Major Actinides Minor Actinides (MA)
 Fission products Activation products

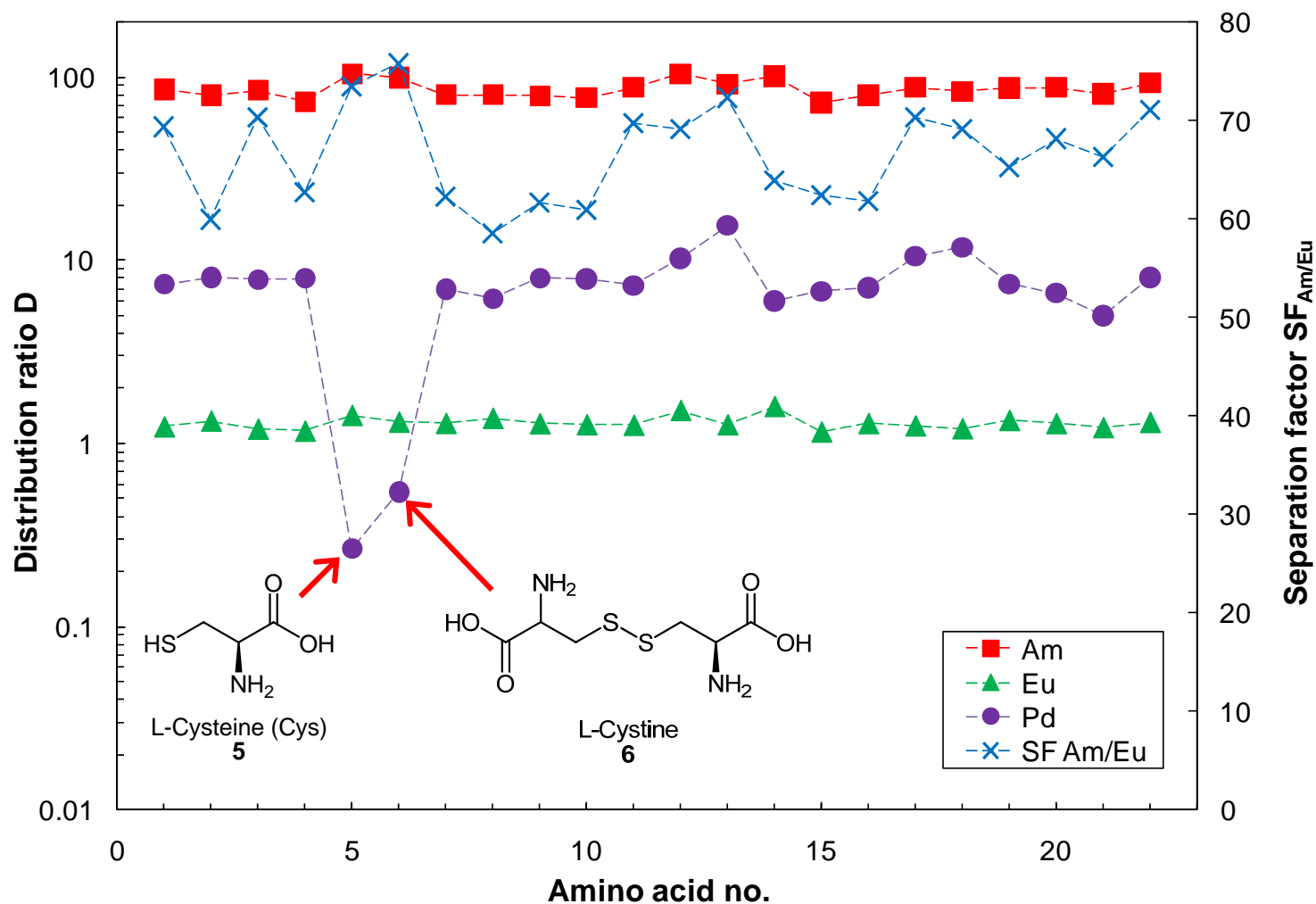
Screening of amino acids and derivatives

Overview of the tested substances



Screening of amino acids and derivatives

Results of the screening tests



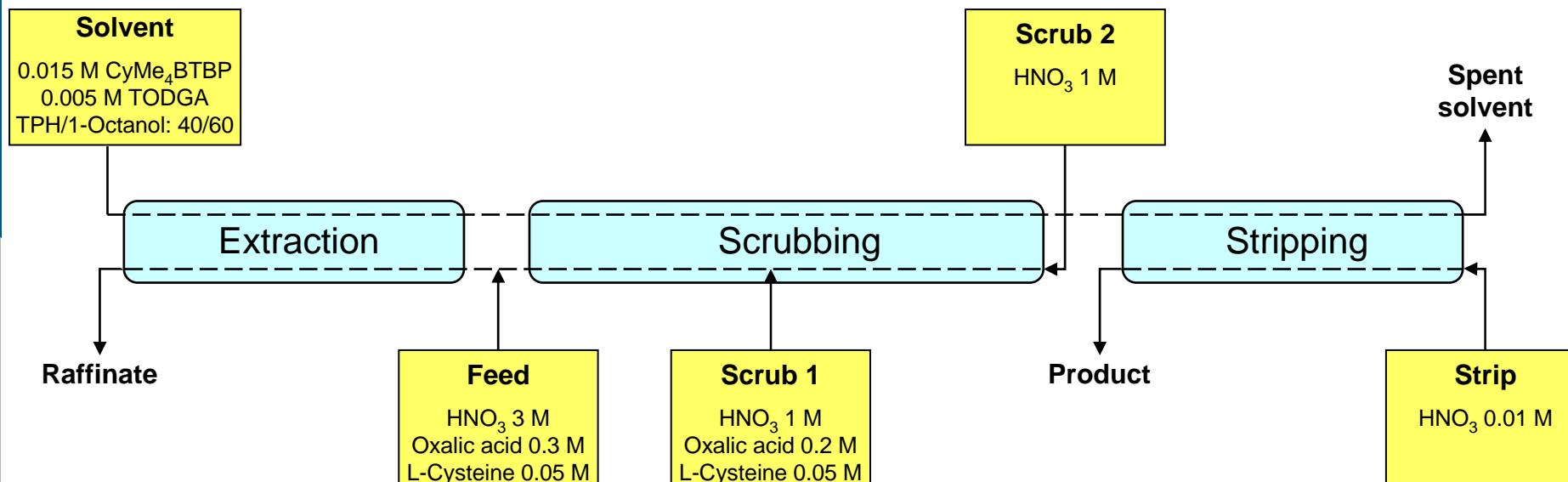
org. phase: 0.015 M CyMe₄BTBP, 0.005 M TODGA in TPH/1-Octanol (40/60)
 aq. phase: 3 M HNO₃, 150 mg/L Pd (0.0015 M), 0.1 M amino acid + radiotracer, 15 min., 22°C

01.04.2010

$$D = \frac{c_{\text{org}}(M)}{c_{\text{aq}}(M)}$$

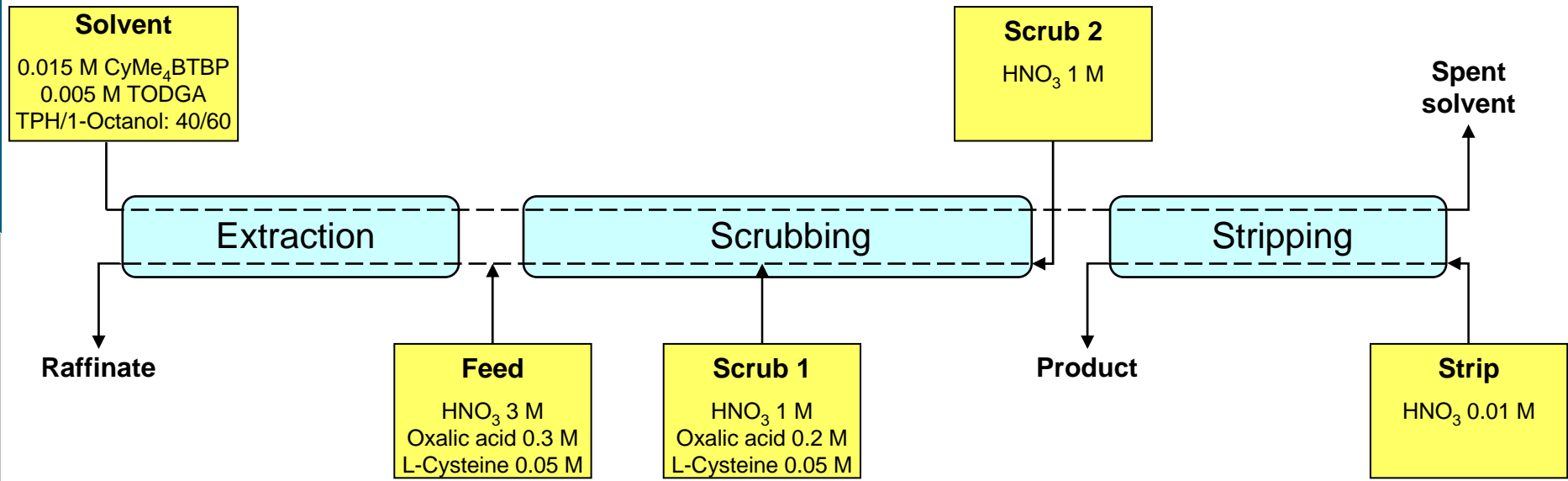
$$SF = \frac{D(M_1)}{D(M_2)}$$

Batch-tests with simulated HAR-solution

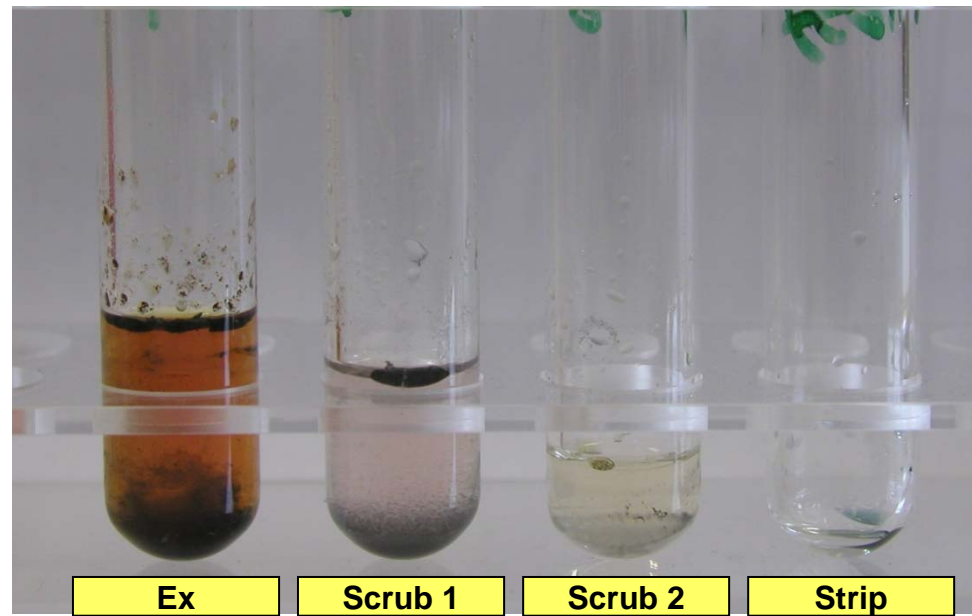


Element	Conc. [mg/L]	Element	Conc. [mg/L]	Element	Conc. [mg/L]
Fe	1900	Mo	678	Ni	40
Zr	1071	Cs	542	Cu	19
Na	1600	Ru	356	Cd	15
Nd	718	Ba	259	Ag	12
Ce	567	Sr	177	Sn	11
La	239	Pd	168	Se	10
Pr	223	Te	165	Sb	4.6
Sm	149	Cr	93	Al	2
Gd	51	Y	90	²⁴¹ Am	trace amounts
Eu	34	Rh	73	²⁴⁴ Cm	trace amounts
¹⁵² Eu	trace amounts	Rb	63		

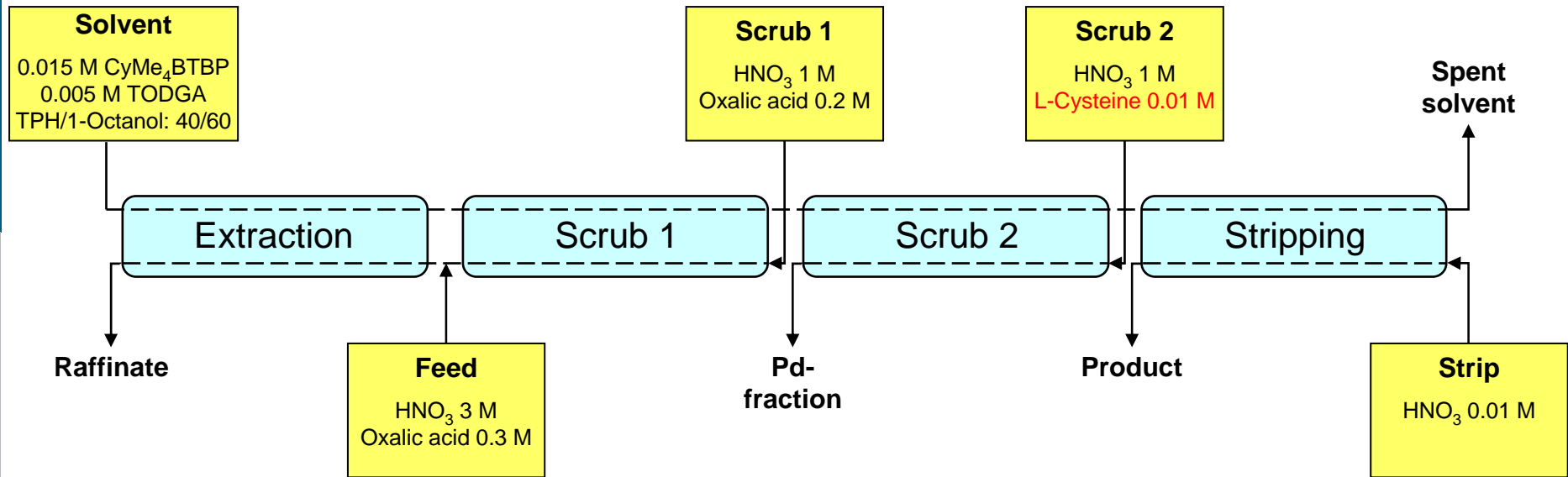
Batch-tests with simulated HAR-solution



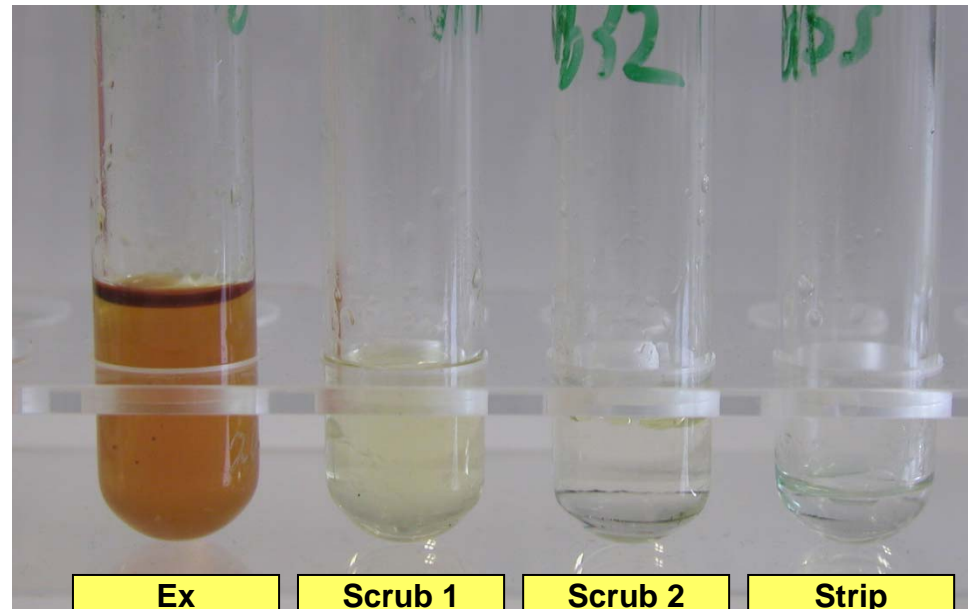
- Voluminous Precipitation during the extraction with L-Cysteine
- Precipitation should be avoided!



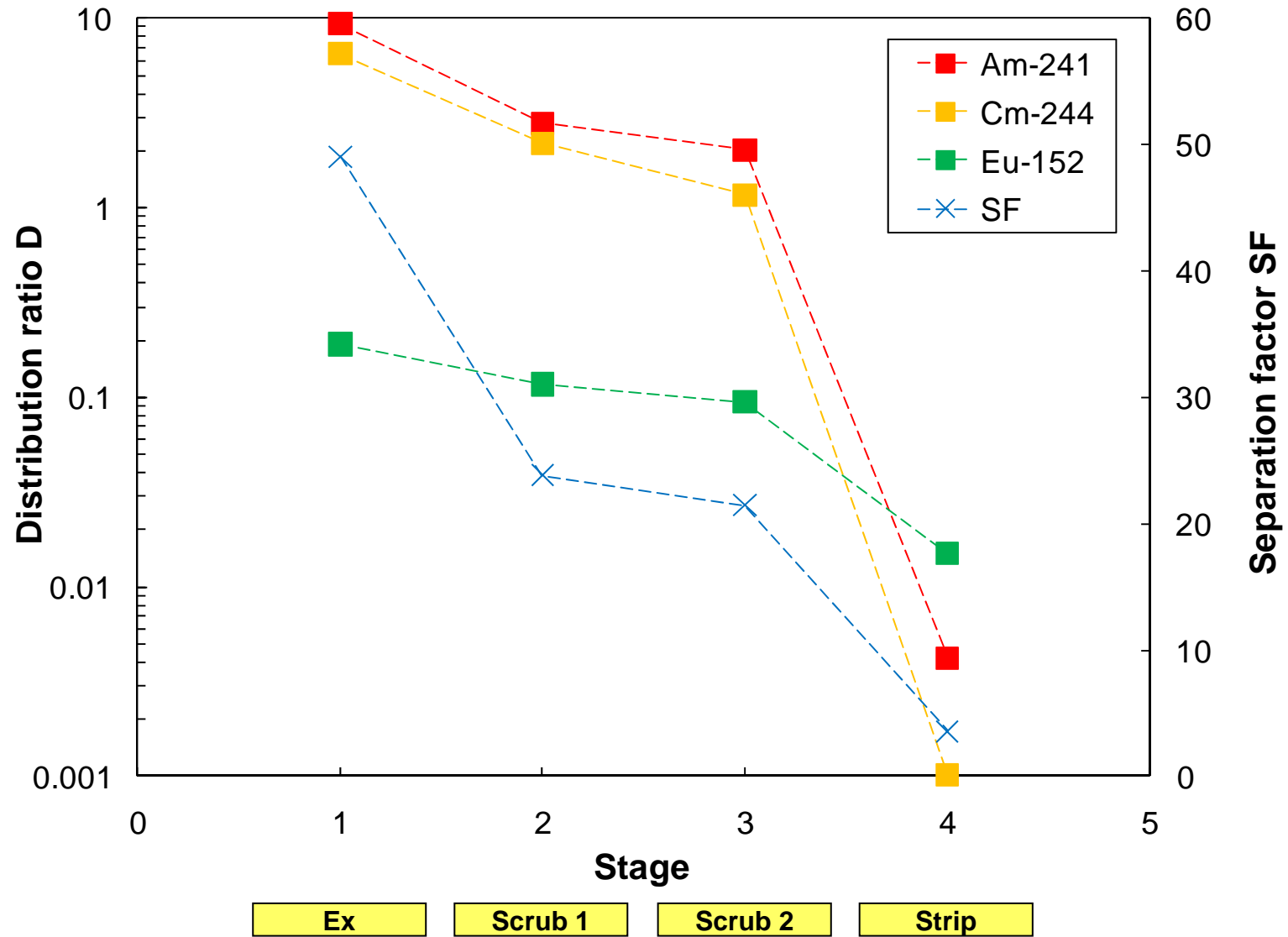
Batch-tests with simulated HAR-solution



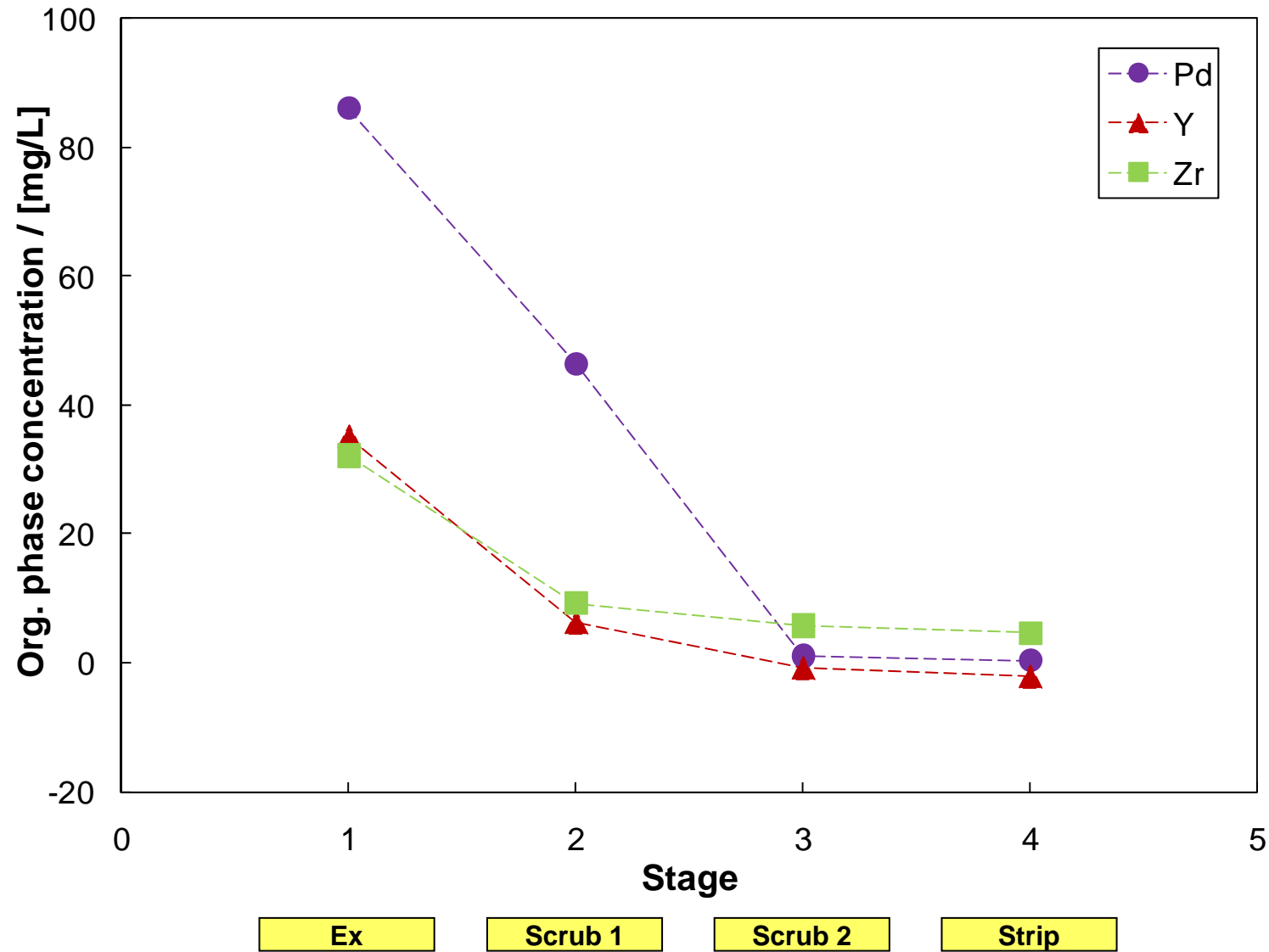
+ No Precipitation!



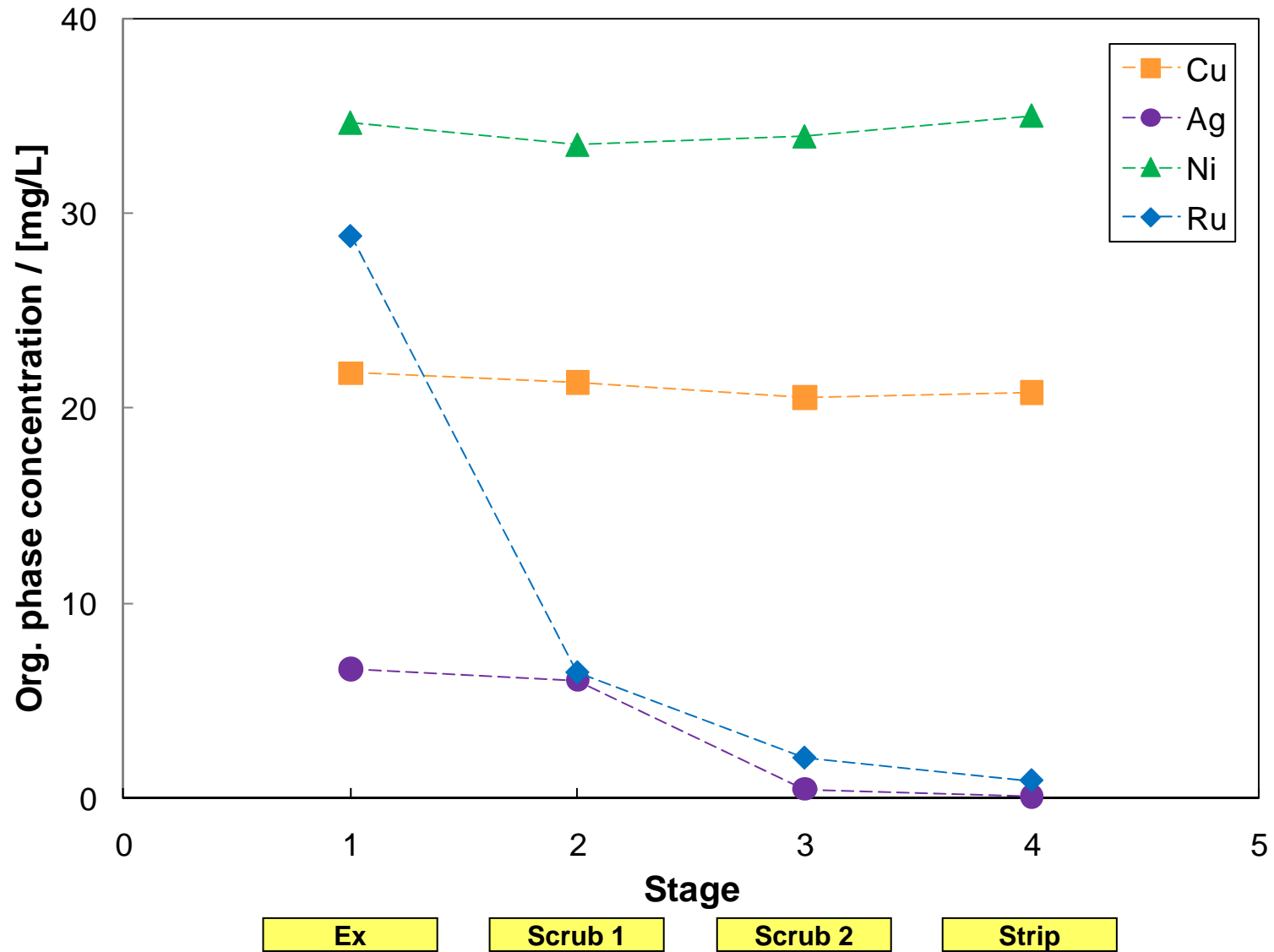
Batch-tests with simulated HAR-solution



Batch-tests with simulated HAR-solution



Batch-tests with simulated HAR-solution



Conclusions and outlook

- + It is possible to directly extract An(III) from a PUREX raffinate!
- + No Precipitation during the extraction
- + No 3rd phase formation
- + Am and Cm are selectively extracted and stripping is possible
- + Ag, Y and Ru are scrubbed in the scrubbing sections
- + Zr is complexed by oxalic acid
- + Pd is scrubbed in the third stage (Scrub 2) by L-Cysteine

- Cu and Ni are nearly completely extracted and remain in the org. phase
 - Further extractant optimization
 - Process development studies
 - Single centrifuge tests
 - Flow sheet design
 - Process test

Acknowledgements

Co-Workers:

- Giuseppe Modolo
- Christian Schreinemachers
- Paul Kluxen
- Michal Sypula
- Stefan Neumeier
- Henrik Daniels
- Carole Babelot

BTBP

- Reading University

Financial support



FP7-CP-2007-211 267

SPONSORED BY THE



Federal Ministry
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02NUK012E

Thank you for your
attention!