Horizon 2020
Programme

GENIORS

Research and Innovation Action (RIA)

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 755171.

Start date : 2017-06-01 Duration : 48 Months
http://geniors.eu/

GENIORS

WPASR 2

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GENIORS - Contract Number: 755171
GEN IV Integrated Oxide fuels recycling strategies Roger Garbil

Document title	WPASR 2
Author(s)	Mr. Stéphane BOURG, S. Bourg, CEA
Number of pages	71
Document type	Deliverable
Work Package	WP11
Document number	D11.2
Issued by	CEA
Date of completion	2018-11-23 16:02:55
Dissemination level	Public

Summary

WPASR2

Approval

Date	By
2018-11-23 16:03:06	Mr. Stéphane BOURG (CEA)
2018-11-23 16:03:12	Mr. Stéphane BOURG (CEA)
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CONTENT

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WP1

INTRODUCTION

WP1 aims at investigating the solution and extraction chemistry of key fission products. This research is important to improve the actinide/fission product separation and then to further optimize the separation processes. It includes extraction chemistry of problematic fission products (Task 1.1) and the optimization of scrubbing steps during co-extraction (Task 1.2). The total man power (pm) dedicated in WP1 to the first 2 semester is summarized in Table 1.

Table 1: Manpower devoted to WP1

Partner	WP1 effort	HYPAR 1 realised	HYPAR2 realised
CEA	7.9	0	0
ICHTJ	9.00	0	1.2
JUELICH	9.80	0	1
KIT	5.90	1	0.25
TWENTE	1.8		180h (1)
UREAD	5.8	0	0
Total	**40.2**	**1**	**3.45**

MAIN RESULTS

TASK 1.3

MANPOWER

3.45 pm was realized this semester by ICHTJ,JUELICH, KIT, and TWENTE. Together with the first semester (1pm by KIT) it is only 12 % of effort which is planned for the complete project (Task 1: 37.2 pm).

MAIN PROGRESSES

Extraction studies of technetium into TODGA system (ICHTJ)

Solvent extraction of technetium(VII) was studied in the two-phase system: TODGA in kerosene vs. aqueous HNO3 solutions. The TcO4– anion is well extracted from this acidic medium, probably as an ion pair with the protonated form of the extractant as the counter-ion. A hydrophilic DTPA ligand does not prevent the co-extraction of TcO_4^- with actinides to the organic phase under conditions corresponding to the second cycle of the GANEX process. Only cationic forms of technetium can be stripped to the aqueous phase.

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Extraction of Ru and PD into TODGA (FZJ)

First extraction tests were carried out with the TODGA system on Ru and Pd extraction. Organic samples will be analysed by ESI-MS. Results will be reported in HYPAR3

Extraction of Sr(II) into TODGA (KIT)

Distribution data for the extraction of Sr(II) into TODGA solvents (containing 1-octanol or TBP as modifier) were determined as a function of HNO3 and TODGA concentrations. An attempt at establishing an equilibrium model for Sr(II) extraction into TODGA was made. The dependence of lg DSr(II) vs. lg [TODGA] yielded a slope of 2.7.

ACTION PLAN

ICHTJ: Studies on the reduction of TcO_4^- by AHA in HNO3 solutions. Studies on the effect of technetium concentration in HNO3 solutions (under reducing conditions) on liquid-liquid distribution of Tc in the TODGA extraction systems.

JUELICH: Further extraction studies will be carried out and organic phases will be analysed by ESI-MS (collaboration with Uni. Hannover).

KIT: Determine Sr(II) distribution data in the TODGA + 5% octanol in TPH / HNO3 system at varied temperature and for Sr(II) loading conditions. Sr(II)/TODGA equilibrium modelling, in collaboration with NNL is planned.

TASK 1.2

No action for this semester

WP2

INTRODUCTION

The general objective of WP2 is to ensure a safe long-term performance of a chemical system submitted to radiation. To improve the resistance of the ligands as well as of the systems where they are involved will be the main goal. Solvent degradation may lead to many undesirable effects, for that, the identification of loses of efficiency, the behaviour troublesome degradation products or any mal operation situation due to degradation will be the key issues. Work is divided in four task: T21 Radiolysis & degradation products; T22 Destruction of organics; T23 Gas generation and T24 Radiolysis modelling.

MAIN RESULTS

TASK 2.1 RADIOLYSIS & DEGRADATION PRODUCTS (TASK LEADER: JULICH)

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MANPOWER

19.58 mp realized this semester (8.4 pm CEA, 1.5 pm CTU, 2.43 pm IIC, 1.5 pm CIEMAT, 1 pm Julich, 2 pm POLIMI, 2 UNIPR-synthesis, 0.75 KIT).

MAIN PROGRESSES

Studies of $CyMe_4$-BTBP and $CyMe_4$-BTPhen:

- *New fluorinated diluents BK-1:*

  During the second semester characterization of extraction systems with the fluorinated diluent BK-1 as a diluent has continued. Extraction of Am, Eu and Cm from HNO_3 solutions into solvents containing CyMe4 BTBP or CyMe4-BTPhen in BK-1 was tested by CTU. For the system $CyMe_4$-BTBP – BK-1 – HNO_3 was found the highest SF(Am/Cm) value at 1 mol/L HNO_3 where the Am(III) equilibrium was reached within 45 min, Eu(III) equilibrium in 30 minutes (but reached only app. 10 % of extraction percentage), and extraction of Cm(III) was in equilibrium approximately after one hour of shaking. However for the system $CyMe_4$-BTPhen – BK-1 – HNO_3 the highest value of SF(Am/Cm) was found at 4 mol/L HNO_3, with a fast kinetic for Am and Cm (around 15 min) , however Eu wasn't in the equilibrium even after 2 hours of shaking. It was also checked that there is not Am(III), Eu(III), and Cm(III) extraction by the neat diluent.

- *Stability studies of $CyMe_4$-BTBP:*

  IIC has continued with the analysis by HPLC and MS of CyMe4-BTBP samples irradiated at Chalmers facilities (uniform γ-dose 200 and 500 kGy) to study their composition and find effective conditions for separation of degradation compounds. A new series of CyMe4-BTBP samples have been selected to be irradiated and cover a broad range of approved solvents for better inspection of degradation pathways (FS-13, FS-13+TBP, octanol+TBP and octanol) and under different conditions (with and without contact with 4 mol/L HNO_3). In fact, the degree of degradation and composition of degradation products have been found to substantially depend on diluent. Positive influence of presence of nitric acid was observed, particularly for samples in Ganex diluent (FS-13+TBP) and octanol. Complete degradation was observed in octanol samples without contact with nitric acid. However, compared with previous series of analytical samples irradiated in similar systems, some differences in both, the residual concentration of CyMe4-BTBP and ratios of degradation could be observed. The larger-scale separations of main degradation products are being currently in progress.

Stability studies of diglycolamides:

- *Hydrophilic diglycolamides*

  The study on the radiolysis of hydrophilic diglycolamides performed by Julich in collaboration with Bruce Mincher (INL, USA) and Steve Mezyk (California State University Long Beach, USA) was finished; a paper was written and submitted to Solvent Extr. Ion Exch.

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- *Lipophilic diglycolamides (TODGA based systems):*

  In connection with experiments done for process development within WP5, CIEMAT has gone further in structural TODGA degradation study under different irradiation conditions (air, argon and air-sparging) and in contact with different aq. phases (0.5 mol/L HNO_3 and 0.018 mol/L SO_3PhBTP in 0.5 mol/L HNO_3). 9 typical TODGA DCs were identified in those systems irradiated using air or argon atmosphere. However, in those system irradiated using an air flow sparging it was detected different proportions of TODGA DCs and new signals corresponding to three possible unidentified TODGA DCs. Therefore, it seems air flow bubbled changes the dominant degradation pathway, and it should be taken into account in stability studies for process development.

  During this semester CEA has been focused on determining the differences between in-situ alpha (provided by the decay of ^{241}Am) and external gamma radiolysis (by a ^{60}Co source) of extractant TODGA and on determining how the degradation products formed affect metal complexation in the organic solvent. External gamma radiolysis (250 and 500 kGy) of GANEX/SANEX/DIAMEX solvent has been performed under several solvent conditions where numerous signals of possible degradation products have been identified by ESI-MS, although most of these are likely so low in intensity as to not affect the overall extraction behaviour. From the ESI-MS, it appears than many of the same products appear when the solution is subject to alpha irradiation (100 kGy until now). A couple of additional products associated with the breakage of the alkyl chains and conversion of the amide to an ester have been observed in the alpha irradiated solutions, but it more irradiation time is needed to conclusively determine if this is the case. Several Nd(III) species involving TODGA and degradation products have been identified in the mass spectra also by ESI-MS. The strongest signal for a Nd-DC complex is a $Nd(TODGA)_2((Octyl)_2NCOCH_2OCH_2COO^-)^{2+}$. To complement this experimental characterization, Fukui functions (used to determine the likely site for nucleophilic, electrophilic, and radical attack on a molecule) have been calculated for TODGA. As it can be expected, CH_2O protons of DGA are the most likely site for radical attack when metals are complexed. However, when water and nitric acid is extracted it would restrict access to these sites.

Stability studies of stripping or masking agents:

- *PyTriDiol :*

  A new batch of PyTriDiol was produced by UNIPR to feed the studies carried out by other partner. A sample has been sent to POLIMI in January 2018.

  During the second semester POLIMI has started the irradiation experiments on concentrated PyTri-Diol (PTD) solutions, in order to be able to identify the main radiolytic degradation products. The radiolytic degradation of irradiated PTD stripping solutions will be investigated firstly by ESI-MS, possibly coupled to HPLC-MS. Since these irradiations are expected to produce higher by-products concentration, attempts will be made in order to recover from the

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irradiated samples a sufficient amount of the hypothesized by-products for HPLC-MS and NMR characterization. Furthermore, hydrolytical stability of PTD aged in the dark at ambient temperature in about 5 mol/L HNO3 is under investigation by ESI-MS.

- *SO_3PhBTP and SO_3PhBTBP*

  Ciemat has continued with the setting up of qualitative and quantitative analysis of SO3PhBTP by HPLC-MS. Identification of DCs from a degraded sample is ongoing and it is expected to be able to quantified remaining concentrations of SO3PhBTP after irradiation in samples treated with H2SO4/H2O/MeOH.

  The stability of SO3PhBTBP vs. nitric acid and alpha radiolysis has been tested by INE. A solvent sample (0.2 mol/L TODGA + 5% octanol in Exxsol D80) loaded with 2.5 mmol/L 243Am(III) was contacted with an aqueous solution containing 20 mmol/L SO3PhBTBP in 0.36 mol/L HNO3, resulting in an equilibrium HNO3 concentration of 0.5 mol/L and an aqueous phase Am(III) concentration of 2 mmol/L. After 10 kGy (21 day) distribution ratio was kept and no precipitation was observed, however after 28 kGy (60 days) Am(III) distribution ratio increased 50% and a pinkish precipitate was observed.

- *CDTA :*

  During this semester CIEMAT has analyzed the behavior of CDTA degraded solvents as part of Euro-GANEX process. For that, irradiated solutions of CDTA (0.055 mol/L in 4 mol/L HNO3) in presence and absence of simulated HAR solution have been contacted with the corresponding organic solvents (0.2 ml/L TODGA + 0.5 mol/L DMDOHEMA in OK). Samples irradiated in presence of HAR solution gave placed to a white precipitate forms mainly by Zr and Pd, metals that CDTA complexes. However, it has been also detected by ICP-MS in those samples where HAR solution was added after irradiation. Therefore, DCs of CDTA could form insolubilities of Zr and Pd in the aqueous phase after the lowest dose tested, 200 kGy. Ln extraction is not affected in any of cases studied. New irradiation experiments taking into account a real concentration of HAR solution and real estimated doses for a CDTA solvent are needed.

DIFFICULTIES

No major difficulties have been reported. Although there was some delays due to:

- UNIPR has reported difficulties on the time scale for the hiring a PhD student working on the project.
- No degradation products/adducts of $CyMe_4$-BTBP and $CyMe_4$-BTPhen synthesized yet.
- IIC: Some additional work is still necessary to develop efficient recovery of degradation products from HPLC buffer components used for separation.
- CIEMAT reported delay due to no availability of ^{60}Co-sources at Náyade facility for all irradiation experiments.

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ACTION PLAN

POLIMI: On going activities to confirm the hypothesized degradation products.

CTU: Testing of extraction systems based on fluorinated carbonates will continue. If available, testing of extraction properties of radiolysis products synthesized at University of Reading will start.

IIC: on going activities to develop efficient recovery of degradation products.

CIEMAT: New irradiation experiments of CDTA taking into account a real concentration of HAR solution and estimated doses are needed.

TASK 2.2 DESTRUCTION OF ORGANIC (TASK LEADER: CEA)

MANPOWER

2.5 mp realized this semester (1.5 pm CEA, 1 pm UNIPR).

MAIN PROGRESSES

TODGA:

During this semester CEA has started the studies addressed to deliberate destruction of TODGA by at high acidity and high temperature. Results will be reported in the following HYPARS.

PTD:

A literature search allowed UNIPR to identify Differential Scanning Calorimetry (DSC) and Accelerating Rate Calorimetry (ARC) as interesting and informative techniques to assess preliminary hazard level of organic azides (synthesis intermediate) and the extractant of interest PTD. Shock sensitive or demonstrate explosive propagating properties could be predicted.

DIFFICULTIES

UNIPR has reported difficulties on the time scale for the hiring a PhD student working on the project.

ACTION PLAN

CEA: Start of stability studies of TODGA at high acidity and high temperature.

UNIPR: DSC studies to carry out the stability studies of PTD ligand alone, its complexes and intermediates of the synthesis.

TASK 2.3 GAS GENERATION (TASK LEADER: POLIMI)

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MANPOWER

4.2 mp realized this semester (2.7 NNL, 1.5 POLIMI).

MAIN PROGRESSES

Gas generation of TODGA based systems:

During the second semester NNL has undertaken irradiations of relevant solvents for the i-SANEX and GANEX processes (0.2 M TODGA, 5 v/v.% 1-octanol in odourless kerosene; 0.2 M TODGA, 0.5 M DMDOHEMA in hydrogenated tetrapropylene) with He^{2+} ion implantation using the UoM-DCF (University of Manchester Dalton Cumbria Facility) tandem accelerator. For both solvent systems G_{H2} values of ~3.5 molecules/100eV across the entire dose range were obtained despite the variation in solutes and diluent. 1-octanol is not expected to have a large effect on G_{H2} due to its similar polarizability to n-alkanes but a marked effect would be expected for DMDOHEMA with the relatively large concentration and therefore number of additional amides groups. Further analysis of TODGA degradation is ongoing. These experiments are also linked with studies in WP3.

Gas generation of PTD based systems:

POLIMI has planned and started a series of irradiation experiments on the PTD-TODGA extracting system, in order to investigate the generation of gaseous products from each phase during exposure to gamma radiation. PTD solutions (with and without pre-equilibration) and biphasic system (org. + aq.) as well as systems degassed or in presence of oxygen are being explored in these experiments. Radiation treatments are in progress.

DIFFICULTIES

NNL has reported delays waiting for time on pelletron at DCF, and insufficient dose to degrade sufficient solvent for solvent clean up and flowsheet test.

ACTION PLAN

NNL: Consider further data needs. Draft paper on hydrogen generation.
POLIMI: When ready the irradiated aqueous and organic phases will be analyzed by means of micro GC-MS under optimized conditions.

TASK 2.4 RADIOLYSIS MODELLING (TASK LEADER: CTU)

MANPOWER

1.3 mp realized this semester (CTU).

MAIN PROGRESSES

The theoretical study of CyMe4-BTBP has been continued in the second semester in more detail. All studies performed until now (HYPAR-1 and HYPAR-2) point out to the prevailing

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degradation product found in solution was 1-octanol adduct on *meta* position of pyridine ring of CyMe4-BTBP as the most preferred one.

DIFFICULTIES

CTU: No degradation products/adducts of BTBP/BTPhen synthesized, yet.

ACTION PLAN

As a new topic, the simulation for comparison of TMDGA, TEDGA, Me-TEDGA, and Me_2-TEDGA were started by CTU and it is planned to proceed in this topic during the next semester.

WP3

INTRODUCTION

The aim of this work-package is to improve the understanding and optimization of the reference chemical systems for advanced solvent extraction separation processes: grouped separation of the actinides (EURO-GANEX), separation of the minor actinides (i-SANEX), or separation of americium only (EURO-EXAM). It includes (i) the understanding of extraction chemistry to support concept process flowsheets (Tasks 3.1 & 3.3), (ii) the acquisition of extraction data to support the conception of process flowsheets (Task 3.2), and (iii) the identification of process options for clean-up of solvents to allow recycle on plant (Task 3.4).

MAIN RESULTS

TASK 3.1 OPTIMISATION OF SYSTEMS (TASK LEADER: POLIMI)

MANPOWER

11.6 pm realized this semester (CEA, ICHTJ, POLIMI, UNIPR).

19.1 pm realized since the beginning of the project, corresponding to 24.5 % of the 78 pm planned for the complete project.

MAIN PROGRESSES

Hydrophilic complexation:

1) BTP based complexing agents

- Study of Am(III) and Eu(III) stripping from the organic phase containing a T-DGA ligand as the extractant to an acidic aqueous phase containing a hydrophilic SO_3-Ph-BTP^{4-}

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ligand. The obtained unreal non-integer slopes of a basic dependence used in the simple model of extraction were interpreted in terms of a new model which assumed the extraction of not only $[M(T\text{-}DGA)]^{3+}$ but also of the other – heteroleptic $[M(T\text{-}DGA)(SO_3\text{-}Ph\text{-}BTP)]^{-}$ complex. That was explained in terms of a hypothesis on steric constraints that make the T-DGA molecule a hexadentate, not nonadentate ligand.

2) Pi-Tri-Diol (PTD)

- As a continuation of the studies dedicated to fully understand the extracting behavior of PTD based systems, further experiments were performed at POLIMI on the metal to ligand complex formation by means of ESI-MS, in order to gain further information about such aspect.
- Within a collaboration with CEA, POLIMI is performing the study of Pu(IV) complexation with PTD by UV-Vis spectroscopy in the process conditions, that is in HNO_3 = 0.44 M. The results will be compared with previously obtained data for Am(III) by UV-Vis analyses and Cm(III) and Eu(III) by TRLFS analyses, thus confirming TRU selectivity of PyTri-Diol at process conditions
- A new batch of PyTriDiol was produced to feed the studies carried out by partner. A sample was sent to POLIMI.

Organic phase modifier:

- New modifiers of the TODGA-containing organic phase were quested, which would exert stronger synergistic enhancement of the extraction of the early lanthanides than of Am^{3+}, in order to improve the separation of Am^{3+} ions from the troublesome early lanthanides by selective Am(III) stripping to HNO3 solutions by a CHON stripping agent. The results of the experiments allowed us to determine physicochemical properties of the optimum modifiers and to select some suitable compounds for further studies.

DIFFICULTIES

- Some delay in the development of the studies on the Pu complexation with PTD due to technical problems at CEA laboratories
- Difficulties on the time scale for the hiring a PhD student working at UNIPR on the project.

TASK 3.2 DISTRIBUTION DATA AND CHEMICAL MODELLING (TASK LEADER: KIT)

MANPOWER

0.25 pm realized (KIT)

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4.25 pm realized since the beginning of the project, corresponding to 31.2 % of the 13.6 pm planned for the complete project.

MAIN PROGRESSES

- Equilibrium modelling based on TRLFS results was done for the extraction of Am(III) into DMDOHEMA.

TASK 3.3 PHYSICO-CHEMICAL & LOADING (TASK LEADER: CIEMAT)

MANPOWER

7 pm realized (CEA, CIEMAT, KIT, POLIMI)

10.5 pm realized since the beginning of the project, corresponding to30.9 % of the 34 pm planned for the complete project.

MAIN PROGRESSES

The loading of TODGA organic phases studies have been continued:

- Solvent extraction experiments have been started to prepare different organic phases (TODGA with or without octanol, and TODGA/DMDOHEMA mixture) with various Nd(III) concentrations. Some third phases have also been prepared. The characterisation of these organic phases and the corresponding molecular dynamics simulations are in progress.
- Am(III) and Ln(III) loading experiments were performed with the AmSel system (SO_3-Ph-BTP/TODGA)
- Influence of the most detrimental TODGA DCs (diglycolamic acid, **V** (DODGAA); Oct2NH, **VII** (DOA)) on An (0.35 mol/L HNO3) and Ln stripping step (0.5 mol/L citric acid) during an *i*-SANEX

Loading of aqueous phases containing Py-Tri-Diol as a selective complexing agent have been started:

- In view of the application in the *i*-SANEX process and, possibly, in the EURO-GANEX ones, the study of the loading capability of PTD-based stripping solutions is ongoing. In particular, experiments with macro-concentrations of Am (0.01M) and Pu (0.1M), in presence of 0.02M lanthanides, are in progress. This activity is developed within a collaboration between POLIME and CEA in Marcoule, which is hosting a POLIMI Ph.D. student for 2 months, thanks to a special Grant from the GENIORS project.

TASK 3.4 SOLVENT CLEAN-UP & RECYCLE (TASK LEADER: NNL)

MANPOWER

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1.6 pm realized (NNL, POLIMI)

2.0 pm realized since the beginning of the project, corresponding to 9.3 % of the 21.5 pm planned for the complete project.

MAIN PROGRESSES

- Radiation experiments performed by NNL at Dalton Cumbria Facility using pelletron to simulate alpha degradation of i-SANEX solvent. Hydrogen generation measured and TODGA performance post-irradiation characterised. (Linked study with NNL studies in WP2).
- Within Task 3.4 POLIMI is going on both in evaluating the technical feasibility of the cleanup and recycling of used PTD solutions and in considering the related advantages and positive effects on the whole separative process. To this purpose, several PTD solutions were prepared: some aliquots are ageing under controlled conditions and other ones are under gamma-irradiation. When ready, the PTD solutions will be used in further experiments aiming at recovering purified PTD solutions ready to be reused.

DIFFICULTIES

- TODGA experiments: insufficient dose to degrade sufficient solvent for solvent clean up experiments and a flowsheet test. May necessitate new irradiation or spiking with artificially generated degradation products

WP4

The main objectives of the WP solid/liquid interface chemistry is to better understand the phenomena occurring at the solid/liquid interfaces during spent nuclear fuel reprocessing in order to support potential processes. It is divided in two main topics. The first is devoted to dissolution step (Task 4.1). It will be examined not only considering direct interactions between the chemical species coming from the solid and the solution, but also through the development of catalytic reactions at the interface. The second topic is focused on the conversion of actinides by precipitation of original precursors coming from new chemical processes based on wet chemistry routes (Task 4.2). The total man power (pm) dedicated to WP4 (Tasks 4.1 and 4.2) are summarized in the table below

Partner	Total planned	WPASR 1	WPASR 2
CNRS/ICSM	38.5	1.6	2.2
CEA	15.4	0.0	3.0
SCK.CEN	20.0	2.5	2.5
UNIMAN	11.0	0.0	0.0
Total	**84.9**	**4.1**	**7.7**

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MAIN RESULTS

TASK 4.1 STUDY OF THE SOLID/LIQUID INTERFACES DURING DISSOLUTION [1-48]

Task Leader : CNRS

MANPOWER

0.4 pm (CNRS) – 3 pm (CEA)

MAIN PROGRESSES

Multiparametric study of the dissolution of $(U,Ce)O_2$ and $(U,Ln)O_{2-x}$ solid solutions – CNRS/ICSM

In order to develop the multi-parametric dissolution tests, the preparation then characterization of a large defined panel of uranium-lanthanide solid solutions has begun in month 6. The first set of data regarding dissolution tests on $(U,Ce)O_2$ are expected by the end of august.

Innovative dissolution routes for highly plutonium doped $(U,Pu)O_2$ and $(U,Pu,MA)O_2$ samples – CEA

The study of the solid/liquid interfaces during dissolution has begun through the synthesis of CeO_2 and $Ce_{0.8}Gd_{0.2}O_{1.9}$ samples using oxalic route and calcined at different temperatures to obtain samples exhibiting different levels of crystal defects and morphological parameters. A Netzsch micro-series milling device has been bought to study the milling/dissolution couplings.

ACTION PLAN

CNRS/ICSM

The first dissolution experiments on $(U,Ce)O_2$ solid solutions have been launched in May 2018. They will be extended during the next semester and the first set of data are expected by the end of the year. Complementary experiments will be performed on $(U,Ln)O_{2-x}$ solid solutions.

CEA

A PhD work started in Nov. 2017 to study the innovative dissolution routes for highly plutonium doped $(U,Pu)O_2$ and $(U,Pu,MA)O_2$ samples. During the next semester, the main aspects of the work will be the continuation of the characterization of the synthesized samples, the milling of the samples using different energy to modify the defect generation, the observation of the samples by TEM to determine the crystal defects, the development of new syntheses using chemistry routes and finally the beginning of dissolution experiments.

TASK 4.2 STUDY OF THE SOLID/LIQUID INTERFACE DURING CONVERSION [1-48]

Task Leader : SCK.CEN

MANPOWER

1.8 pm (CNRS/ICSM) + 2.5 pm (SCK.CEN)

MAIN PROGRESSES

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Studies of non-powder routes for the synthesis of MOX fuels materials, potentially bearing minor actinides and blanket fuel materials – SCK.CEN

The thermal behavior of the U/Ce particles was studied via TG-DSC and the gelled spheres were calcined then sintered. For compositions containing less than 20% of cerium, the spherical shape was maintained during the thermal treatment. The products were analyzed using powder XRD and a fluorite type structure was found. A linearly decreasing lattice parameter with increasing Ce content up to 20% was observed.

Hydrothermal precipitation of uranium oxides for simplified fuel fabrication – CNRS/ICSM

The direct precipitation of uranium oxides has been undertaken under hydrothermal conditions through the *in situ* conversion of uranium oxalate. Below 180°C, the usual structure of $U(C_2O_4)_2.2H_2O$ was observed whereas $UO_{2+x}.nH_2O$ was prepared above this temperature. SEM observations of the resulting samples further revealed the loss of the classical square platelet shape of the oxalate, while the evaluation of the residual carbon content led to values significantly lower than that usually obtained through thermal conversion. Additionally, variation of the pH allowed to optimize the uranium precipitation yield.

Innovative precursors for morphology-controlled UO_2 and $(U,Ln)O_2$ oxides – CNRS/ICSM

$UO_2.nH_2O$ microspheres were obtained through hydrolysis of uranium(IV) in presence of aspartic acid under hydrothermal conditions (160°C). A multi-parametric study involving time, temperature, concentration of the reactants was then undertaken. It showed that a 50% excess in aspartic acid led to monodisperse powders. The addition of mechanical stirring during the hydrothermal process allowed to accurately control the average size of the particles produced (200 – 1200 nm range). For all the conditions tested, the characterization of the powders showed the formation of fluorite type $UO_2.nH_2O$ samples with traces of residual organics at the surface. Both water and residual organics were eliminated by heating at 600°C. Preliminary sintering tests are now under progress on such precursors.

Conversion of uranium nitrate into uranium oxide by Solution Combustion Synthesis – CNRS/ICSM

Actinide surrogate systems $GdNO_3$/fuel (fuel = glycine, urea, citric acid) were investigated to propose an experimental protocol for the conversion of uranium nitrate into uranium oxide by Solution Combustion Synthesis (SCS). The fuel over gadolinium nitrate ratio appeared as a critical parameter during the conversion, and may induce phase transitions for the final product. The highest temperature of conversion was obtained for a ratio of 1 leading to the formation of monoclinic form of Gd_2O_3. For higher ratios, the conversion temperature was lowered and the cubic form of Gd_2O_3 was observed. The end-products exhibited interesting characteristic for sintering (specific surface area typically of 10-20 m^2/g). Preliminary studies were performed on the conversion of uranyl nitrate into uranium oxide.

DIFFICULTIES

Studies of non-powder routes for the synthesis of MOX fuels materials, potentially bearing minor actinides and blanket fuel materials – SCK.CEN

The thermal treatment has to be improved to prevent the fragmentation of spherical particles with Ce contents higher than 20%.

ACTION PLAN

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SCK.CEN

During the next semester, the products will be analysed using SEM and different sintering conditions will be investigated. Moreover, comparable composition will be fabricated by the same method but using Ce(IV) (cerium ammonium nitrate) a precursor instead of Ce(III) (cerium nitrate).

CNRS/ICSM

The direct precipitation of uranium oxides under hydrothermal conditions will be extended to (U,Ln)O_2 mixed oxides.

The sintering capability of UO_2 microspheres will be extensively studied through dilatometry tests and further SEM examinations.

The experimental protocol developed for the conversion of gadolinium nitrate to gadolinium oxide by Solution Combustion Synthesis is now under transposition to uranium oxide powders. Extensive experiments will be performed during the next periods.

UNIMAN

The lab work will begin following safety training and induction of the new PDRA. A master student for the coming academic year will be allocated to assist in the lab at no extra cost for the project. UNIMAN anticipate that the first binary f element oxide solid solutions will be available, prepared as coatings on substrates using polymer assisted deposition, at the end of the next semester.

WP5

MAIN RESULTS

TASK 5.1

MANPOWER

- CIEMAT : 4 pm
- ULANC: 6 pm
- CNRS: 3.5 pm
- ULEEDS: 0.3(+6) pm
- CEA: 0 pm

MAIN PROGRESSES

- CIEMAT:
  - Designs and adjustment for the implementation of an irradiation loop at Náyade facility:
    - Fabrication of a new irradiation device for dynamic experiments.
    - Fabrication of 3 new reactors to contact phases during dynamic irradiation (inside and outside of the pool).
  - On-going activities to find the relevant conditions to simulate effects of radiation along *i*-SANEX process (TODGA/SO_3PhBTP systems):

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- Quantitative studies of TODGA and its DCs after irradiation (200 and 500 kGy) of two phases in contact (air, argon, air-sparging) (Work related to WP2 task 1).

- ULEEDS:
  - Progress has been made in the multiphase modelling procedure for pulsed sieve plate extraction columns (PSPECs) using the open source software OpenFOAM. First studies include development of a single phase and multiphase model which has been completed and used as the basis of the conference proceedings paper for WM2018 Phoenix. Subsequent studies and publications involve the comparison of various CFD modelling approaches (RANS or LES) and justification for the one chosen (LES - large eddy simulation) and used going forward. Progression in this new study has been made, improved LES model, RANS model set-up and ready to run.
- ULANC
  - Rotating Diffusion Cell (RDC) studies have been performed on the solvent extraction kinetics of lanthanide fission products by TODGA and the effects of solvent phase acidity on the effectiveness of this organic extractant molecule. Quantification of the extraction kinetics indicates that HNO3 co-extracts with the metal ion, although metal ion extraction kinetics are a factor of 10 faster than those of HNO3.
  - We have observed a reduction in the rate of cerium extraction at long RDC experiment times. Earlier studies (see HYPAR1) have shown that this is a result of the increase in solvent phase acidity arising from the co-extraction of HNO3 with the metal ion. Quantification of solvent phase acidity associated with the onset of extraction inhibition indicates that it correlates strongly with the solvent phase acidity that induces TODGA reverse micelle formation in the solvent phase.
  - Thus, we attribute the observed inhibition of the extraction to aggregation of the extractant. This effect is not expected to obtain when the extraction is run at aqueous phase acidities lower than 0.7 mol dm-3 and this is the subject of further work.
  - New PhD student, Alex Jackson, has completed his intensive 3 month training course and has begun work on refining the theoretical model of the RDC such that it may be applied in a wider range of extraction systems. Upon completion of this modelling study, he begin work on the further work highlighted in point 3 above.
- CNRS
  - The kinetics of extraction of radioactive 152Eu(III) from aqueous HNO3 solution by the extractant TODGA (+5% vol. octanol-1) in TPH have been studied using the rotating membrane cell (RMC) technique. The effects of varying the aqueous concentration of HNO3, of TODGA and of SO3-Ph-BTP (on the kinetics of stripping in the latter case) have been studied. Simple explanations of the effects of the latter two concentrations may be given. This is not the case for the effect of [HNO3] in aqueous phase.

DIFFICULTIES

- ULEEDS:
  - Modelling of multiphase PSPECs is computationally expensive, finding the resources in order conduct studies has been difficult. My availability on the University of Leeds HPC (ARC3) has been throttled due to heavy use, as such progression of models has been limited.
- CNRS

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- The new radioactive solution of 152Eu(III) has been received with some delay. During that period we have implemented experiments of diffusion by NMR and of extraction of HNO3.

- CEA
  - Temporary unavailability of the single drop experimental device.

ACTION PLAN

- CIEMAT
  - First tests of the new devices for an irradiation loop.
    Material evaluation to radiation, organic solvents and high nitric acid concentrations to build a robust irradiation loop.
    On-going activities to find how to simulate relevant-process conditions:
  - TODGA-DMDOHEMA/SO_3PhBTP system.
  - TODGA-DMDOHEMA/AHA-SO_3PhBTP system.
- ULANC
  - Alex Jackson to have completed work on validating existing analytical model (developed during SACSESS) of the Rotating Diffusion Cell (RDC) and to have begun experimental study of the effect of reduced acidity on suppressing the extraction inhibition effect.
- CNRS
  - We will investigate more in depth the kinetics of extraction and stripping of Eu(III) and probably start that of Am(III) (if it is available from our supplier LEA-CERCA).
  - The extractants will be TODGA (+5% octanol) and CyMe4BTBP (+TODGA).
  - The concentrations of the extractants will be varied.
  - The effect of aqueous complexing agents (SO3-Ph-BTP + possibly AHA, TPAEN, PTD,…) on the rates of stripping will be investigated by varying their concentrations.
- ULEEDS
  - Pulsed Column: To apply alterations to the boundary conditions developed from the rudimentary model to the complete model. To conduct the parametric study on the complete model, potential for a scientific paper to be produced from these results alone. Continue with original plan outlined in the previous HYPAR report, to conduct an in-depth study comparing the different turbulence (flow) modelling approaches to justify the use of the method chosen (large eddy simulation).
  - Centrifugal contactors: Continuation of the development of the numerical method for simulating centrifugal contractors, improving the code. In addition, some more numerical tests for the developed methodology in two and three dimensions will be carried out for the assessment of the new algorithm. Finally, the details of the developed method will be presented in a journal paper together with numerical results showing the capabilities of the method
- CEA
  - Single drop device to be restored. Determination of mass transfer coefficients of Am and Eu with the single drop technic in the stripping conditions of Euro-GANEX (TPAEN solution and a TODGA-octanol solvent)

TASK 1.2

NO ACTIVITY

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TASK 1.3

MANPOWER

- JUELICH: 0.5 pm
- KIT: 1 pm
- NNL: 0.6pm

MAIN PROGRESSES

- JUELICH
  - TASK T53. FLOWSHEET AND MODELS: Data from single centrifugal contactor tests (AmSel system) were shared with KIT.
- KIT:
  - Flow-sheet calculations for the selective stripping/re-extraction sections for the PTD-i-SANEX process were performed.
  - Flow-sheet calculations for the selective stripping/re-extraction sections for the SO3-Ph-BTBP AmSel process were started.
- NNL
  - Draft paper on modelling HNO3 extraction into i-SANEX solvent was revised.

DIFFICULTIES

- NNL
  - Unavailability of key resource

ACTION PLAN

- JUELICH
  - An AmSel process flow-sheet will be developed in collaboration with KIT, based on the results of single centrifugal contactor tests. The flow-sheet will be tested in our centrifugal contactor battery
- KIT
  - Complete AmSel flow-sheet calculations
- NNL
  - Modeling will now start in semester

WP6

INTRODUCTION

The objectives of WP6 are to optimise the efficiencies and safety of separation processes developed under the SACSESS project (*i.e*. i-SANEX, EURO-GANEX, EXAm, CHALMEX) The main emphasis is on process development through flowsheet testing. WP6 focuses on optimisation of the reference separation processes, particularly where significant simplification is possible or replacement of non-CHON ligands by CHON molecules can be proposed. It also includes extension of process envelopes to

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more challenging GenIV feeds and integration of the minor actinide SX cycles with upstream and downstream stages, particularly where interfaces may cause issues.

MAIN RESULTS

TASK 1.1: HOMOGENEOUS RECYCLING

MANPOWER

A total of 4 man-months were booked by CHALMERS for Task 1.1 (homogeneous recycling).

	task 1		task 2	
	expected	achieved	expected	achieved
JUELICH	0	0	3	3
KIT	2	0	0.5	0
CHALMERS	4	4	0	0
POLIMI	0	0	0.25	0.5

MAIN PROGRESSES

Investigations of a mixture of TBP and FS-13 as diluent in a GANEX (CHALMEX) process have been continued. In this semester, kinetics experiments, stripping experiments, experiments with varying concentration of CyMe4-BTBP have been performed, along with experiments with varying volume ratios of TBP and FS-13.

Kinetics experiments, where contact times between phases were varied, concluded that plutonium extraction is stable at high distribution ratios (D>10) in the system, and reaches equilibrium within 5 minutes, regardless of the plutonium concentration. Am extraction decreases with increasing plutonium concentrations, and only reaches equilibrium in a system containing 10 g/L Pu. Europium extraction appears to decrease with increasing plutonium concentrations, and equilibrium is reached after 10 minutes regardless of plutonium concentrations. Stripping experiments showed that after 1 stripping stage, approximately 90% of plutonium is stripped, while only approximately 10% of americium is stripped. After a second stripping stage, practially all americium is stripped out, while ~1% of plutonium remains.

In systems with 30%vol TBP and 70%vol FS-13, but varying concentrations of $CyMe_4$-BTBP, it was found that both plutonium and americium extraction increase with increasing extractant concentrations. A low separation factor between europium and neptunium is achieved, and at $CyMe_4$-BTBP concentrations above 35 mmol dm^{-3}, a higher extraction of europium compared to neptunium was observed.

DIFFICULTIES

Availability of key ligands such as PTD and m-TDDGA in sufficient quantities has been highlighted as a potential problem (for new EURO-GANEX process development).

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ACTION PLAN

A GANEX sub-team has been proposed to plan the experiments needed to demonstrate use of PTD and m-TDDGA in place of the sulphonated-BTP – TODGA/DMDOHEMA system used in EURO-GANEX at present. A first sub-meeting is now required.

TASK 1.2: HETEROGENEOUS RECYCLING

MANPOWER

A total of 3.5 man-months were booked against Task 2 (heterogeneous recycling) by JUELICH and POLIMI.

	task 1		task 2	
	expected	achieved	expected	achieved
JUELICH	0	0	3	3
KIT	2	0	0.5	0
CHALMERS	4	4	0	0
POLIMI	0	0	0.25	0.5

MAIN PROGRESSES

Additional single centrifugal contactor experiments were carried out using the AMSEL system, which is based on the i-SANEX process – except a modification will made to the scrubbing section (0.5 mol/L HNO_3 only). Stripping of Am(III) is done using a solution of SO_3-Ph-BTBP in nitric acid. In the first single centrifugal contactor test 0.78 mol/L HNO_3 was used, but this was reduced in the second test to 0.70 mol/L HNO_3. Additionally, the Cm + Ln re-extraction was tested. Figure 1 shows a tentative flowsheet for an AMSEL process, which was used to elaborate appropriate conditions for the single centrifugal contactor tests.

For the single centrifugal contactor tests a loaded organic solvent was prepared by batch contacts following the flow-sheet in Figure 1. First, a simulated HAR solution containing 0.05 mol/L CDTA was contacted with fresh solvent. Then, the separated loaded solvent was scrubbed twice with 0.5 mol/L HNO_3. During the second scrubbing step ^{152}Eu, ^{241}Am, and ^{244}Cm tracer were added to the aqueous phase. According to the flow-sheet in Figure 1 this loaded organic solvent was diluted 1:1 with pre-contacted solvent (pre-contact with 0.5 mol/L HNO_3), as such a dilution will occur in the Am stripping section. The first single centrifugal contactor test was run using two flow-rates: 80/40 and 40/20 ml/h (org./aq.). When the steady-state was reached for the first flow-rate, it was set to the new flow-rate. After reaching the second steady-state, the experiment was stopped and the content of the contactor's mixing chamber was shaken for 15 min to reach the chemical equilibrium. Figure 2 shows the Am(III), Cm(III), and Eu(III) distribution ratios as a function of the experiment run time and the equilibrium distribution ratios. The stage efficiencies were relatively low, as stage efficiencies of 25% and 35% were reached for Am(III) at the higher and lower flow-rate, respectively. The selectivity of the system was good, with Eu/Am separation factors of ca. 400 and Cm/Am separation factors of 2.5 – 2.6.

The second single centrifugal contactor test was run using two flow-rates: 40/20 and 20/10 ml/h (org./aq.). When the steady-state was reached for the first flow-rate, it was set to the new flow-rate. After reaching the second steady-state, the experiment was stopped and the content of the contactor's mixing chamber was shaken for 15 min to reach the chemical equilibrium. Figure 3 shows the Am(III), Cm(III), and Eu(III) distribution ratios as a function of the experiment run time and the equilibrium

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distribution ratios. This time, the distribution ratios of Am and Cm were acceptable for further flow-sheet development. The stage efficiencies were higher compared to the first test, as stage efficiencies of 50 - 60% were reached for Am(III). The selectivity of the system was good, with Eu/Am separation factors of ca. 400 and Cm/Am separation factors of 2.5 – 2.6.

In a third single centrifugal contactor test, the conditions for Cm + Ln re-extraction were tested, as shown in Figure 1. Here, a 1:1 phase ratio is assumed, therefore a flow-rate of 20/20 ml/h (org./aq.) was tested. The aqueous samples of the second single centrifugal contactor test were combined and used as the aqueous feed, while fresh 0.2 mol/L TODGA solution in TPH + 5 vol.-% 1-octanol, pre-contacted with 0.5 mol/L HNO_3 was used as the organic feed. Figure 4 shows the Am(III), Cm(III), and Eu(III) distribution ratios as a function of the experiment run time and the equilibrium distribution ratios. Good distribution ratios for Am(III) of D~0.7 and Cm(III) of D~1 were obtained. Interestingly, the selectivity of the system was different in the transient samples and the equilibrium sample, as transient $SF_{Cm/Am}$ values of 1.6 were obtained, while the equilibrium $SF_{Cm/Am}$ value was 2.5. This resulted in different stage efficiencies for Am and Cm of 47 and 33%, respectively.

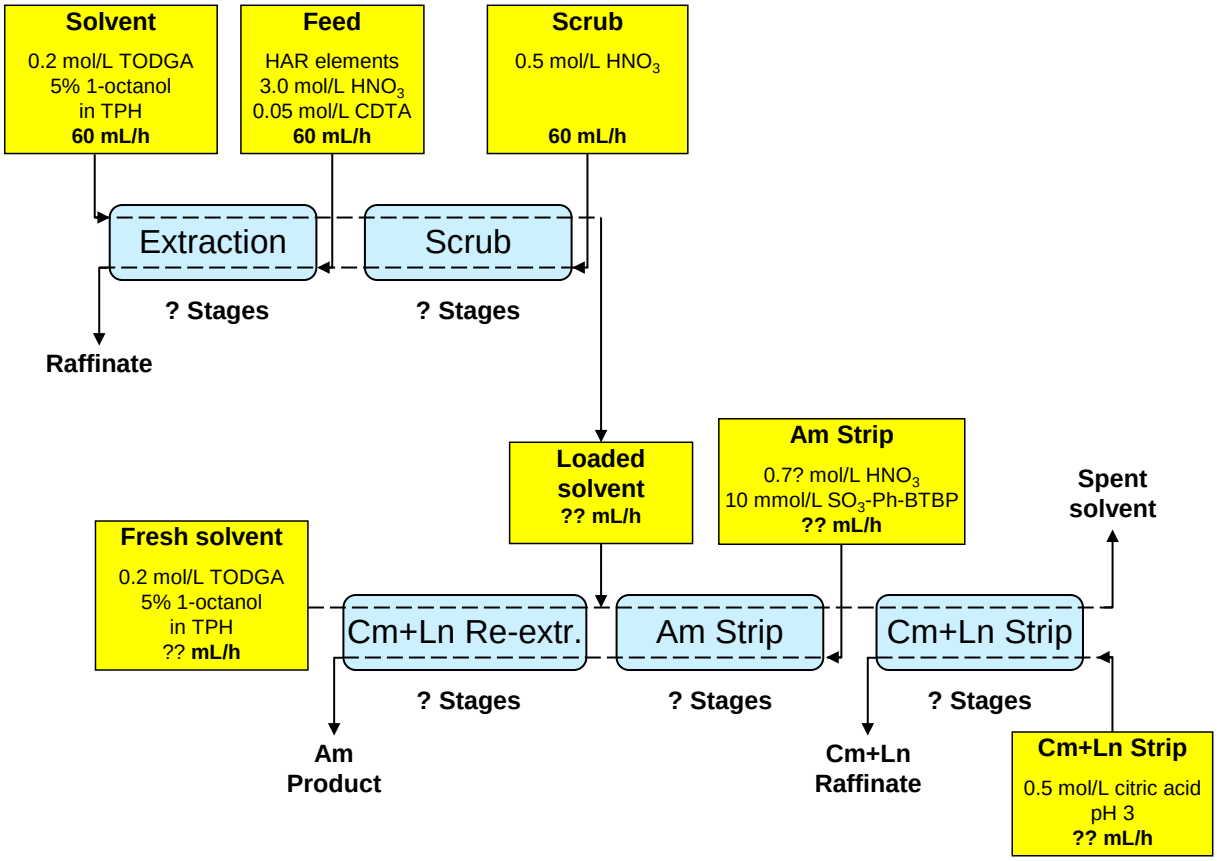

Figure 1. Tentative flowsheet of an AMSEL process.

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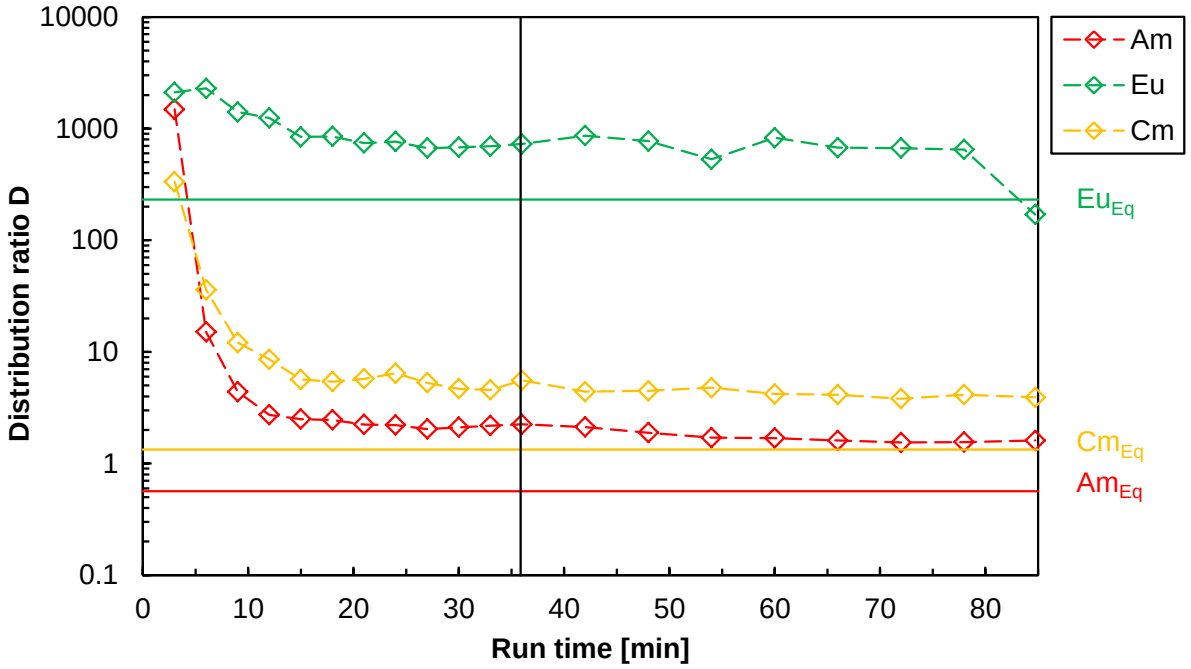

Figure 2. Am(III), Cm(III), and Eu(III) distribution ratios as a function of the experiment run time of the first single centrifugal contactor test (data points, dashed line to guide the eye) and equilibrium distribution ratios (horizontal lines).

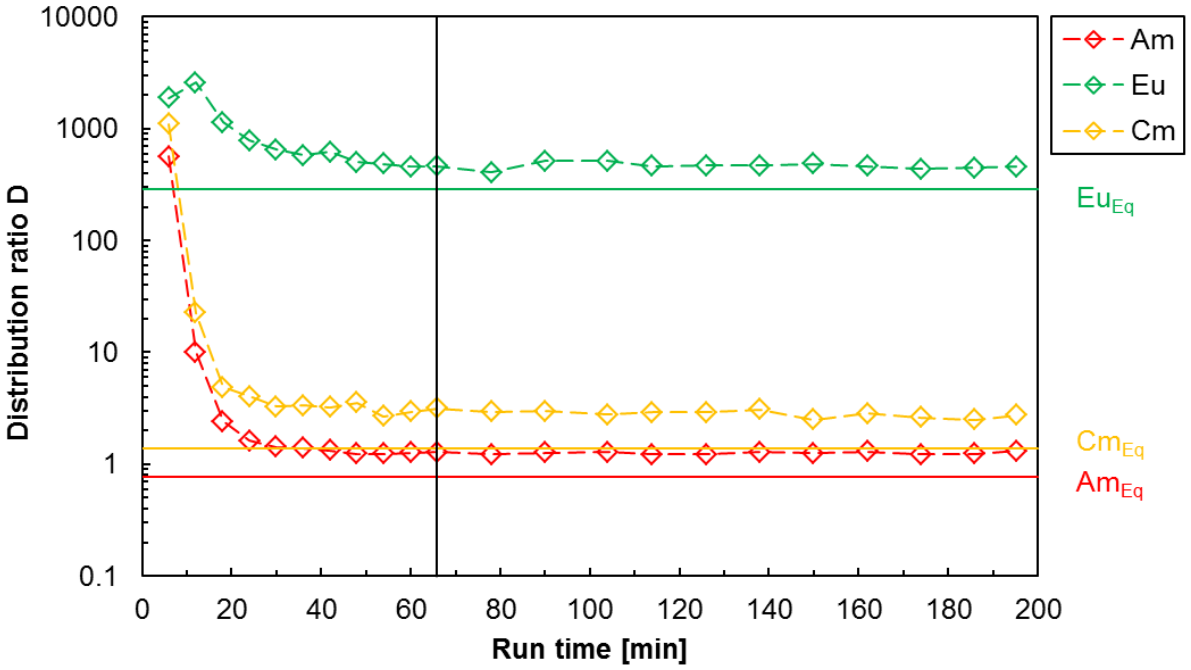

Figure 3. Am(III), Cm(III), and Eu(III) distribution ratios as a function of the experiment run time of the second single centrifugal contactor test (data points, dashed line to guide the eye) and equilibrium distribution ratios (horizontal lines).

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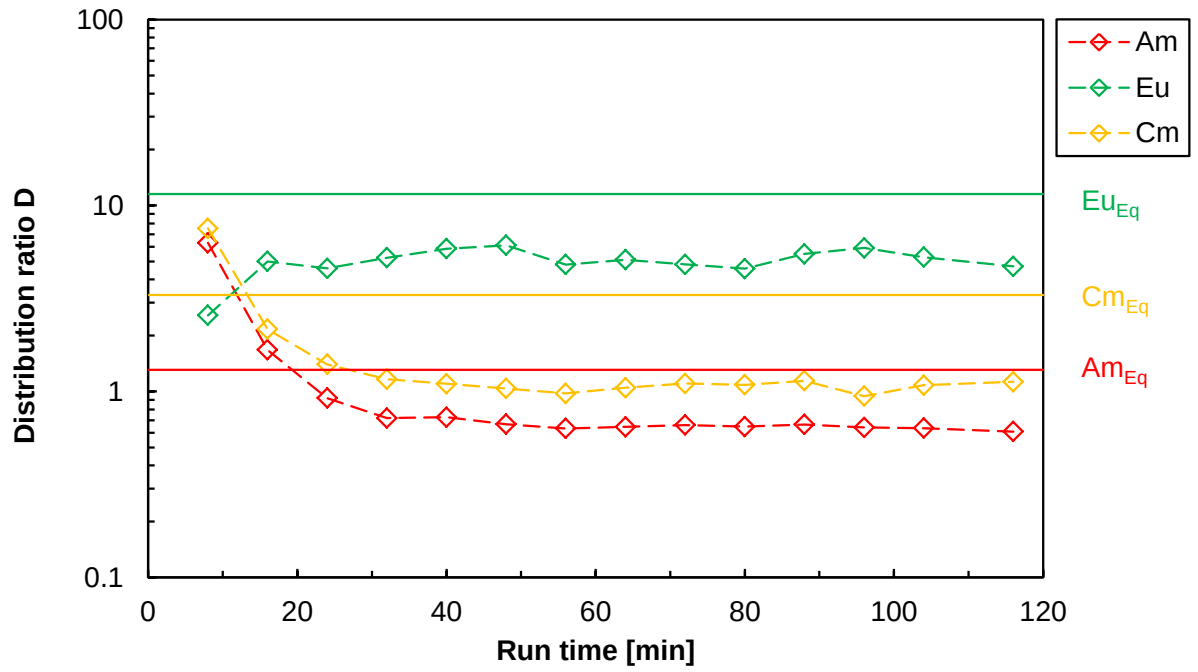

Figure 4. Am(III), Cm(III), and Eu(III) distribution ratios as a function of the experiment run time of the third single centrifugal contactor test (data points, dashed line to guide the eye) and equilibrium distribution ratios (horizontal lines).

ACTION PLAN

The data of the different single centrifugal contactor tests will be used for flow-sheet development at KIT. A process flow-sheet will then be tested in the JUELICH centrifugal contactor battery in the next semester.

WP7

INTRODUCTION

The objective of WP7 is to provide relevant dissolution and conversion processes linkable with the separation processes. When necessary, interfaces between dissolution/separation and separation/conversion are considered. For dissolution, the focus is put on the completion of data relevant for defining a dissolution model. The conversion issues of solutions from SX-process are the development of safe synthesis routes, the destruction of prejudicial organics from SX-process and characterization of synthesized MABB precursors. The workpackage is divided into 4 tasks: Task7.1 on dissolution, Task7.2 on destruction of organics, Task7.3 on development of safe synthesis routes and Task7.4 on characterization of MABB precursors. This semester major progresses were achieve in Task 7.1 with fthe first dissolution data obtained on model compounds, and in Task 7.3 with $U_{0.9}Am_{0.1}O_{2-\delta}$ synthesis using weak acid resin route as dust-free MABB precursors while a concept flowsheet for (U,Pu) and MA co-conversion processes have been designed in EXCEL for the definition of the work program to be done at NNL.

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MAIN RESULTS

TASK 7.1

MANPOWER

Expected effort for the period 2 men.month

Actual effort spent during the period 2 men.month

MAIN PROGRESSES

In the previous semester eighteen samples of (U,Pu)O_2 were synthetized and characterized with a Pu amount ranging from 0 to 100 % and with different morphologies in order to be able to collect data on dissolution kinetics on single phases depending on the dissolution parameters on one hand and on the solid parameters (Pu amount, morphology parameters: crystallite size, SSA,…) on the other hand. During this semester, the first dissolution experiments were carried out after setting up the dissolution apparatus in a glove box (**Figure 5**). This semester was dedicated to the dissolution of PuO_2 samples. Each dissolution test was conducted in a dissolution reactor of 60mL with 15 mL of nitric acid and 300 mg of oxide powder. The lid of the reactor was equipped with a cooling column allowing potential nitrous vapors to condense. For safety issues the top of this cooling column was connected to a gas cleaner composed of soda 2M to ensure no nitrous emission inside the globe box. The solution was heated using a hot plate; temperature was followed using a thermometer. The solution was homogenized using a magnetic stirrer at 300rpm. Once the solution at the right temperature, the oxide powder was introduced inside the reactor at t=0. Then, aliquots of the dissolution liquor were sampled using a syringe and analyzed by alpha counting to follow the dissolution kinetics.

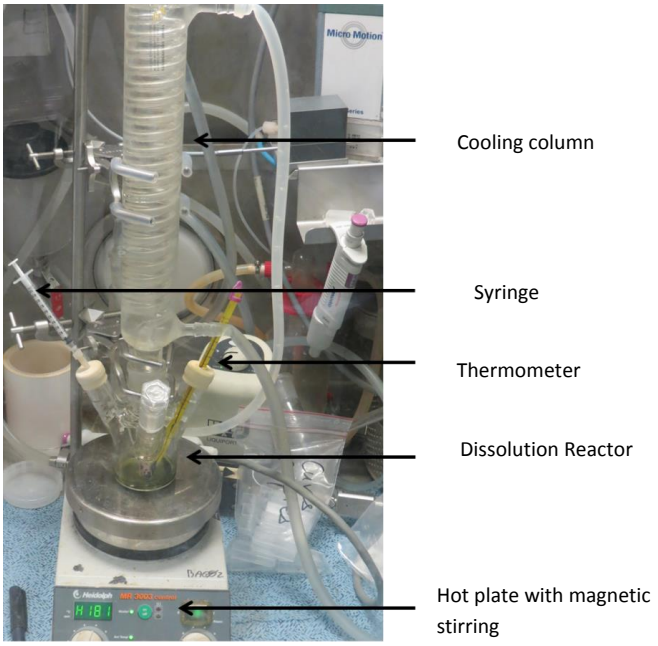

Figure 5: Dissolution apparatus set up in glove box

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In order to determine the impact of the morphology of the PuO_2 powder on its dissolution kinetics, dissolution tests were carried out using [HNO_3] 8.5M at 95°C on the four different PuO_2 samples previously synthetized. Characterization parameters of these 4 PuO_2 powder samples are given in **Table 1**. Dissolution tests were conducted for 6h. At the end, dissolution liquors remained colorless (**Figure 6**) reflecting the very low dissolution kinetics of PuO_2 even in very aggressive conditions, as it is already well known. At the end of the dissolution experiments more than 90% of the powders were recovered after filtration.

Table 1: Morphological characteristics of PuO_2 powders.

Compound	Precursors route	Average crystallite size (nm)	Specific surface area ($m^2.g^{-1}$)
1	Sol-gel 850°C	17 (1)	6.3 ± 0.5
2	Sol-gel 1500°C	270 (15)	0.2 ± 0.2
3	Oxalic platelets	92 (3)	3.3 ± 0.4
4	Oxalic sticks	75 (2)	2.7 ± 0.4

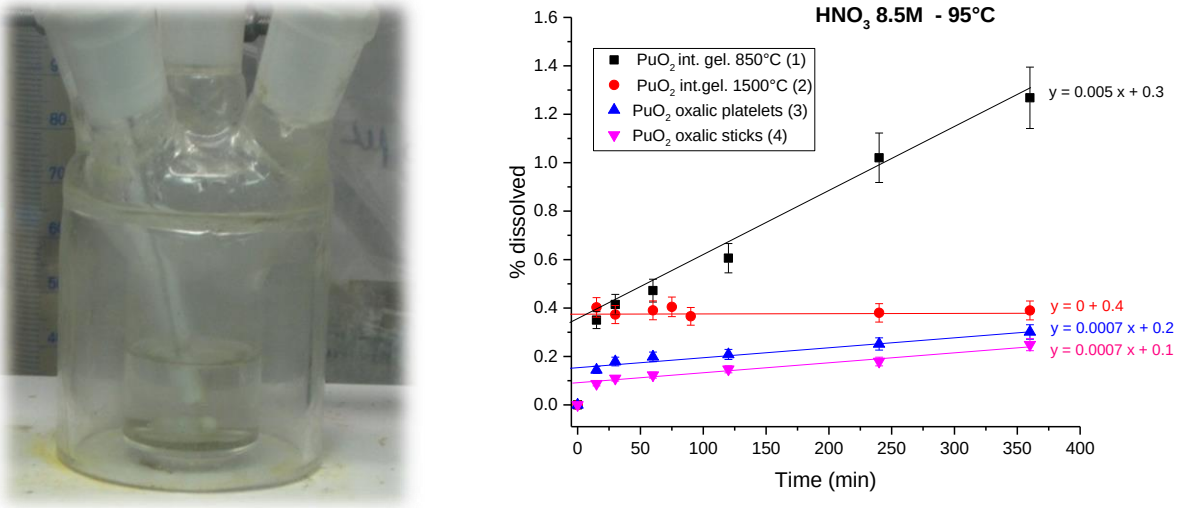

Figure 6 : Picture of dissolution of PuO_2 sample in HNO_3 8.5M 95°C after 6h (left) Dissolution kinetics of PuO_2 powders in HNO_3 8.5M – 95°C. (right)

Dissolution kinetics rates are plotted in **Figure 6**. The determination of the plutonium concentration in solution allows determining the mass of dissolved plutonium dioxide and thus the percentage of powder dissolved at each time. All these percentages lead to a straight line and by linear regression, the slope of the line corresponds to the dissolution kinetics rate. An initial dissolution kinetics regime is observed corresponding probably to an erasing of the crystalline defects more reactive with respect to dissolution and is expressed by the non-nil y-intercepts.

Sample 1 obtained by internal gelation calcined at 850°C has the higher dissolution kinetics in agreement with the morphology parameters (higher specific surface area and smaller crystallite size). Then, both samples obtained by oxalic route have similar dissolution kinetics whatever the platelet or stick morphology despite different specific surface area and crystallite size values. It seems that the

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effects of these two parameters annihilate each other to lead to similar dissolution behavior for both samples. Finally, PuO_2 obtained by internal gelation calcined at 1500°C exhibit dissolution kinetics rate close to nil. This powder has very low specific surface area and a crystallite size sixteen times higher than the same sample calcined at 850°C that can explain this dissolution resistance.

Based on these data it was possible to establish a kinetics law with respect to the two morphology parameters (specific surface area and crystallite size) for these 4 samples:

$$r\ PuO_2 = k * SSA^{0,33} * Sc^{-1}$$ **Equation 1**

with r : dissolution kinetic (%dissolved / min) ; k=0.04 , SSA: specific surface area ($m^2.g^{-1}$) ; S_C: average crystallite size (nm)

The correlation coefficient associated with this formula is 0.96. This coefficient is acceptable considering the errors of the quantification of the powder average crystallite sizes and specific surface areas.

ACTION PLAN

Dissolution experiments will continue on the other samples in order to collect data to build a dissolution model.

TASK 7.2

Nothing done on this Task during this semester

TASK 7.3

MANPOWER

Expected effort for the period 3 men.month

Actual effort spent during the period 3 men.month

MAIN PROGRESSES

$U_{0.9}Am_{0.1}O_{2-\delta}$ synthesis using weak acid resin route as dust-free MABB precursors.

Up to now $U_{1-x}Am_xO_{2\pm\delta}$ pellets are produced by powder metallurgy processes [1,[2–[4]. One route used was recently optimized and called UMACS process [5,[6]. In this process, preparation of a single phase mixed oxide involves several steps: blending of AmO_2 and UO_2 initial powders, pelletization, heat treatment and grinding are necessary before compaction into pellets. At an industrial scale, the retention of very fine and highly contaminant particles generated in the fabrication line could be a disadvantage for the use of powders as precursors. Furthermore the use of homogeneous precursors was found to be profitable in terms of sintering to optimize both compound density and microstructural homogeneity [5,[7]. The fabrication of $U_{1-x}Am_xO_{2\pm\delta}$ pellets from mixed uranium-americium oxide obtained by oxalate co-conversion [8] was thus recently reported [9]. The reported results evidenced the advantages of such a process with compounds reaching 95% TD (of the

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theoretical density). This process is, however, also based on the use of powders, which should be avoided when working with highly contaminant materials such as americium. A dust free route involving the handling of americium-containing precursor in the form of millimetric spheres is therefore recommended and led to the development of alternative processes using sol–gel method combined with impregnation [10] or weak acid resin as organic template [11]. This later alternative process, called Calcined Resin Microsphere Pelletization (CRMP) [12,[13], consists in pelletizing and sintering oxide microspheres issued from mineralization of metal-loaded ion exchange resin beads. Tests of the process have been carried out on different metals such as lanthanides and uranium. Influence of heat treatment on the characteristics of cerium oxide microspheres and on the quality of final sintered pellet was studied and dense cerium oxide pellets were produced in improved conditions. Uranium dioxide pellets with different porosity contents were also obtained by mixing U_3O_8 and UO_2 like microspheres in different ratios, pelletizing and sintering [14]. In the present study, the CRMP process is applied to the fabrication of uranium-americium mixed oxide compounds in the form of dense $U_{0.90}Am_{0.10}O_{2\pm\delta}$ pellet. The work done during this last semester was the production of the microspheres that will be used to produce the dense pellets during the next semester.

1. Experimental

1.1. Oxide microsphere synthesis

1.1.1. Resin preparation

The ion exchange resin used for the fixation was an IMAC HP333 carboxylic resin supplied by Dow Chemicals (Dow Chemicals, Chauny, France). The resin was sorted beforehand under wet condition and the 630–800 µm size range was selected for experiments. An extensive washing cycle was then performed on the resin (ammonia, nitric acid and demineralised water) in order to remove the synthesis residues and cationic impurities still present. Eventually, the washed resin was prepared in its protonated form.

1.1.2. Preparation of the loading solution

The loading solution was prepared in two steps. A stock solution of americium (III) nitrate ($Am(NO_3)_3$) was first obtained by the dissolution of 297 mg of AmO_2 powder in a 1 M nitric acid solution. This solution corresponded to a volume of 12.5 mL and to a 0.08 mol L^{-1} Am nitrate solution and to an excess of nitric acid of 0.11 mol L^{-1}. Then uranium trioxide solid (UO3) (prepared as in Ref. [11]) was added to this solution as well as deionized water and molar nitric acid. The role of UO_3 dissolution was to increase both pH and uranium concentration by reacting with acid excess and by forming hydrolysed uranyl species. The final solution corresponded to an Acid Deficient Nitrate Uranyle solution (ADUN) [15] mixed to americium nitrate with the following uranium formula $UO_2(NO_3)_{1.2}(OH)_{0.8}$ and with uranium and americium concentrations of 0.101 and 0.012 mol L^{-1}, respectively, an Am over metal ratio of 10.5 at.% and a volume of 83 mL. Characteristics of the resin and initial load used for the fixation are gathered in Table 2.

Table 2 Characteristics of the resin and loading solution.

Resin	Dow chemicals H^+ form	IMAC HP333
	m (g)	0.904
	Q_{weight} (meq/g)	11.6
Loading solution	V (mL)	83
	[U] (mmol L^{-1})	101
	[Am] (mmol L^{-1})	11.9
	[Am/(Am + U)] (%)	10.5
	pH	3.93

Q_{weight}: scientific weight capacity in meq/g dry H^+ resin
meq/g dry H^+ :milliequivalent per gram of dry H^+ resin

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1.1.3. Cation exchange

Resin beads were rehydrated and introduced into a glass column. The load solution was recirculated 6 times through the resin bed at a controlled flow rate of 2 mL min^{-1} for a total contact time of 4 h. pH of the load solution was recorded during fixation. From the moment when pH did not change significantly, exchange reaction was considered to be achieved. The loaded resin was then washed with deionized water and drained under low vacuum. The initial load and eluate solution were then analyzed by thermal ionization mass spectrometry (TIMS), in order to determine metal concentrations and to express the fixation yield. The metal loaded resin was then directly heat treated without any more drying.

1.1.4. Thermal treatments

A first thermogravimetric analysis (Netzsch STA 449 thermogravimetric analyser) was carried out on a sample of the metal loaded resin under a reconstituted air flow (Airliquide, 20 vol.% O_2–80 vol.% N_2) up to 800 °C. Gas releases (i.e., H_2O, CO_2, CO) were monitored using a coupled gas micro-chromotography module (SRA), (TGA–µGC). Two thermal treatments were then applied to the microsphere lot in a tubular furnace (Lenton LTF). The resin was firstly calcined at 700 °C for 1 h under a reconstituted air flow (10 NL h^{-1}). Initial heating rate of 5 °Cmin^{-1} was reduced to 1 °C min^{-1} from 200 °C up to final temperature in order to ensure an optimal evacuation of the calcination gas generated during the degradation of the polymer and avoid crack formation **[12]**. The oxide formed at 700 °C was secondly reduced under a reducing treatment in Ar/H_2 (4 vol.%) at 700 °C for 6 h with a constant heating rate of 10 °C min^{-1}.

1.2. Oxide microsphere characterization

The crystalline structure of the resulting oxide was characterized by X-ray diffraction (XRD) using a $\theta-\theta$ D8 advance BRUKER diffractometer equipped with a copper anticathode ($\lambda(K^{Cu}_{\alpha 1})$ = 1.54056 Å, $\lambda(K^{Cu}_{\alpha 2})$ = 1.54439 Å). XRD patterns were collected in presence of an internal Au standard at room temperature by step scanning (0.02° steps at 1 s/step) across the angular range $10° \leq 2\theta \leq 80°$. A Field Emission Gun Scanning Electronic Microscope (FEG-SEM, Zeiss Supra-55 VP) was used in order to observe the morphology and the microstructure of the synthesized oxide microspheres.

The apparent density of the microspheres was determined by volume and mass measurements of a representative sample of a few hundreds of beads. The number of spheres and the sample volume were obtained from image analysis of one layer of microspheres (assumed as perfect spheres) using an optical microscope (Olympus BX30 M) coupled to an Ellix pattern recognition software (Microvision).

The residual carbon content of the reduced oxide was determined by monitoring the CO_2 emissions occurring during calcination up to 1000 °C under air of the oxide precursors, via TGA–µGC analysis.

The metal contents of the oxide were determined from TIMS analysis of dissolved oxide.

2. Results and discussion

2.1. Loading of the resin

2.1.1. Exchange equilibrium

The different hydroxide complexes of the ADUN solution, mainly $(UO_2)_2(OH)^{3+}$ and $(UO_2)_2(OH)_2^{2+}$, constitutes the driving force of the exchange reaction between the counter-ions H^+ in the resin and UO_2^{2+} and Am^{3+} cations in solution **[7]**. Acting like a buffer, it notably prevents from pH increase caused by the release of H^+ in solution and allows the ion exchange reaction to take place as shown in **Equation 2** in the case of U exchange only (R representing the exchangeable site of the resin):

$$2RH + (UO_2)_2(OH)_2^{2+} + 2NO_3^- \rightarrow R_2(UO_2) + UO_2^{2+} + 2NO_3^- + 2H_2O \qquad \textbf{Equation 2}$$

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The determinations of Am/(U + Am) ratios in the loading solution (10.5 ± 0.3 at.%) and in the oxide (10.7 ± 0.2 at.%) are not significantly different. Therefore, the resin does not exhibit any selectivity between UO_2^{2+} and Am^{3+} cations at this Am content level.

2.1.2. Kinetic of the exchange reaction

Evolution of pH in the load solution during exchange (**Figure 7**) describes the exchange kinetics. The pH of the load solution significantly decreases during the first hour and a half when exchange is occurring and then stabilises after 4 h of contact when equilibrium is reached. Exchange is slow because it is mainly governed by interdiffusion of monovalent and divalent/trivalent cations in the resin **[16]** with weak diffusion coefficients (typical values of 8 x 10^{-9} cm^2s^{-1} for trivalent cations and 1.6 x 10^{-7} cm^2s^{-1} for monovalent H^+) and conversion times are about 3 h in this case **[17]**.

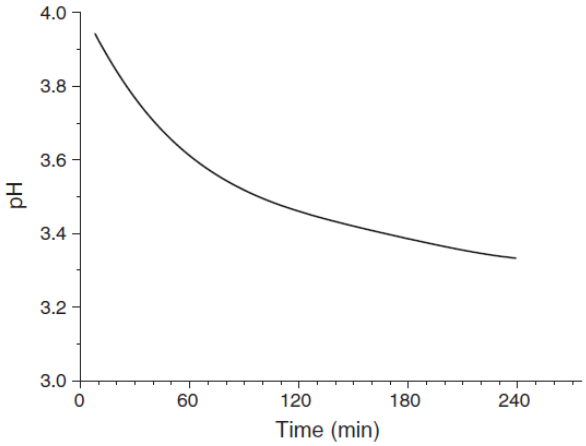

Figure 7 Evolution of pH in the loading solution as a function of time of exchange.

2.1.3. Fixation yield

Fixation yield is calculated from the ratio of the amount of actinide cations fixed in the resin, expressed in meq, over the capacity of the resin used (Table 3). Its value equals 66% which is in fair agreement with previous studies concerning the loading of UO_2^{2+} alone which reports a yield fixation of 57% **[13]**. Taking into account Am/(U + Am) ratio in the resin and considering that the resin is exchanged up to 66%, the formulation of the metal loaded resin may be written as:
$R_{21}(UO_2)_9Am;(RH)_{11}$ (with R representing the resin exchangeable site).

Table 3 : Fixation yield of UO_2^{2+} and Am3+ in the resin.

n (UO_2^{2+}) loaded (mmol)	3.0
n (Am^{3+}) loaded (mmol)	0.3
Fixed An (meq)	6.9
Resin capacity (meq)	10.4
Fixation yield (%)	66

2.2. Oxide microspheres synthesis

2.2.1. Thermogravimetric and gas analysis

Thermogravimetric analysis by TGA–µGC was carried out on the loaded resin and is shown in Figure 8. The monitoring of the gas released during calcination in air is reported in Figure 9. A first 15.4% weight loss was recorded between 30 °C and 250 °C associated with an endothermic reaction. This phenomenon is attributed to the dehydration of the resin which

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gives two H_2O emissions peaks revealed by µGC analysis. The peak (a) between 30°C and 70 °C is associated with a mass loss of 3.2% and corresponds to the evacuation of H_2O still present in the resin pores. The peak (b) is recorded between 70 °C and 180 °C with a weight loss of 12.2%. It would correspond to the water of hydration of uranyl(VI) and americium(III) cations bound to the resin [16]. The mean hydration number of the actinide cation inside the resin would equal 4.

Degradation of carboxylic skeleton in the form of CO_2, H_2O and CO occurs between 250 °C and 480 °C in an exothermic reaction with a weight loss of 44.8%. A maximum release peak is observed at 370 °C. Experimental weight loss of 44.8% of the hydrated loaded resin, which corresponds to a 53.2 ± 0.8% weight loss of the fully dehydrated loaded resin, can be compared to the theoretical weight loss calculated form the following thermal degradation reaction:

$$R_{21}(UO_2)_9Am,(RH)_{11} \xrightarrow{+O_2,-CO_2,-CO,-H_2O} 3\,(U_3O_8) + AmO_2$$

With R standing for the ionogenic group of the resin and having a molecular weight of 75.5 g mol^{-1} [18].Experimental value of 53.2% is in fair agreement with the theoretical value of 53.0% which confirms the formulation of the loaded resin.

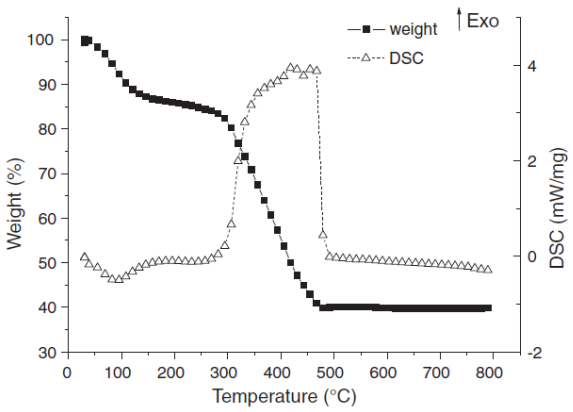

Figure 8 TGA/DSC analysis of the thermal decomposition of UO22+–Am3+ loaded resin in air.

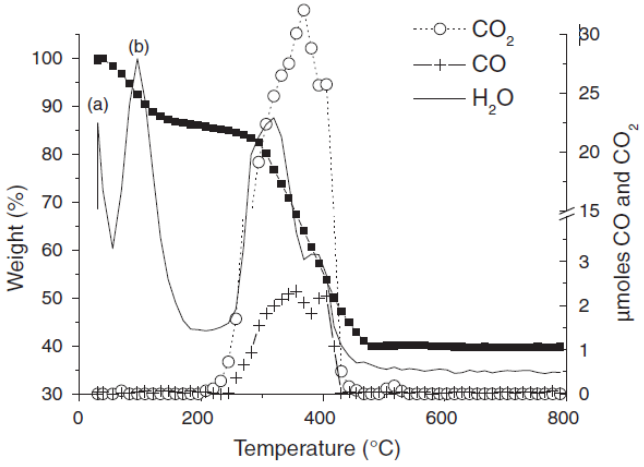

Figure 9 : TGA analysis and µlGC monitoring of H_2O, CO_2 and CO emission during the calcination of UO_2^{2+}–Am^{3+} loaded resin in air.

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2.2.2. Characterization of the oxide microspheres
Loaded resin was calcined by applying the two thermal treatments exposed previously and the reduced oxide microspheres obtained were characterized.

2.2.2.1. XRD analysis. The XRD pattern obtained from the powdered oxide microspheres is shown in Figure 10. It is characteristic of a single fluorite phase with a lattice parameter of 5.4511(2) Å (refined by Pattern Matching using the Fullprof Suite software [19]). This parameter is inferior to usual lattice parameter found for $U_{0.90}Am_{0.10}O_{2\pm\delta}$ compound (5.469 Å) [20,[21], and would indicate that the synthesized oxide is more oxidized than samples usually reported. The single-phased compound would suggest a homogeneous distribution of cations to be confirmed by WDX spectroscopy. Redox states of uranium and americium in the reduced compound have yet not been studied. From the literature on compounds prepared in similar conditions, americium is present as Am(III), while uranium is partially oxidized thus presenting a mixed U(IV)/(V) valence, with close Am(III) and U(V) contents [21–[24]. Based on the relatively low lattice parameter, a higher ratio of U(V) is expected.

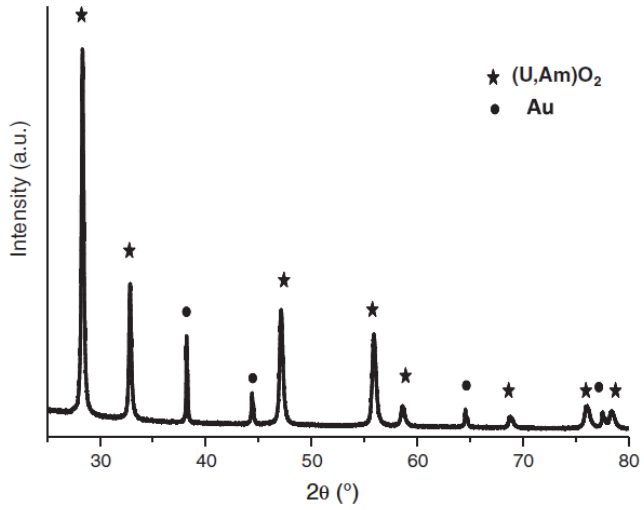

Figure 10 : XRD pattern of the oxide microsphere precursor (Au used as internal standard).

2.2.2.2. Residual carbon content. Thermogravimetric analysis (TGA–µGC) was performed in air up to 1000 °C on the oxide precursor obtained after the two thermal treatments and results are shown in Figure 11. Most CO_2 gas release was recorded between 170 and 550 °C with a maximum at 275 °C corresponding to oxidation of residual carbon. A second zone was observed in the range of 925 °C and 1000 °C but at a lower extent. Total residual carbon content measured in the reduced oxide is equal to 1500 ± 100 ppm and is close to that obtained for co-converted oxalates [9]. As the carbon content did not preclude the densification in the latter case, carbon content in microspheres should not be a problem.
Reoxidation of the oxide $U_{0.90}Am_{0.10}O_{2\pm\delta}$ was also revealed during the experiment by weight increase.

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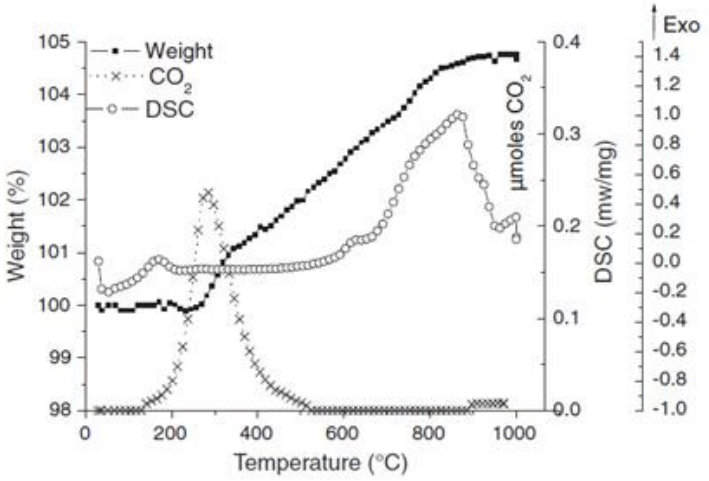

Figure 11 : TGA/DSC analysis and µGC monitoring of CO2 emission during calcination under air of reduced $U_{0.9}Am_{0.1}O_{2\pm\delta}$ microspheres.

2.2.2.3. Microstructural characteristics. SEM micrographs showing the morphology and the internal microstructure of oxide spheres are given in **Figure 12.** They prove that the spherical morphology of the resin bead is preserved during heating treatments and that the precursor microstructure is homogeneous and composed of micron-sized aggregates bounded in a large porous network. The microstructure already indicates that this material is very brittle and should be suitable for pelletization.

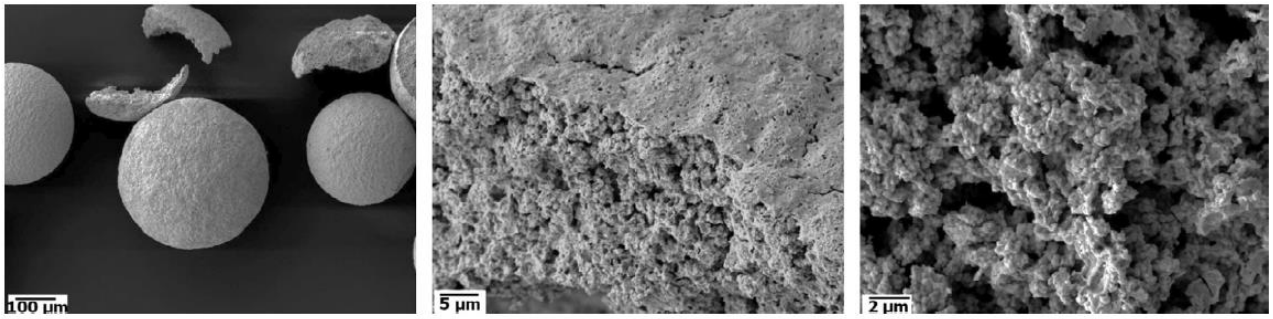

Figure 12 : FEG-SEM micrographs in secondary electron mode showing reduced oxide microsphere morphology and internal microstructure.

2.2.2.4. Density. The bulk density of oxide spheres was measured from a set of 144 microspheres. The average diameter of the spheres is 375 ± 50 µm, and the apparent density measured is of 2.7 ± 0.1 g cm^{-3}. This represents 24.0 ± 1.3% TD of the mixed oxide $U_{0.90}Am_{0.10}O_{2\pm\delta}$ calculated from the experimental refined lattice parameter obtained on reduced oxide precursors, i.e., 11.09 g cm^{-3}. This relatively low value confirms the presence of a highly porous material.

3. Conclusion and perspectives

Mixed oxide microspheres $U_{0.90}Am_{0.10}O_{2\pm\delta}$ were fabricated by thermal treatments of ion exchange resin loaded with Am^{3+} and UO_2^{2+} cations. The degradation of the polymeric skeleton under air followed by reducing heat treatment led to the synthesis of spherical precursors with diameter comprised between 325 µm and 425 µm and apparent density of (24 ± 1)% TD. A solid solution was formed during the reducing heat treatment and ensured a homogeneous distribution of uranium and americium atoms in the solid.

Fabrication of a pellet using this precursor will be attempted during the next semester.

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ACTION PLAN

After this success in the production of dense $(U,Am)O_2$ pellet with less dust generation than conventional routes, using oxalic co-converted precursors, work will now be focused on another synthesis route avoiding powder precursor generation.

TASK 7.4 CHARACTERIZATIONS

MANPOWER

Expected effort for the period 1.3 men.month

Actual effort spent during the period 1.3 men.month

MAIN PROGRESSES

(U, Th) oxalic conversion

1. Introduction

The aim of this study is to build on work that had previously been carried out by Carrigan *et al.1* and calcine the mixed uranium and thorium oxalate samples to the oxide. The original samples of UxTh1-x(C2O4)2 were produced in a range of ratios from 0:100 % Th:U to 100:0 % Th:U in increments of 10 % and precipitated at three different temperatures, 25, 40 and 60 °C. These mixed oxalates are to be calcined at 900 °C under an air or inert argon atmosphere.

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Once the oxides have been produced they will be analysed via Raman spectroscopy and X-Ray Diffraction. These techniques will enable the characterisation of the mixed oxides to investigate the behaviour of uranium when mixed with a redox inert thorium. Further experiments are planned to be undertaken to produce a mixed uranium and plutonium oxalate and thus mixed oxide. As the behaviour of uranium has been characterised with a redox inactive thorium, it will be possible to see how being mixed with a more redox active and radioactive material, plutonium, affects the behaviour of uranium along with being able to characterise plutonium in the same manner.
Three sets of calcinations have been carried out so far. The first two sets of calcinations were carried out using the 0, 50 and 100 % uranium mixed oxide samples. The first calcination batch was carried out under an air atmosphere and the second batch, using the same set of samples, was carried out under an inert argon atmosphere. The third set of calcinations looked at calcining the whole set of mixed oxalate samples precipitated at 60 °C under an argon atmosphere.
Data from Raman spectroscopy and XRD analysis has been collected from the first two sets of calcinations. All of the samples calcined under argon and air atmospheres and have been analysed via XRD. Only the 0 % uranium samples have been analysed via Raman spectroscopy. Analysis of the third set of calcinations is currently being carried out via Raman spectroscopy and XRD. The analysis of these samples is aimed to be completed by May 2018.

2. Initial Calcinations

All calcinations have been carried out using the same heating conditions and used the same set up. A Vecstar Tube Furnace Model VCTF4 has been used to heat each sample to 900 °C at a rate of 10 °C per minute. Under inert conditions, a flow of 100 mL per minute of analytical grade (N6) argon is passed through the furnace tube during the calcination. This inertion aimed to limit the potential for uranium to oxidise from UO_2 to intermediate UO_{2+x} phases and ultimately U_3O_8.

Three sets of calcinations have been carried out to date. The first set of calcinations took the 0, 50 and 100 % uranium mixed oxalate samples precipitated at 25, 40 and 60 °C and calcined them under an air atmosphere. The second batch of calcinations took the same set of samples and calcined them under an inert argon atmosphere. Finally, the third set of calcinations took the whole range of mixed oxalate samples precipitated at 60 °C and calcined them under and inert atmospheres.

After carrying out the first two sets of calcinations, the new oxide samples were weighed and the percentage mass losses are presented in Table 4. The data shows a relationship between the uranium to thorium ratio in the mixed oxalate and the percentage of mass lost when the oxalate is converted to the oxide. This trend shows a decrease in mass loss when the ratio of uranium to thorium increases. This trend was seen whether the samples were calcined under an air or an inert atmosphere. All of the calcinations for the third set of samples have been carried out but the data have not been fully analysed yet. (The excess sample loss in the U50_25 sample is due to a small spillage that occurred).

Table 4 : Percentage mass losses of each sample from the first batch of calcinations. Sample Label: U – Uranium; 100, 50, 00 – percentage of uranium to thorium; _25, _40_60 – precipitation temperature.

Sample Label	% Mass loss under Air	% Mass loss under Ar
U100_25	48.1	48.0
U50_25	48.8	55.6
U00_25	50.2	50.2
U100_40	47.9	47.1
U50_40	48.5	48.0

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U00_40	50.9	50.6
U100_60	48.0	N/A
U50_60	48.8	N/A
U00_60	51.5	N/A

3. Results

Two methods of analysis were selected to analyse the mixed oxide samples; X-Ray Diffraction (XRD) and Raman spectroscopy. Only the 0 % uranium samples from these calcination sets have been analysed via Raman spectroscopy. The reasoning behind this is the samples of 50 % and 100 % uranium were too dark to be analysed using this method as the quantity of light reflected by the sample was too small to gain any reasonable peak data. Contrary to this, all of the samples from calcination batches 1 and 2 were analysed via XRD analysis and the data has been processed and analysed.

None of the Raman spectroscopy data has been processed to date.

3.1. XRD Data

After carrying out the first two batches of calcinations, it was possible to see how the crystal structure of thorium oxide changed with the addition of uranium. Figure 13 compared the two XRD spectra of 0 and 50 % uranium mixed oxide. From the spectra it is possible to see that uranium has incorporated itself into the crystal and had an effect on the peak positions produced by the thorium oxide (0 % uranium). This shift in position can be down to the larger atomic radius of the uranium. The extra lone peaks can be characterised as ingress of U3O8 produced during the calcination.

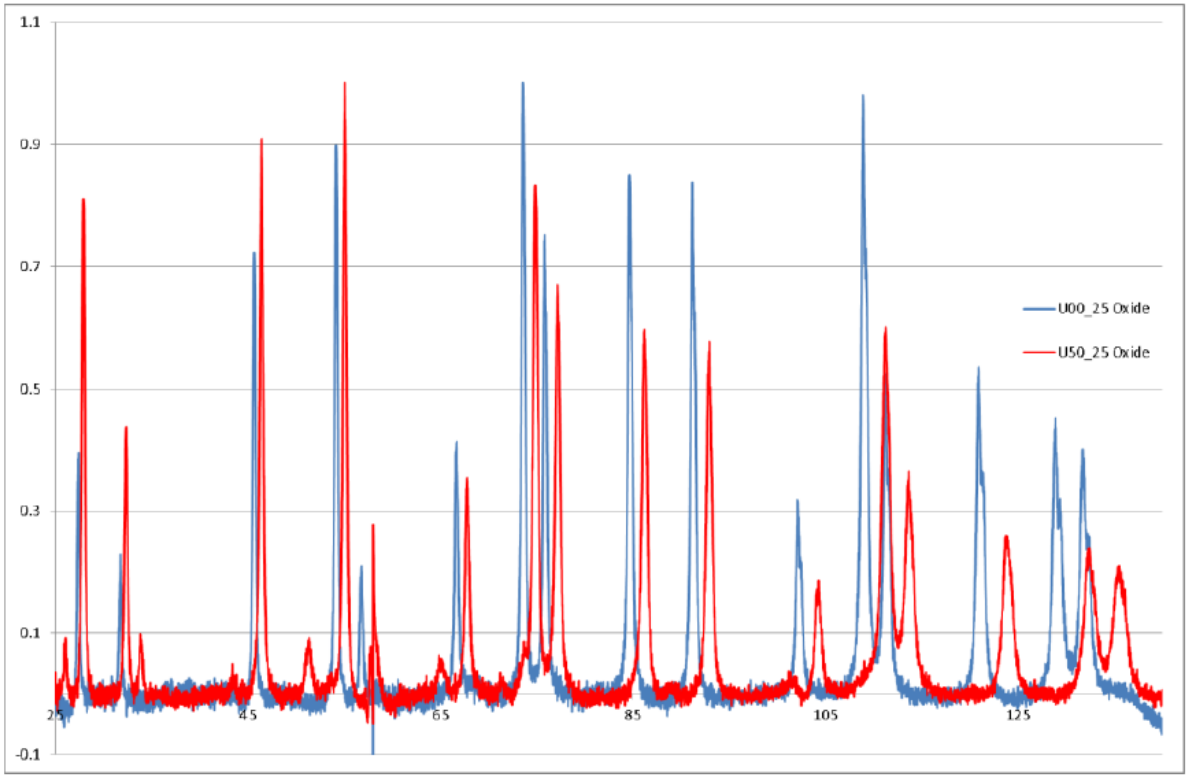

Figure 13 : XRD spectra of 0 % uranium (shown in blue) and 50 % uranium (shown in red) mixed oxide calcined in air.

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When these two spectra are compared with the 100 % uranium mixed oxide sample, it can be noted that the presence of thorium stabilises the uranium against oxidation. Figure 14 shows the comparison of all three XRD spectra of samples precipitated at 25 °C. The more disordered of the three spectra represents the 100 % uranium sample which shows an array of peaks which can be attributed to multiple phases of uranium.

Further analysis is planned to be carried out on the most recent set of calcined mixed oxide samples. As this is a fuller array of ratios, it will present a better representation of how uranium and thorium interact after being calcined to the oxide and a picture will be able to be built up around how the change in ratio of uranium to thorium affects the crystal structure of the oxide before moving on to (U,Pu) and (U,MA) co-precipitated and co-calcined oxides.

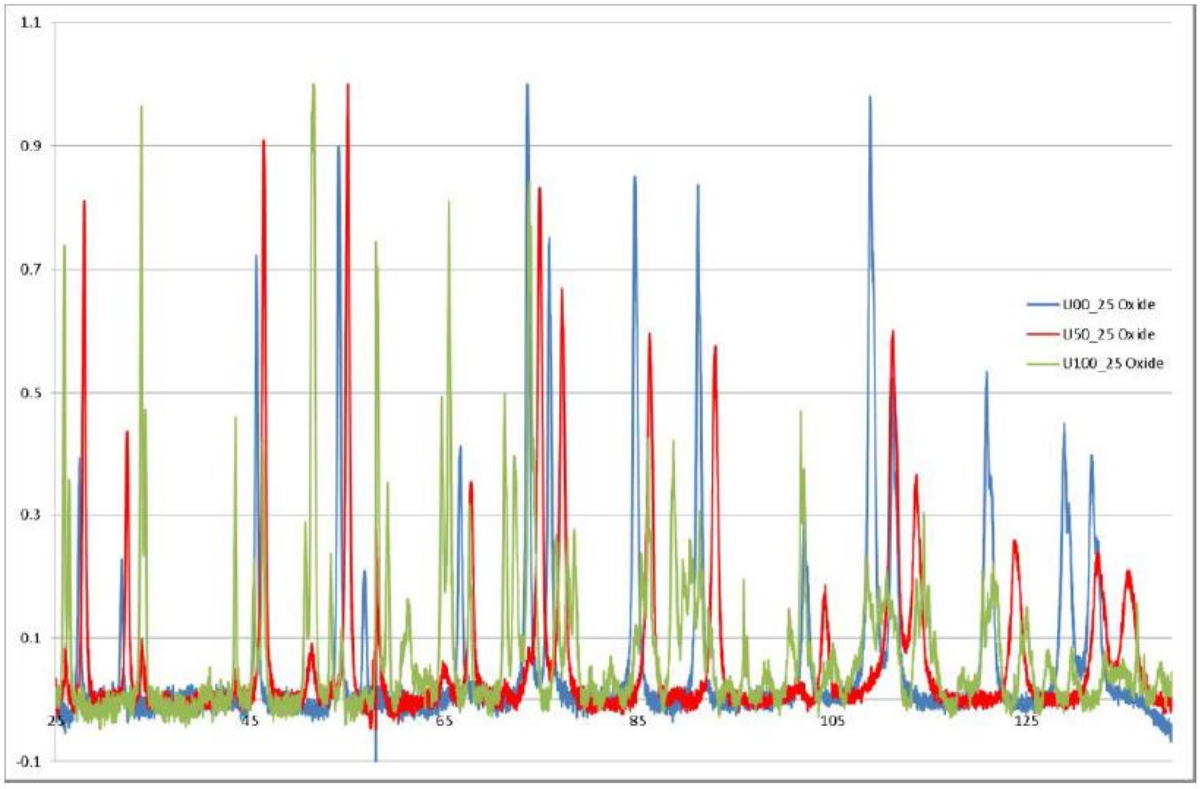

Figure 14: Comparison of XRD spectra collected from the 0 (Blue), 50 (red) and 100 % (green) uranium mixed oxide samples calcined at 25 °C.

WP8

INTRODUCTION

This WP addresses the industrialisation and scale up of some of the chemical projects studied in GENIORS. To develop processes towards industrialisation studies that consider the holistic impacts of the flowsheet are necessary. The tasks in this work package seek to asses and illustrate the holistic effects on the nuclear fuel cycle that occur from fundamental changes to

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the chemistry at the heart of its key processes. Appropriate technology deployment and consideration of potential issues and impediments to industrialisation will also be assessed.

MAIN RESULTS

TASK 8.1

MANPOWER

Total: NNL 3.5pm

MAIN PROGRESSES

Produce a concept design of an entire EURO-GANEX reprocessing plant, from receipt of spent oxide fuel through head-end process and dissolution to chemical separation and the production of oxide powders for re-use. The design pack will include a flowsheeting exercise to determine the mass flows of the main components and isotopes of interest through the process.

Progress: Flowsheet build of EURO-GANEX process from fuel receipt to powder finished.

DIFFICULTIES

2 weeks delay in submission.

ACTION PLAN

D8.1 “Process description report of concept Euro-GANEX plant” was submitted.

TASK 8.2

MANPOWER

Total: KIT 2.9pm

MAIN PROGRESSES

Undertake a study comparing the different options for solvent extraction. The comparison will not be made with respect to the TRL of the individual processes but with respect to factors such as compatibility with upstream and downstream processes, compactness (number of stages, size of contactors) and auxiliary steps (solvent clean-up etc.)

Progress: This task is finished.

ACTION PLAN

D8.2 – Report on the comparison of SX processes for heterogeneous recycling - delivered on M12.

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TASK 8.3

MANPOWER

Total: LGI 3.9pm

MAIN PROGRESSES

This task proposes a mapping of the different status and alternatives for each ESNII concept waste treatment process.

The study will undertake a comparative assessment of the different recycling needs and options considered and will identify the facilities that could integrate the reprocessing options at EU level. Building on links with existing gen IV concepts (ASTRID, ALFRED, MYRRHA) the task will evaluate the possibilities of increasing the capacities of existing EU nuclear facilities

Progress: state of the art, methodology well established, content in progress

DIFFICULTIES

None

ACTION PLAN

Report to be delivered M18

TASK 8.4

MANPOWER

Total: NNL 6.5pm

MAIN PROGRESSES

Develop a “SimPlant” model to illustrate how changes to a baseline could affect the size of the reprocessing and associated plants with which it has close interactions, e.g. waste treatment and vitrification plants. This will allow comparison of the holistic effect of chemical process changes on nuclear plants and site footprints.

Progress: Flowsheet build of EURO-GANEX process from fuel receipt to powder finishing has begun

DIFFICULTIES

None

ACTION PLAN

Report to be delivered M48

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TASK 8.5

MANPOWER

Total: LGI 3.9pm

MAIN PROGRESSES

The last task of WP8 will assess the impact of implementing a closed cycle across the full nuclear fuel cycle, from mining and fuel fabrication to spent fuel conditioning. The task will use the results from previous tasks and will organise a set of face-to-face semi structured interviews to identify the main changes, main issues, blocking points and impact on the actors involved (some will disappear; some new actors will appear from opportunities in the cycle). The stakeholders

will encompass actors from industry, nuclear associations, policy makers, research centres, etc.

Progress: no progress has been reported in this period

DIFFICULTIES

None

ACTION PLAN

Report to be delivered M36

AREVA (1pm) and EDF (1.5pm) participate in the whole WP as expert, participating to interviews and reviews of the deliverables.

WP9

INTRODUCTION

The objectives of this task are:

- To perform corrosion assessments for stainless steels SS304L & S316L in the presence of as many of the GANEX inventory permutations as resource permits.
- To perform long term corrosion studies in relevant inventory permutations.
- To conduct corrosion and plant material compatibility tests for extraction and stripping stream compositions proposed for the EXAm process.

In conjunction with electrochemical quartz crystal microbalance (QCM) experiments, linear sweep voltammetry (LSV) studies have been conducted on the corrosion of SS304L and SS316L in the presence of the 1st cycle GANEX ligand, DEHiBA.

The presence of DEHiBA in the organic phase is found to have no effect on the corrosion rate of SS304L and SS316L in Exxsol D80. The presence of DEHiBA in the organic phase when that phase is contacted with nitric acid is found to reduce the rate of SS316L in HNO_3, but has no effect on SS304L.

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Design and prototyping of a suitable experimental setup for long term corrosion studies has also been carried out.

Four steel types have been acquired for testing (SS316L, SS304L, NAG 18/10 and SS310), characterised and turned into rotating disk electrodes. A double junction arrangement suitable for long term corrosion testing has been developed with the aid of the National Nuclear Laboratory (NNL) and a prototype setup is under construction.

Based on work carried out in SACSESS, SO_3-Ph-BTP and AHA in HNO_3 has been identified as the most suitable corrosion system to initially be studied long term. Rotating disk and rotating cylinder electrodes will be manufactured and used for these tests, to provide similar conditions to those expected in storage tanks, process pipework and centrifugal contactors.

MAIN RESULTS

TASK 9.3

MANPOWER

18 months – but work to date performed using Lloyd's Register Foundation funding

MAIN PROGRESSES

N, N-di(2-ethyl hexyl) isobutyramide (DEHiBA) was the first ligand to be selected for study.
As the organic phase extraction ligand for uranium in 1st cycle extraction, it is present as 0.1 mol dm^{-3} DEHiBA in odourless kerosene in contact with 5 mol dm^{-3} HNO_3 aqueous phase.

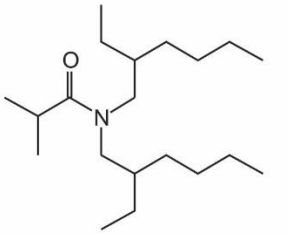

Figure 1: DEHiBA

Seven sets of linear sweep voltammetry (LSV) and electrochemical quartz crystal microbalance (QCM) experiments were conducted on each of the steels, SS316L and SS304L as follows.

- LSV and QCM experiments in 5 mol dm^{-3} HNO_3
- LSV and QCM experiments in 5 mol dm^{-3} HNO_3 contacted with Exxsol D80
- LSV and QCM experiments in 5 mol dm^{-3} HNO_3 contacted with 0.1 mol dm^{-3} DEHiBA in Exxsol D80
- LSV and QCM experiments in Exxsol D80
- LSV and QCM experiments in 0.1 mol dm^{-3} DEHiBA in Exxsol D80
- LSV and QCM experiments in Exxsol D80 contacted with 5 mol dm^{-3} HNO_3
- LSV and QCM experiments in 0.1 mol dm^{-3} DEHiBA in Exxsol D80 contacted with 5 mol dm^{-3} HNO3

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Results were as follows.

For the organic phase experiments, both LSV and QCM measurements confirm corrosion in the organic phase (Octan-1-ol and Exxsol D80) is insignificant compared to aqueous phase (HNO_3) corrosion.
DEHiBA appears to have little effect on corrosion in the organic phase, if anything acting as a slight corrosion inhibitor.
Contact of organic phase with a 5 mol dm^{-3} HNO_3 aqueous phase produces no significant increase in corrosion in the presence or absence of DEHiBA.

For the aqueous phase experiments, LSV measurements show that SS304L and SS316L are significantly corroded in 5 mol dm^{-3} HNO_3 over the potential range studied (0.3 to 1.2 V vs saturated silver chloride reference electrode).
SS316L is significantly more susceptible to transpassive dissolution than SS304L
Contact with the organic phase (Exxsol D80), inhibits transpassive corrosion of SS316L at potentials > 1V).

The open circuit corrosion rate as determined by calculation of the equilibrium corrosion current i_{corr} almost halves after contact with an exxsol organic phase.
The presence of DEHiBA in the organic phase has little effect on SS304L corrosion rates, but further reduces corrosion of SS316L over Exxsol contact alone.

Design and prototyping of a suitable experimental setup has also been carried.

To maximise data and allow for optimal materials selection for a real reprocessing setup, the number of steel types has been expanded to four for long term corrosion tests. Again SS316L and SS304L samples will be tested, but also NAG 18/10, currently utilised at THORP and supplied by NNL, and SS310, a high Cr steel suitable for 'head end' dissolution vessels. For initial testing, these materials in the form of rod or plate have been turned into 9.85 mm cylinders and mounted into epoxy resin to form rotating disk electrodes (RDE).

A full compositional analysis has been carried out using a combination of X-ray fluorescence (XRF) elements and energy dispersive X-ray analysis (EDX), the results of which are shown in Table 1:

	Weight Percentage / wt. %																
	Al	**Si**	**P**	**S**	**Ti**	**V**	**Cr**	**Mn**	**Fe**	**Ni**	**Cu**	**Se**	**Nb**	**Mo**	**W**	**Zr**	**Sn**
SS304L	0.040	0.289	0.030	0.030	0.026	0.040	18.762	0.910	70.028	8.850	0.534	0.006	0.006	0.188	0.250	0.003	0.008
SS316L	0.030	0.215	0.000	0.110	0.023	0.080	18.124	1.640	66.397	10.791	0.455	0.005	0.012	1.865	0.240	0.002	0.010
SS310	0.020	0.791	0.060	0.000	0.057	0.038	25.287	1.880	52.216	18.855	0.248	0.006	0.014	0.229	0.280	0.002	0.016
NAG	0.030	0.000	0.060	0.060	0.025	0.037	18.919	1.070	69.171	10.338	0.083	0.005	0.004	0.043	0.140	0.005	0.012

Table 1: Compositional analysis of selected steel types.

A suitable long term corrosion measurement prototype setup has been created.
The first junction consists of glass wire string soaked in the working solution of choice. This is threaded between the working chamber and a second chamber partially filled with the same solution to create a simple salt bridge.

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The second bridge consists of a glass double junction filled with NaCl saturated agar. The top of the second junction is then filled with a saturated NaCl solution and a normal Ag/AgCl reference electrode inserted.
To complete the three electrode cell a separate fritted Pt mesh counter electrode compartment and steel RDE mounted on a motor are also used. The full prototype setup is shown in Figure 2.

Figure 2: Prototype setup for long term corrosion measurements.

The use of custom small volume glassware enables the rate steel corrosion in the presence of high concentrations of reprocessing ligands to be studied either under static or flow conditions.
For the final long term studies the prototype setup shown in Figure 2 will be replicated four times, using a potentiostat MUX controller to measure each steel type in turn by potentiometry, linear resistance polarisation (LPR) and electrochemical frequency modulation (EFM).
Rotating disk electrodes (RDE's) offer excellent hydrodynamic control suitable for studies of corrosion under either static (representative of storage tanks) or laminar flow conditions (representative of stream flow in pipework). However, such electrodes do not replicate the sort of turbulent flow patterns found in centrifugal contactors (Taylor-Couette flow). Thus, time permitting, roughened rotating cylinder electrodes (RCE's) of each steel will also be created and tested. A schematic of a typical rotating cylinder electrode system is shown in Figure 3:

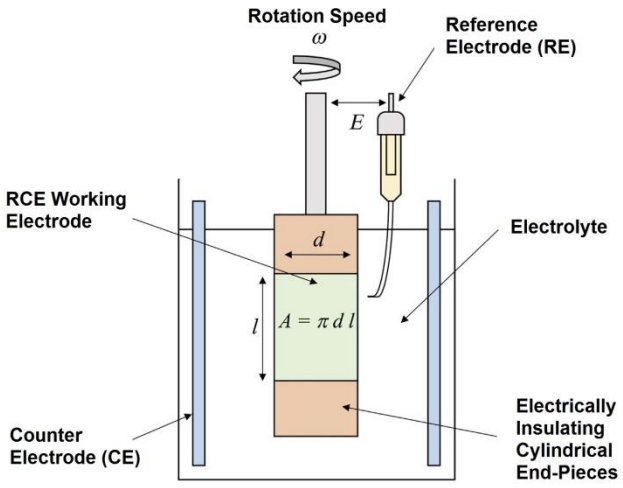

Figure 3: Schematic diagram of a rotating cylinder electrode (RCE) system [2].

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Finally, Quartz crystal microbalance and linear sweep voltammetry studies carried out in SACSESS have shown that SO_3-Ph-BTP and AHA in HNO_3 can accelerate transpassive corrosion at high enough solution potentials.
This system will be the first to be studied using the prototype setup in Figure 2 for periods of one week at a time, testing both the effect of flow and the effectiveness of the double salt bridge arrangement. Further tests with the completed four cell long term corrosion rig will then be performed over time periods of 6 months to a year. It should be noted that this activity will run simultaneously with those being carried out in objective 1, corrosion tests of GANEX inventory permutations, therefore, should another corrosion vulnerability be identified this will also be tested using the long term corrosion setup.

ACTION PLAN

In the immediate term, electrochemical impedance spectroscopy (EIS) measurements will be conducted on SS304L and SS316L in 5 mol dm^{-3} HNO_3 contacted with DEHiBA to determine if the formation of an adsorbed layer of DEHiBA at the steel surface is the cause of surface corrosion inhibition. This will be followed by a program of experiments, similar to those described above looking at TODGA (aqueous and organic), CDTA/hydrazine (aqueous, varying nitric acid concentrations) and DMDOHEMA (aqueous and organic).

In parallel with these studies will be testing of the prototype long term corrosion setup with SO_3-Ph-BTP and AHA described above.

In the longer term as well as construction of the four cell long term corrosion rig (and associated experimental series) corrosion experiments under radiation flux or in the presence of deliberately added radiolysis products (e.g. H_2O_2) are also planned.

WP12

INTRODUCTION

This work package covers Training and Education and Knowledge Management. Work on the tasks has proceeded as planned.

MAIN RESULTS

Task by task, highlight the main progresses, delays, difficulties with analysis comments, action plan if needed. Give information and short analysis on manpower (expected, realized, explanation on discrepancies)

TASK 421

MANPOWER

ULEEDS: 0.25 pm

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MAIN PROGRESSES

Two travel awards – totalling – €1,982 - have been made.

TASK 422

MANPOWER

ULEEDS: inc in above

MAIN PROGRESSES

The technical challenge for the Think Tank has been selected – "Evaluation of the safety and technical issues behind the implementation of a reprocessing facility using the Euro-Ganex process" – and organisation of the event is in hand – see attached appendix.

ACTION PLAN

The Think Tank has been arranged for 23rd/24th October. it will be a run jointly by UnivLeeds and NNL.

TASK 423

MANPOWER

ULEEDS: inc in above

MAIN PROGRESSES

A draft syllabus has been agreed. The Digital Education Service at UnivLeeds has been engaged to produce the course. A kick off meeting is arranged for 19th September.

ACTION PLAN

A project plan will be produced after the kick off meeting on the 19th September.

WP13

INTRODUCTION

WP13 aims to engage with stakeholders and raise awareness on GENIORS and its achievements through specific communication actions. It will also ensure the dissemination of knowledge acquired towards the scientific community and other relevant stakeholders, including publications in scientific journals and participation in conferences.

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MAIN RESULTS

TASK 13.1 – COMMUNICATION TOOLKIT

MANPOWER

LGI was the only contributor to this task during this reporting period: 0.3 PM

MAIN PROGRESSES

The second elements of the communication toolkit were designed and distributed to all GENIORS partners to promote the project: this included the project flyer and the project roll-up.

ACTION PLAN

The project roll-up and flyers had been used for the 2nd Project Meeting, taking place in Würzburg (Germany), from 16-18 April 2018. Moreover, they will be used for other upcoming events in order to increase the project's visibility: some flyers had been distributed to partners who will attend events, conference and/or workshops.

TASK 13.2 – COMMUNICATION & DISSEMINATION PLAN

MANPOWER

LGI was the only contributor to this task during this reporting period: 0 PM

MAIN PROGRESSES

The Communication and Dissemination Action Plan deliverable D13.2 was submitted. It includes:

- Communication strategy: definition of audiences and key messages
- A detailed planning of all communication actions over the project duration according to the defined target audiences, including press releases
- An event and publications management plan

Task 13.2 will also disseminate the results of the GENIORS project to the scientific community and stakeholders by:

- Coordinating scientific publications, including open access journals, free (online) journals, and online repositories.
- Coordinating the participation of partners in conferences to disseminate knowledge and results.

As we are early in the project, no results have yet been disseminated.

ACTION PLAN

The action plan will be implemented and monitored during the course of the project in order to identify areas of improvement and measure its success.

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TASK 13.3 – ANNUAL NEWSLETTERS

MANPOWER

LGI was the only contributor to this task during this reporting period: 0.2 PM

MAIN PROGRESSES

The first electronic project newsletter was designed (and distributed in July 2018) to inform stakeholders of the project's progress. It included:

- An interview of the project coordinator
- A recap on the 2nd project meeting in Würzburg
- Social media recap: reminder to follow GENIORS on Twitter (@Geniors_H2020) + the public website (www.geniors.eu)
- A short blurb on the GENIORS flyer available to download
- Past events attended by the GENIORS partners (Juelich contribution)
- Relevant news and highlights per WP (KIT and CIEMAT contributions)

The newsletter is available here: www.geniors.eu/resources

ACTION PLAN

Attracting more people to subscribe to receive the future GENIORS newsletters via Twitter and the project website.

TASK 13.4 – PROJECT WEBSITE

MANPOWER

LGI was the only contributor to this task during this reporting period: 0.5 PM

MAIN PROGRESSES

A website was designed and released in September 2017. It is regularly maintained and updated (deliverables for download, news and upcoming events…). In addition, the online audience is monthly measured through Google Analytics.

DIFFICULTIES

Traffic to the website is still limited. It is important to have news/updates from the various work packages shared with WP13 in order to update the website and make it a lively platform.

ACTION PLAN

The website will be maintained and continuously updated.

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LIST OF PUBLICATIONS

Publications :

1. Colin R. Gregson, Gregory P. Horne, Robin M. Orr, Simon M. Pimblott, Howard E. Sims, Robin J. Taylor, Kevin J. Webb, *Molecular Hydrogen Yields from the Self-Radiolysis of Plutonium or Americium Irradiated Nitrate Solutions*, J. Phys. Chem. B, 122 (9), pp 2627–2634 (2018).
2. Wilden, Andreas, Mincher, Bruce J., Mezyk, Stephen P., Twight, Liam, Rosciolo-Johnson, Kristyn M., Zarzana, Christopher A., Case, Mary E., Hupert, Michelle, Stärk, Andrea, Modolo, Giuseppe. *Radiolytic and Hydrolytic Degradation of the Hydrophilic Diglycolamides*, Solvent Extr. Ion Exch., submitted.
3. J. Halleröd, Ch. Ekberg, T. Authen, L. Bertolo, Mu Lin, B. Grüner, J. Švehla, Ch. Wagner, A. Geist, P. Panak, and E. Aneheim. *On the basic extraction properties of a phenyl trifluoromethyl sulfone based GANEX system containing CyMe4-BTBP and TBP*. Solv. Extr. , Ion Exch., submitted.
4. J. Narbutt, I. Herdzik-Koniecko, M. Rejnis-Strzelak, "The use of synergistic effects of lipophilic modifiers of TODGA-containing organic phase for improving separation of americium(III) from the early lanthanides in the process of stripping the metals with hydrophilic ligands. A novel concept for solving the problem" (in Polish), Raport IChTJ, Seria B nr 2/2017, Warszawa 2017, 14 pp.
5. Daniel Whittaker, Andreas Geist, Giuseppe Modolo, Robin Taylor, Mark Sarsfield, Andreas Wilden, Applications of Diglycolamide Based Solvent Extraction Processes in Spent Nuclear Fuel Reprocessing, Part 1: TODGA, accepted, Solv. Extr. Ion Exch. (2018).

Publication submitted:

6. *Mossini, E. et al. Optimization and single-stage centrifugal contactor experiments with the novel hydrophilic complexant PyTri-Diol for the i-SANEX process, Solvent Extr. Ion Exch., submitted*
7. **Synthesis of size-controlled UO_2 microspheres from the hydrothermal conversion of U(IV) aspartate,** J. Maynadié, V. Trillaud, J. Manaud, J. Hidalgo, D. Meyer, R. Podor, N. Dacheux, N. Clavier**,** *Cryst Eng. Com.*, 2018 / *Submitted - Under Review*.
8. R. Malmbeck et al., J. Radioanal. Nucl. Chem, Modified diglycolamides for grouped actinide separation, 2017, in press.
9. Halleröd, J., Ekberg, C., Authen, T., Bertolo, L., Lin, M., Grüner, B., Svehla, J., Geist, A., Panak, P., Wagner, C. and Aneheim, E.: On the basic extraction properties of a phenyl trifluoromethyl sulfone based GANEX system containing CyMe4-BTBP and TBP. Submitted to Solvent extraction and Ion exchange.
10. Eros Mossini, Elena Macerata, Andreas Wilden, Peter Kaufholz, Giuseppe Modolo, Nicolò Iotti, Alessandro Casnati, Andreas Geist, Mario Mariani, Optimisation and single-stage centrifugal contactor experiments with the novel hydrophilic complexant PyTri-Diol for the i-SANEX process, submitted to Solvent Extraction and Ion Exchange.

Oral communications:

1. A. Wilden, et al. *Radiolytic Degradation of the Hydrophilic Diglycolamides*, 11th International Conference on Methods and Applications of Radioanalytical Chemistry (MARC XI), April 8-13, 2018, Kailua-Kona, Hawaii, USA.
2. A. Kimberlin, "*Investigation of TODGA radiolysis by combining experimental and computational approaches*". Second edition of Radical Behaviour workshop (RB2018), 19-20 April 2018, Würzburg, Germany.
3. L. Berthon "*Investigation of Pu(IV) – N,N-dialkylamide complexes in solution under ionizing radiation*" Second edition of Radical Behaviour workshop (RB2018), 19-20 April 2018, Würzburg, Germany.
4. M.C. Charbonnel, "*Importance of stability studies in the development of a new solvent extraction process for the multi-recycling of uranium and plutonium from spent nuclear fuels*". Second edition of Radical Behaviour workshop (RB2018), 19-20 April 2018, Würzburg, Germany.

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5. A. Wilden, "*Radiolytic Degradation of the Hydrophilic Diglycolamides*". Second edition of Radical Behaviour workshop (RB2018), 19-20 April 2018, Würzburg, Germany.
6. E. Macerata, "*A review of ageing, hydrolysis and radiolysis effects on PyTri-based stripping solvents for i-SANEX/GANEX processes*". Second edition of Radical Behaviour workshop (RB2018), 19-20 April 2018, Würzburg, Germany.
7. D. Whittaker, "*Tracking hydrogen formation in static vessels containing solutions relevant to the i-SANEX process under gamma irradiation*". Second edition of Radical Behaviour workshop (RB2018), 19-20 April 2018, Würzburg, Germany.
8. Robin Orr, "*Modelling the long-term radiation chemistry of nitrate and nitric acid solutions*". Second edition of Radical Behaviour workshop (RB2018), 19-20 April 2018, Würzburg, Germany.
9. **Caractérisation *in situ* par MEBE-HT et MET-HT du premier stade du frittage de UO_2,** V. Trillaud, R. Podor, C. Ricolleau, N. Dacheux, N. Clavier**,** Journées de la division Chimie du Solide de la SCF, Montpellier, 8-10 november 2017 / *Given*.
10. **Synthèse directe par voie hydrothermale d'oxydes d'uranium hydratés UO_2, nH_2O,** J. Manaud, J. Maynadié, D. Meyer, N. Dacheux, N. Clavier**,** JECRRC, Strasbourg, 28 may – 1 june 2018 / *Given*.
11. **Caractérisation in situ par MEBE-HT et MET-HT du premier stade du frittage de UO_2**, V. Trillaud, R. Podor, C. Ricolleau, N. Dacheux, N. Clavier, Matériaux 2018, Strasbourg, 19-23 november 2018 / *Accepted.*
12. **Wet chemistry route to uranium oxide microspheres as reference materials for nuclear safeguards,** N. Clavier, J. Maynadié, V. Trillaud, J. Manaud, L. Sangély, T. Tranpaphan, N. Dacheux**,** MRS Fall Meeting 2018, Boston / *Accepted.*

CONCLUSIONS

In WP1, since the start of the GENIORS project only little R&D related to the fission product chemistry was carried out. It is expected that the planned work will gain momentum in the next semester. CEA and JUELICH are planning to speed up their planned work.

After first year of GENIORS project it can be reported a more than optimal development of WP2 activities. Particularly, during this second semester 27.58 pm has been reported over the 13.4 pm estimated average, mainly due to the effort dedicated to Task1 Radiolysis & degradation products (19.58 pm). Activities regarding gas generation and modelling (Task 3 and 4 respectively) continue as expected and activities related to destruction of organics (Task 2) has recently started (2.5 pm).

In WP3, Some small difficulties, which can be overcome have been identified but most of the actions in this workpackage have started as planned. After one year, 35.85 pm have been realized within WP3, corresponding to 24.4 % of the 147.1 pm planned for the complete project.

In WP4, several ways of preparation of precursors are now under progress. They will allow to provide a large variety of materials, with various compositions and microstructures. This will allow to developed multiparametric dissolution experiments on the prepared samples (planned to begin soon). Only few difficulties encountered by the partners for both tasks: T4.1 and T4.2.

In WP5, A round ruby study of the different irradiation facilities was started. Data has been collected and subsequent meetings are to be held at the 2018 GENIORS autumn meeting.

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In WP6, The work package concerns flowsheet development of key processes for homogeneous and heterogeneous recycling so it is expected that most studies will commence later in the project. No significant delays or causes for concern were raised by the partners in the HYPARs.

In fact, despite the low levels of manpower dedicated to WP6 in semester 1-2 there has been already one significant development – the promising results obtained by JRC on the modified DGA ligand that may be a candidate to replace the TODGA+DMDOHEMA combination in the EURO-GANEX process. A journal paper is already published on this subject. In the last semester good progress has been made on both the CHALMEX and AMSEL flowsheet development.

In WP7, as planned, the first dissolution data on model compounds were obtained during this semester. For the conversion work, dust free MABB precursors were synthetized using WAR process. Concerning the characterization work, as a pre-cursor to studies with plutonium and minor actinides, a range of (U,Th) mixed oxalate precipitates were calcined to mixed oxides under air and inert atmospheres. Raman and powder X-ray diffraction analysis are ongoing.

In WP8, The deliverable D8.3 is well advanced in terms of methodology, main sections and content. Firstly, the work focuses on the state of the art of ASTRID, ALFRED and MYRRHA. It is followed by a description of the system's cycles, possible waste treatment strategies and the proposition and description of three waste treatment scenarios. There have been several interactions with partners and external experts to validate the work and provide relevant information. A meeting with the partners from CEA, ORANO and EDF was held on June at LGI offices in Paris. The objective of the meeting was to agree on the focus of the deliverable, content and structure to follow. A follow-up meeting was planned in August to inform the partners about the advancement of the deliverable.

In WP9, the presence of DEHiBA in the organic phase is found to have no effect on the corrosion rate of SS304L and SS316L in Exxsol D80. The presence of DEHiBA in the organic phase when that phase is contacted with nitric acid is found to reduce the rate of SS316L in HNO_3, but has no effect on SS304L.

Design and prototyping of a suitable experimental setup for long term corrosion studies has also been carried out.

Four steel types have been acquired for testing (SS316L, SS304L, NAG 18/10 and SS310), characterised and turned into rotating disk electrodes. A double junction arrangement suitable for long term corrosion testing has been developed with the aid of the National Nuclear Laboratory (NNL) and a prototype setup is under construction.

Based on work carried out in SACSESS, SO_3-Ph-BTP and AHA in HNO_3 has been identified as the most suitable corrosion system to initially be studied long term. Rotating disk and rotating cylinder electrodes will be manufactured and used for these tests, to provide similar conditions to those expected in storage tanks, process pipework and centrifugal contactors.

In WP10, no activity in period 2. A cluster meeting is planned on period 3

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In WP12, Two awards for travel bursaries have been made totaling €1,982. This brings the total awarded to date to €8,762 leaving €11,238 in the fund. The operational process for the Think Tank has been developed and agreed. There has been an initial meeting to identify existing examples of online training packages. Delivery platform options have been identified.

WP13 is on track. However, the challenges that lie ahead are mainly the creation of content in order to populate the website, the GENIORS Twitter account and the future project newsletters. It is important for work package leaders to share updates in their WPs with the communications team in order to have information on which to communicate.

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ANNEXES

DOMAIN	1	WP	1	WP Leader	G. Modolo
Manpower dedicated to this WP on the period				3.45	
Contribution to Deliverables (number and title of each deliverable)					
D1.1 : Report on the speciation and extraction of key fission products [due month 36]					
Contribution to Milestones (number and title of each milestone)					
MS1 : provide information on WP activity for the Work-Package Activity Summary Report 1 (month 7) MS2 : provide information on WP activity for the Work-Package Activity Summary Report 2 (done)					
Main achievements - Progress					
Task 11 Problematic fission products - extraction chemistry Distribution data for the extraction of Sr(II) into TODGA solvents (containing 1-octanol or TBP as modifier) were determined as a function of HNO3 and TODGA concentrations. An attempt at establishing an equilibrium model for Sr(II) extraction into TODGA was made. Solvent extraction of technetium(VII) was studied in the two-phase system: TODGA in kerosene vs. aqueous HNO_3 solutions.					
Main difficulties - Delays					
Partners CEA, ICHTJ, Juelich, KIT, Twente and UREAD are involved within WP 1. However, only small amount of work was reported with the first 2 semesters. CEA will start this work on month 12. Juelich is planning to initiate the work on month 11. A technician was hired for this WP. Task 1.2 Optimization of scrubbing steps during co-extraction. Not yet started.					

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DOMAIN	1	WP	2	WP Leader	Hitos Galán
Manpower dedicated to this WP on the period				27.58	

Contribution to Deliverables (number and title of each deliverable)

D2.1 Stability and safety studies of hold extraction systems (1 for each CyMe4BTBP, HidroBTBP, TODGA & PTD) (due m39, IIC).
D2.2 Stability studies of stripping agents (due m24, UNIPR).
D2.3 Destruction of organic (due m30, CEA).
D2.4 Impacts on process safety of gas generation (due m32, POLIMI).
D2.5 Molecular modelling of particular degradation mechanism of extracting and complexing agents (due m36, CTU).

Contribution to Milestones (number and title of each milestone)

MS1: provide information on WP activity for the Work-Package Activity Summary Report 1. CEA (due m7)

Main achievements - Progress

Task 2.1 Radiolysis & degradation products (19.58 mp)

- Study about differences between in-situ alpha (provided by the decay of ^{241}Am) and external gamma radiolysis (by a ^{60}Co source) on TODGA degradation. Study of metal complexation by TODGA and some of DCs (CEA, 8.4 pm).
- Stability study of aqueous phases: Setting up of HPLC-MS analysis conditions of SO_3PhBTP degraded samples. CDTA solvent behaviour after gamma irradiation: FP and Ln extraction (CIEMAT, 1.5 pm).
- More studies about CyMe4-BTBP and CyMe4-BTPhen using the new fluorinated diluents BK-1 (CTU, 1.5 pm).
- Analysis of the degradation of a new series of CyMe4-BTBP irradiated samples using different approved solvents (FS-13, FS-13+TBP, octanol+TBP and octanol) and under different conditions (with and without contact with 4 mol/L HNO_3). (IIC, 2.43 pm).
- Stability studies of hydrophilic diglycolamides was finished; a paper was written and submitted to Solvent Extr. Ion Exch. (Julich, 1 pm).
- Irradiation experiments on concentrated PyTri-Diol (PTD) solutions to identify and separate their main radiolytic degradation products (POLIMI, 2 pm).
- A new batch of PyTriDiol was produced to feed the studies carried out by other partners (UNIPR, 2 pm-synthesis).
- Stability study of SO_3PhBTBP again nitric acid and alpha radiolysis (INE-KIT, 0.75 pm).

Task 2.2 Destruction of organics (2.5 mp)

- First stability studies of TODGA at high acidity and high temperature. (CEA, 1.5 pm), result not reported yet.
- Literature search for the implementation of a method to study the safety of PTD and of the chemicals for its production. Differential Scanning Calorimetry (DSC) and Accelerating Rate Calorimetry (ARC) as interesting and informative techniques to assess preliminary hazard level of organic azides and ligands of interest such as PTD. (UNIPR, 1 pm).

Task 2.3 Gas generation (4.2 mp)

- Irradiations of relevant solvents for the i-SANEX and GANEX at Dalton Cumbria Facility using pelletron to simulate alpha degradation. Hydrogen generation measured and TODGA performance post-irradiation characterised. (NNL, 2.7 ppm)
- Gamma irradiation experiments on the PTD-TODGA extracting system under different experimental conditions (with and without pre-equilibration and degasification). (POLIMI, 1.5 pm).

Task 2.4 Radiolysis modelling (1.3 mp)

- Additional theoretical study of CyMe4-BTBP degradation products. All studies performed until now point out to the prevailing degradation product found in solution was 1-octanol adduct on meta position of pyridine ring of CyMe4-BTBP. (CTU, 1.3 mp).

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Main difficulties - Delays
No major difficulties have been reported. Although there was some delays due to: - Time scale for the hiring a PhD student working on the project (UNIPR). - No degradation products/adducts of CyMe4-BTBP and CyMe4-BTPhen synthesized, yet (CTU). - Availability of ^{60}Co-sources at Náyade facility (CIEMAT). - Delays waiting for time on pelletron at DCF (NNL).
Scientific publications, patents (beneficiary name and type of publication)
Journal: 1 accepted and 2 submitted (1 NNL, 1 Julich submitted and 1 IIC-Chalmers-INE submitted). Int. Conference Oral: 8 (3 CEA, 1 UNIPR, 2 NNL, 2 Julich). Corresponding list is given at the end of the document.
Effective collaboration between Beneficiaries (put crosses in boxes)

	CEA	CHALMERS	CIEMAT	CNRS	CTU	ICHTJ	IIC	IRSN	JRC-ITU	JUELICH	KIT	LGI	NNL	POLIMI	SCK-CEN	TWENTE	UEDIN	UNIMAN	UNIPR	UNIVLEEDS	UREAD	ULANC	EDF	AREVA
CEA														■					■					
CHALMERS							■																	
CIEMAT													■											
CNRS																								
CTU							■			■											■			
ICHTJ																								
IIC		■			■																			
IRSN																								
JRC-ITU																								
JUELICH					■																			
KIT																								
LGI																								
NNL			■																					
POLIMI	■																		■					
SCK-CEN																								
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UNIPR	■													■										
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DOMAIN	1	WP	3	WP Leader	P. Guilbaud
Manpower dedicated to this WP on the period				19.45	
Contribution to Deliverables (number and title of each deliverable)					
D3.1 : Status on the PyTri-Diol properties [due m36] *D3.2 : Status on Distribution data and chemical modelling [due m40]* *D3.3 : Status on TODGA organic phase loading [due m36]* *D3.4 : Status on solvent clean-up & recycling [due m40]*					
Contribution to Milestones (number and title of each milestone)					
MS1 : provide information on WP activity for the Work-Package Activity Summary Report 1 (done) *MS2 : provide information on WP activity for the Work-Package Activity Summary Report 2 (done)*					

Main achievements - Progress

Task 3.1 Optimisation of systems

Studies on hydrophilic complexing agents such as SO_3-Ph-BTP or Pi-Tri-Diol (PTD) have been performed, in the extension of what has already been done in the 1st semester of the project.

Additional studies have been started on new organic phase modifiers for the processes using TODGA extractants.

Task 3.2 Distribution data and chemical modelling

Equilibrium modelling based on TRLFS results was done for the extraction of Am(III) into DMDOHEMA.

Task 3.3 Physico-chemical & loading

- Am(III) and Ln(III) loading experiments were performed for :
  - TODGA extracting systems in presence of previously identified degradation products
  - The AmSel system (SO3-Ph-BTP/TODGA)
- Loading of aqueous phases containing Py-Tri-Diol as a selective complexing agent were started in collaboration between POLIMI, UNIPR and CEA
- Structural characterization of TODGA organic phases coupling experiments with molecular dynamics simulations have been started for TODGA alone in an aliphatic diluent, TODGA + 5%vol octanol, and TODGA + DMDOHEMA

Task 3.4 Solvent clean-up & recycle

- Radiation experiments have been performed by NNL at Dalton Cumbria Facility using pelletron to simulate alpha degradation of i-SANEX solvent.
- The evaluation of the technical feasibility of the cleanup and recycling of used PTD solutions has been started

Main difficulties – Delays

Task 3.1 Optimisation of systems

- delay in the studies on the Pu complexation with PTD due to technical problems at CEA laboratories
- Difficulties on the time scale for the hiring a PhD student working at UNIPR on the project.

Task 3.4 Solvent clean-up & recycle

- TODGA experiments: insufficient dose to degrade sufficient solvent for solvent clean up experiments and a flowsheet test. May necessitate new irradiation or spiking with artificially generated degradation products.

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DOMAIN	1	WP	4	WP Leader	CNRS
Manpower dedicated to this WP on the period					

Contribution to Deliverables (number and title of each deliverable)

No deliverable during the period.

4 deliverables in WP4:

D4.1 Understanding the evolution of an interface during dissolution of actinide oxide materials by macro/microscopic approaches combination [**month 40**] – **CNRS**

D4.2 Innovative dissolution routes for highly plutonium doped $(U,Pu)O_2$ and $(U,Pu,MA)O_2$ samples [**month 45**] – **CEA**

D4.3 Studies of non-powder routes for the synthesis of MOX fuels materials, potentially bearing minor actinides and blanket fuel materials [**month 36**] – **SCK.CEN**

D4.4 Impact of the precursor "history" (nature, structural, microstructural and morphological parameters) during the conversion and sintering to actinide based dioxide materials [**month 45**] – **CNRS**

Contribution to Milestones (number and title of each milestone)

MS2 : provide information on WP activity for the Work-Package Activity summary Report 2

Main achievements - Progress

ICSM/ICSM: The procedure for the recruitment of an engineer working on tasks T4.1 and T4.2 is now finished. Morgan ZUNINO, who has been selected in mid april, has begun his work on June, 18th and will follow the training session for working on/handling radioactive materials (uranium or thorium based).

UNIMAN: To fill the PDRA post to take on the work of preparing solid solutions or uranium oxide and either plutonium oxide or other f element oxides, the position was advertised and interviews held in March 2018. A potential candidate has been identified. He already made an offer. The projected starting date is 1. May 2018. Moreover, the lab work will commence following safety training and induction of the new PDRA. A master student for the coming academic year will be allocated to assist in the lab at no extra cost for the project.

T41 Study of the solid/liquid interfaces during dissolution [1-48] – Task Leader : CNRS

Multiparametric study of the dissolution of $(U,Ce)O_2$ and $(U,Ln)O_{2-x}$ solid solutions – CNRS/ICSM

In order to develop the multi-parametric dissolution tests, the preparation then characterization of a large defined panel of uranium-lanthanide solid solutions has begun in month 6. The first set of data regarding dissolution tests on $(U,Ce)O_2$ are expected by the end of august.

Innovative dissolution routes for highly plutonium doped $(U,Pu)O_2$ and $(U,Pu,MA)O_2$ samples – CEA

The study of the solid/liquid interfaces during dissolution has begun through the synthesis of CeO_2 and $Ce_{0.8}Gd_{0.2}O_{1.9}$ samples using oxalic route and calcined at different temperatures to obtain samples exhibiting different levels of crystal defects and morphological parameters. A Netzsch micro-series milling device has been acquired to study the milling/dissolution couplings.

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T42 Study of the solid/liquid interface during conversion [1-48] – Task leader : SCK.CEN

Studies of non-powder routes for the synthesis of MOX fuels materials, potentially bearing minor actinides and blanket fuel materials – SCK.CEN

The thermal behavior of the U/Ce particles was studied via TG-DSC and the gelled spheres were calcined then sintered. For compositions containing less than 20% of cerium, the spherical shape was maintained during the thermal treatment. The products were analyzed using powder XRD and a fluorite type structure was found. A linearly decrease of lattice parameter with increasing Ce content up to 20% was observed.

Hydrothermal precipitation of uranium oxides for simplified fuel fabrication – CNRS/ICSM

The direct precipitation of uranium oxides has been undertaken under hydrothermal conditions through the *in situ* conversion of uranium oxalate. Below 180°C, the usual structure of $U(C_2O_4)_2.2H_2O$ was observed whereas $UO_{2+x}.nH_2O$ was prepared above this temperature. SEM observations of the resulting samples further revealed the loss of the classical square platelet shape of the oxalate, while the evaluation of the residual carbon content led to values significantly lower than that usually obtained through thermal conversion. Additionally, variation of the pH allowed to optimize the uranium precipitation yield.

Innovative precursors for morphology-controlled UO_2 and $(U,Ln)O_2$ oxides – CNRS/ICSM

$UO_2.nH_2O$ microspheres were obtained through hydrolysis of uranium(IV) in presence of aspartic acid under hydrothermal conditions (160°C). A multi-parametric study involving time, temperature, concentration of the reactants was then undertaken. It showed that a 50% excess in aspartic acid led to monodisperse powders. The addition of mechanical stirring during the hydrothermal process allowed to accurately control the average size of the particles produced (200 – 1200 nm range). For all the conditions tested, the characterization of the powders showed the formation of fluorite type $UO_2.nH_2O$ samples with traces of residual organics at the surface. Both water and residual organics were eliminated by heating at 600°C. Preliminary sintering tests are now under progress on such precursors.

Conversion of uranium nitrate into uranium oxide by Solution Combustion Synthesis –CNRS/ICSM

Actinide surrogate systems $GdNO_3$/fuel (fuel = glycine, urea, citric acid) were investigated to propose an experimental protocol for the conversion of uranium nitrate into uranium oxide by Solution Combustion Synthesis (SCS). The fuel over gadolinium nitrate ratio appeared as a critical parameter during the conversion and may induce phase transitions for the final product. The highest temperature of conversion was obtained for a ratio of 1 leading to the formation of monoclinic form of Gd_2O_3. For higher ratios, the conversion temperature was lowered and the cubic form of Gd_2O_3 was observed. The end-products exhibited interesting characteristic for sintering (specific surface area typically of 10-20 m^2/g). Preliminary studies were performed on the conversion of uranyl nitrate into uranium oxide.

Main difficulties - Delays

Studies of non-powder routes for the synthesis of MOX fuels materials, potentially bearing minor actinides and blanket fuel materials – SCK.CEN

The thermal treatment has to be improved to prevent the fragmentation of spherical particles with Ce contents higher than 20%

Scientific publications, patents (beneficiary name and type of publication)

Synthesis of size-controlled UO_2 microspheres from the hydrothermal conversion of U(IV) aspartate, J. Maynadié, V. Trillaud, J. Manaud, J. Hidalgo, D. Meyer, R. Podor, N. Dacheux, N. Clavier**,** Submitted in *Cryst Eng. Com.*, 2018 / *Under Review*.

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Int. Conference Oral: Yes

Caractérisation *in situ* par MEBE-HT et MET-HT du premier stade du frittage de UO_2, V. Trillaud, R. Podor, C. Ricolleau, N. Dacheux, N. Clavier**,** Journées de la division Chimie du Solide de la SCF, Montpellier, 8-10 november 2017 / *Given*.

Synthèse directe par voie hydrothermale d'oxydes d'uranium hydratés UO_2, nH_2O, J. Manaud, J. Maynadié, D. Meyer, N. Dacheux, N. Clavier**,** JECRRC, Strasbourg, 28 may – 1 june 2018 / *Given*.

Caractérisation in situ par MEBE-HT et MET-HT du premier stade du frittage de UO_2, V. Trillaud, R. Podor, C. Ricolleau, N. Dacheux, N. Clavier, Matériaux 2018, Strasbourg, 19-23 november 2018 / *Accepted.*

Wet chemistry route to uranium oxide microspheres as reference materials for nuclear safeguards, N. Clavier, J. Maynadié, V. Trillaud, J. Manaud, L. Sangély, T. Tranpaphan, N. Dacheux**,** MRS Fall Meeting 2018, Boston / *Accepted.*

Effective collaboration between Beneficiaries (put crosses in boxes)

	CEA	CHALMERS	CIEMAT	CNRS	CTU	ICHTJ	IIC	IRSN	JRC-ITU	JUELICH	KIT	LGI	NNL	POLIMI	SCK-CEN	TWENTE	UEDIN	UNIMAN	UNIPR	UNIVLEEDS	UREAD	ULANC	EDF	AREVA
CEA	X			X																				
CHALMERS																								
CIEMAT																								
CNRS	X			X											P									
CTU																								
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JRC-ITU																								
JUELICH																								
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DOMAIN	2	WP	5	WP Leader	Chalmers
Manpower dedicated to this WP on the period				15.6 (+6?)	

Contribution to Deliverables (number and title of each deliverable)

D5.1 Experimental and modelling studies of transfer kinetics in the reference separation processes (Juelich, ULANC, CNRS, CEA)
D5.2 Impacts of solvent degradation and recycle on the EURO-GANEX process (CIEMAT, CEA)
D5.3 Performances on microelectrode for online monitoring of relevant parameters (UEDIN)
D5.4 contribution of computational multi-disciplinary approaches to the design of solvent extraction devices (ULEEDS)
D5.5 Status of flowsheet calculations made for WP22 process testing (Juelich, KIT, NNL, CEA)

Contribution to Milestones (number and title of each milestone)

Main achievements - Progress

A round ruby study of the different irradiation facilities was started. Data has been collected and subsequent meetings are to be held at the 2018 GENIORS autumn meeting

Main difficulties - Delays

Scientific publications, patents (beneficiary name and type of publication)

Geist, A.et al. The Extraction of An(III), Ln(III) and HNO3 by a TODGA Solvent: Equilibrium Modelling, ISEC International Solvent Extraction Conference 2017, Miyazaki City, Miyazaki Prefecture, Japan, 05.-09.11.2017

H. Galán, A. Núñez, I. Sánchez, A. González, U. Müllich, J. Cobos and A. Geist, "*Relevant experimental conditions to perform stability studies of extraction systems based on DGA and water-soluble stripping agents"*, accepted at RB2018 - Second edition of Radical Behaviour workshop, 19-20 April 2018, Würzburg, Germany.

E. Mossini et al., Optimisation and single-stage centrifugal contactor experiments with the novel hydrophilic complexant PyTri-Diol for the i-SANEX process. Submitted to *Solvent Extraction and Ion Exchange.*

WM2018 conference proceedings, March 2018- Multiphase Large Eddy Simulation of a Pulsed Sieve-Plate Extraction Column

"A Study of Cerium Extraction Kinetics by TODGA in Acidified and Non-Acidified Organic Solvent Phases in the Context of Fission Product Management", M.A.Bromley, C.Boxall, Progress in Nuclear Science & Technology, in press (2018).

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Effective collaboration between Beneficiaries (put crosses in boxes)																								
	CEA	CHALMERS	CIEMAT	CNRS	CTU	ICHTJ	IIC	IRSN	JRC-ITU	JUELICH	KIT	LGI	NNL	POLIMI	SCK-CEN	TWENTE	UEDIN	UNIMAN	UNIPR	UNIVLEEDS	UREAD	ULANC	EDF	AREVA
CEA																								
CHALMERS																								
CIEMAT											x													
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CTU																								
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IRSN																								
JRC-ITU																								
JUELICH											x													
KIT										x				x				x						
LGI																								
NNL										x	x													
POLIMI																								
SCK-CEN																								
TWENTE																								
UEDIN																						x		
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AREVA																								

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DOMAIN	2	WP	6	WP Leader	Robin Taylor
Manpower dedicated to this WP on the period				7.5	

Contribution to Deliverables (number and title of each deliverable)

Write here the N° (Dx.y) of the deliverables concerned by the work done in the semester. Mention when achieved.

D6.1: Assessment of applications of PTD in i-SANEX and EUROGANEX processes

- Contributions from POLIMI

D6.2: Optimized flowsheets for homogeneous recycling of advanced MOX fuel in GEN IV reactors

- Contributions from CHALMERS, JUELICH

D6.3: Optimized flowsheets for heterogeneous recycling of advanced MOX fuel in GEN IV reactors

- Contributions from JUELICH, POLIMI

Contribution to Milestones (number and title of each milestone)

Write here the N° (Dx.y) of the milestones concerned by the work done in the semester. Mention when achieved.

MS1: provide information on WP activity for the Work-Package Activity Summary Report 1

- Completed via this WPASR report

MS2: provide information on WP activity for the Work-Package Activity Summary Report 2

- Completed via this WPASR report

MS3: provide information on WP activity for the Work-Package Activity Summary Report 3
MS4: provide information on WP activity for the Work-Package Activity Summary Report 4
MS5: provide information on WP activity for the Work-Package Activity Summary Report 5
MS6: provide information on WP activity for the Work-Package Activity Summary Report 6
MS7: provide information on WP activity for the Work-Package Activity Summary Report 7
MS9: EURO-GANEX trial complete and TRU nitrate product ready for conversion studies

Main achievements - Progress

JUELICH – Key highlight:

- Single centrifugal contactor tests in the AmSel system for heterogeneous recycling of americium alone were conducted

POLIMI – Key highlight:

- During this semester POLIMI focused on obtaining data and preparing a paper on the optimization and single-stage centrifugal contactor experiments with PTD. This was submitted to a journal.

CHALMERS – Key highlight :

- Work with FS-13 has continued, including a series of experiments with variable concentrations of $CyMe_4$-BTBP and TBP, as well as studies of neptunium extraction and plutonium loading. A successful PhD defence was made.
- A journal paper was submitted.

No progress this semester at: CEA, CIEMAT, ICHTJ, KIT, NNL, TWENTE, UNIPR

No HYPAR report from JRC and UREAD – assumed no activity in WP6.

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DOMAIN	2	WP	7	WP Leader	Leturcq (CEA)
Manpower dedicated to this WP on the period				6.5	

Contribution to Deliverables (number and title of each deliverable)

D7.1 Report on dissolution model development [month 36] (**CEA**)
D7.2 Report on efficiency of the electrochemical decomposition of ligands from SX processes and on its inputs on MABB precursor properties
D7.3 Report on the development of the different MABB precursor synthesis routes and their contribution on MA homogenity, dust reduction, lowering sintering temperature [month 45] (**CEA**)
D7.4 Product quality, characterization and testing (**NNL**)

Contribution to Milestones (number and title of each milestone)

M7.1 “availability of model compounds” **achieved.**

Main achievements - Progress

<u>T71 Dissolution of model compounds</u>

Dissolution of the different PuO_2 samples and modelling the dissolution kinetics using morphology parameters of the plutonium oxide powders

<u>T73 Uranium/minor actinide based oxide conversion dedicated to transmutation</u>

CEA: $U_{0.9}Am_{0.1}O_{2-\delta}$ synthesis using weak acid resin route as dust-free MABB precursors.
NNL: Preparatory experiments to test methods and characterization data obtained when calcining some co-precipitated (U,Th) oxalate simulants are in progress. Precipitates were calcined ready for Raman and XRD analysis.
A concept flowsheet for (U,Pu) and MA co-conversion processes have been designed in EXCEL. This will now frame an experimental programme.

Main difficulties - Delays

None

Scientific publications, patents (beneficiary name and type of publication)

Journal:
Int. Conference Oral:
Int. Conference Poster:
Proceedings:
Patent:

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Effective collaboration between Beneficiaries (put crosses in boxes)																								
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DOMAIN	3	WP	8	WP Leader	LGI
Manpower dedicated to this WP on the period				1 (NNL) + 1 (LGI) + 2 (KIT)	

Contribution to Deliverables (number and title of each deliverable)

D8.1 “Process description report of concept Euro-GANEX plant” (M12)
D8.2 “Report on the comparison of SX processes for heterogeneous recycling” (M12)
D8.4 “SimPlant” – Engineering simulation of integrated plants (due in M48)
D8.3 “Mapping of the different status and alternatives for each ESNII concept” (due in M18)

Contribution to Milestones (number and title of each milestone)

None

Main achievements - Progress

D8.4 - SimPlant
Flowsheet build of EURO-GANEX process from fuel receipt to powder finishing has begun. It should be noted that this also contributes to T8.1.

D8.3 Mapping of the different status and alternatives for each ESNII concept
The deliverable is well advanced in terms of methodology, main sections and content. Firstly, the work focuses on the state of the art of ASTRID, ALFRED and MYRRHA. It is followed by a description of the system’s cycles, possible waste treatment strategies and the proposition and description of three waste treatment scenarios.
There have been several interactions with partners and external experts to validate the work and provide relevant information. A meeting with the partners from CEA, ORANO and EDF was held on June at LGI offices in Paris. The objective of the meeting was to agree on the focus of the deliverable, content and structure to follow. A follow-up meeting was planned in August to inform the partners about the advancement of the deliverable.

D8.1 and D8.2 were successfully delivered on M12. Work is schedule in the second semester of the project to undertake deliverable 8.3. LGI, NNL, KIT reported effort on this WP. EDF and ORANO also contributed to D8.3.

Main difficulties - Delays

2 weeks on D8.1

Scientific publications, patents (beneficiary name and type of publication)

None

Effective collaboration between Beneficiaries (put crosses in boxes)

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DOMAIN	3	WP	9	WP Leader	NNL
Manpower dedicated to this WP on the period	ULanc18 – but work to date funded by Lloyd's Register Foundation IRSN – 0.5pm NNL – 0pm (Concept Design for Safety Review complete under WP 8 – D8.1				
Contribution to Deliverables (number and title of each deliverable)					
D9.1: Safety Review minutes **D9.2:** Report on major hazard analysis for the Euro-GANEX process focusing on criticality aspects **D9.3:** Report on the methodology to establish a density law **D9.6** : Assessment of the corrosion vulnerability of common plant materials in the presence of key process					
Contribution to Milestones (number and title of each milestone)					
MS1: provide information on WP activity for the Work-Package Activity Summary Report					
Main achievements - Progress					

Task 9.1: Safety Review of a Euro-GANEX plant

The Concept Design for the EURO-GANEX plant has been complete under WP8.

An internal NNL Review has been arranged for 26 September. A consortium review will be conducted at the Antwerp meeting in conjunction with B Hanson.

Task 9.2-a: Preliminary hazard analysis focusing on criticality safety

Subtask 1: Carrying out of an independent safety review based on the Euro-GANEX plant design and process flowsheet provided by NNL (WP8) → **NNL Euro-GANEX flowsheet and process data (PD06 - Deliverable 8.1 – NNL 14620 - version 1 issued on 15/06/2018) sent on to the IRSN in August 2018.**

Work done: Internal documents/data collection, first thoughts and pre-evaluation to initiate the IRSN' safety review of the Euro-Ganex reprocessing plant design and flowsheet focused on criticality safety

Subtask 2: Assessment of the compatibility of the process and potential technology with the flows of actinide material to be treated, taking into account the criticality constraints

Step 1: Perform depletion calculations for MOX fuel in fast reactors in order to estimate the isotopic composition of Am, Cm, Pu. In this part, the impact of the following hypothesis could be studied: the initial composition of MOX fuel (in particular Pu/UPu, Pu isotopic composition, the eventual presence of Cm or Am) and the irradiation conditions (burn-up, depletion time) → **Work in progress (definition of the case studies and SFR fuel depletion calculations to be done)**

Task 9.2-b: Criticality studies (verification of the bounding nature of water moderation with regard to new extractants and diluents)

Subtask 1: Collect density of fissile species in GANEX solvents and write a methodology to establish a density law for NCS evaluations, including the need in terms of data and chemical measurements

Deliverable D9.3: Report on the methodology to establish a density law including the need in terms of data and chemical measurements depending of the range of applicability (Month 25) → **The drafting of the methodology report has just been finished. Technical checking and approval of the report are in progress.**

Task 9.3

The objectives of this task are:

- To perform corrosion assessments for stainless steels SS304L & S316L in the presence of as many of the GANEX inventory permutations as resource permits.
- To perform long term corrosion studies in relevant inventory permutations.
- To conduct corrosion and plant material compatibility tests for extraction and stripping stream compositions proposed for the EXAm process.

In conjunction with electrochemical quartz crystal microbalance (QCM) experiments, linear sweep voltammetry (LSV) studies have been conducted on the corrosion of SS304L and SS316L in the presence of the 1st cycle GANEX ligand, DEHiBA.

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The presence of DEHiBA in the organic phase is found to have no effect on the corrosion rate of SS304L and SS316L in Exxsol D80. The presence of DEHiBA in the organic phase when that phase is contacted with nitric acid is found to reduce the rate of SS316L in HNO3, but has no effect on SS304L.

Four steel types have been acquired for long term testing (SS316L, SS304L, NAG 18/10 and SS310), characterised and turned into rotating disk electrodes. A double junction arrangement suitable for long term corrosion testing has been developed with the aid of the National Nuclear Laboratory (NNL) and a prototype setup is under construction. Based on work carried out in SACSESS, SO_3-Ph-BTP and AHA in HNO_3 has been identified as the most suitable corrosion system to initially be studied long term. Rotating disk and rotating cylinder electrodes will be manufactured and used for these tests, to provide similar conditions to those expected in storage tanks, process pipework and centrifugal contactors.

Main difficulties - Delays

T9.1 – None
T9.2 – IRSN needs information quickly on Euro-GANEX solvents chemical data.
T9.3 – None

Scientific publications, patents (beneficiary name and type of publication)

Not applicable.

Effective collaboration between Beneficiaries (put crosses in boxes)

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DOMAIN	4	WP	12	WP Leader	Bruce Hanson
Manpower dedicated to this WP on the period				0.25	

Contribution to Deliverables (number and title of each deliverable)

D421 Report on travel bursary and secondment grant attribution (ULEEDS)
D422 Organisation of the think-tank event (ULEEDS)
D423 Deliverable report on specification for on line packages (ULEEDS)
D424 Draft Purex package ready for testing (ULEEDS)
D425 iSanex/Ganex package ready for testing (ULEEDS)
D426 Packages tested and released (ULEEDS)
D427 Knowledge and Data Management Plan (CHALMERS)

Contribution to Milestones (number and title of each milestone)

MS14 Attribution of travel bursaries and/or secondment grants
MS17 Convene development group from project partners and agree roles for online training courses
MS18 Draft specification for on line packages for consultation

Main achievements - Progress

T421 Two awards for travel bursaries have been made totaling €1,982. This brings the total awarded to date to €8,762 leaving €11,238 in the fund.

T422 The operational process for the Think Tank has been developed and agreed– see Appendix.

T423 There has been an initial meeting to identify existing examples of online training packages. Delivery platform options have been identified.

Main difficulties - Delays

None to report.

Scientific publications, patents (beneficiary name and type of publication)

None planned.

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DOMAIN	4	WP	13	WP Leader	LGI
Manpower dedicated to this WP on the period				1PM	
Contribution to Deliverables (number and title of each deliverable)					
Contribution to Milestones (number and title of each milestone)					

Main achievements - Progress

- Editorial preparation of the first project newsletter (content, design, social media recap…).
- Promoting the 2nd Project Meeting in Würzburg (Germany) followed by the 2nd edition of the Radical Behaviour workshop (RB2018).
- Design of a flyer and a roll-up to promote GENIORS at different upcoming events.
- Monthly reports of online audience through Google Analytics.
- Social media: daily management of the GENIORS Twitter account + monitor the online audience (followers, retweets…).
- Updates of the project website (deliverables for download, news and upcoming events…).

Main difficulties - Delays

- Engaging partners in following the GENIORS Twitter account.
- Attracting audience to the project website and to subscribe to receive the future project newsletters.

Scientific publications, patents (beneficiary name and type of publication)

Journal:
Int. Conference Oral:
Int. Conference Poster:
Proceedings:
Patent:

Effective collaboration between Beneficiaries (put crosses in boxes)

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