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# ASGARD

## Advanced Fuels for Gen IV Reactors: Reprocessing and Dissolution


Contract Number: 295825




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CO	Confidential (only for ASGARD partners)		

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## Relevant domain(s) and workpackage(s)

Tick **ALL** ☒ or select in the following table:

DM	WP
DM 1 <input type="checkbox"/>	WP 1.1 <input type="checkbox"/> WP 1.2 <input type="checkbox"/> WP 1.3 <input checked="" type="checkbox"/>
DM 2 <input type="checkbox"/>	WP 2.1 <input type="checkbox"/> WP 2.2 <input type="checkbox"/> WP 2.3 <input type="checkbox"/>
DM 3 <input type="checkbox"/>	WP 3.1 <input type="checkbox"/> WP 3.2 <input type="checkbox"/> WP 3.3 <input type="checkbox"/>
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## Project information

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Start date – End date:	01/01/12 – 31/12/15 i.e. 48 months
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# INTRODUCTION

The ASGARD project was constructed to bridge the scientific societies working in the area of recycling of nuclear waste. Traditionally these areas have acted rather separately in European framework funded projects even if some projects e.g. ACSEPT originally focussed on separation issues did also have work performed in conversion issues. The main parts of the circular nuclear fuel cycle are shown in Figure 1 together with some of the major EU funded projects in relation to the ASGARD scope.



**Figure 1 The ASGARD project in relation to the steps in a circular nuclear fuel cycle and in relation to other important projects in the domain.**

Essentially the circular nuclear fuel cycle consist of: the spent fuel being dissolved in a suitable matrix (usually nitric acid). Then the elements to be further used in production of new nuclear fuels (the actinides) are separated from the rest of the waste by either hydrochemical or pyrochemical techniques. In ASGARD we mainly aim at the hydrochemical techniques, but a smaller part on specialised fuels are also discussed with respect to pyrochemical methods. After the separation, the product has to be converted to suitable precursors for fabrication of the new fuel whereupon the fabrication process starts. It is in this area where the major part of the work of ASGARD is focussed. In future nuclear systems it is possible to use several different types of fuels whereas today oxide fuels dominate completely. It is expected that regardless of fuel type the processes outlined in Figure 1 will be necessary and thus in ASGARD we have decided to divide the work in domains given by the fuel types and tasks relating to the specific questions arising for that particular fuel type, see Figure 2.

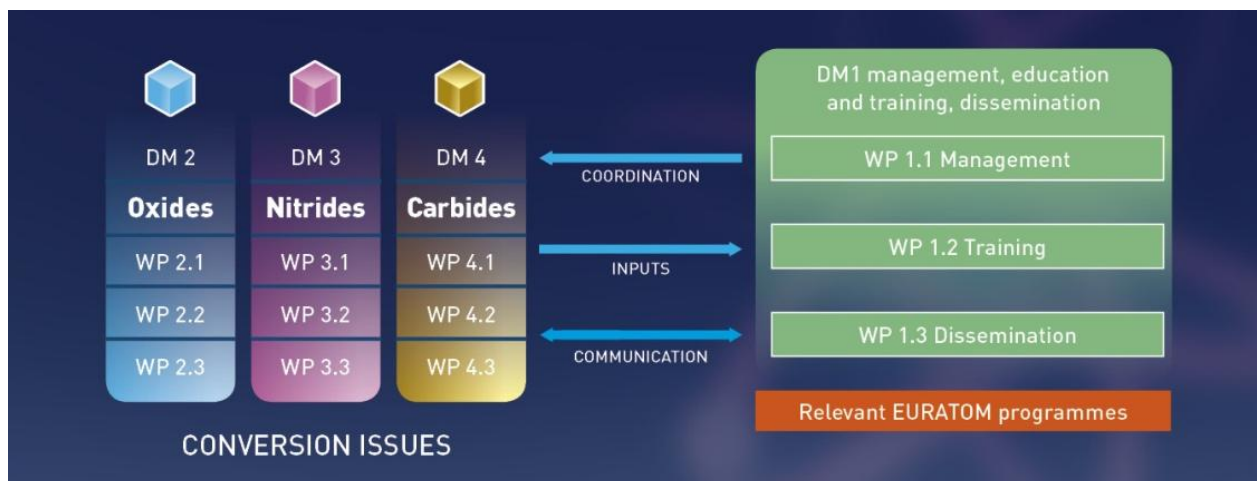


Figure 2 General structure of the ASGARD project.

As seen in Figure 2 there is a clear cross cutting activity when handling the conversion issue. There are also other specific issues that will cut cross the domains in more specific areas.

The work package division for each domain is given in Figures 3-5 below.

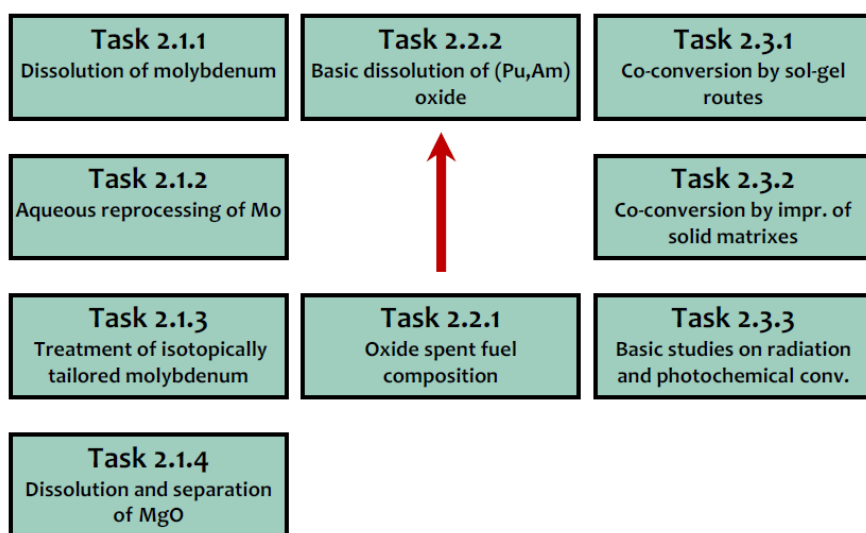


Figure 3 The organisation of the oxide domain.

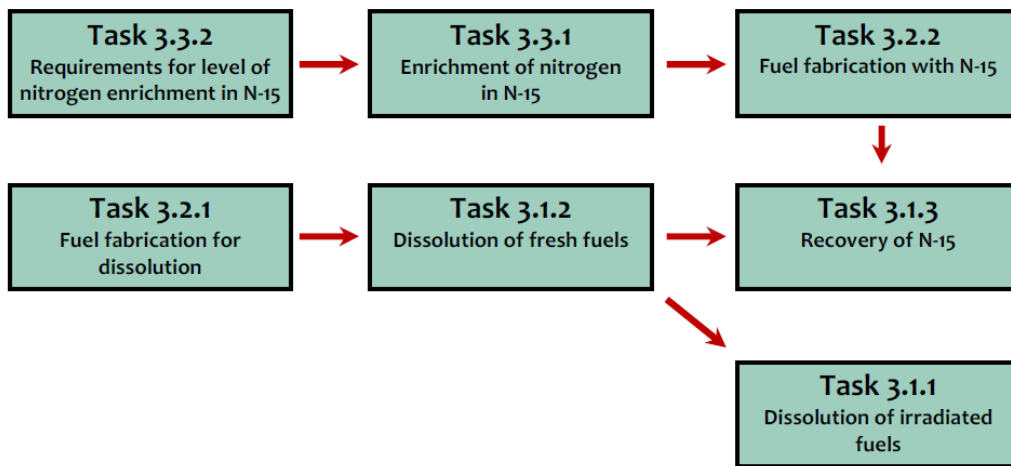


Figure 4 The organisation of the nitride domain.

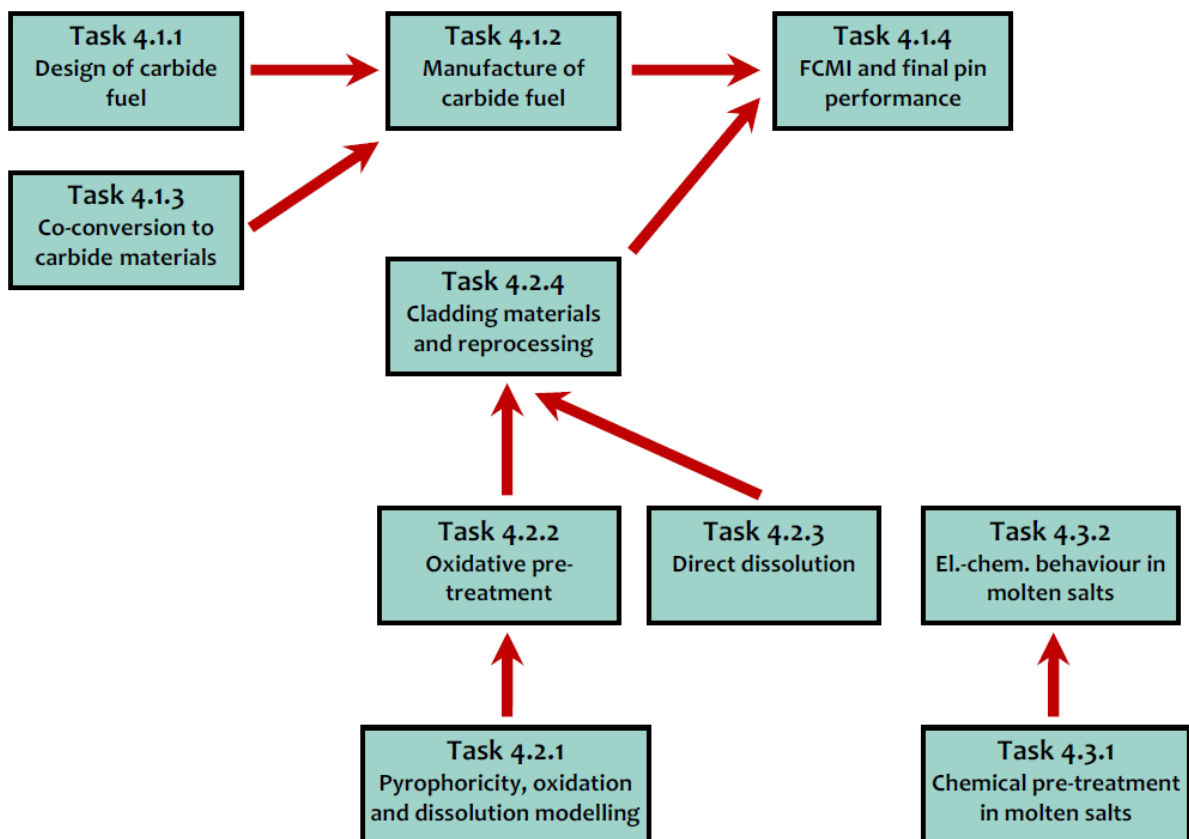


Figure 5 The organisation of the carbide domain.

Within the ASGAR project, 17 partners from 9 countries work together well distributed within the domains described above. The participation of the main nuclear actors in Europe will ascertain that the project will deliver scientific results of the highest standard in a structured and useful way. The last part is being addressed by a selected Scientific

Advisory Board and End Users Group making sure that the results of the ASGARD project are in line with what the future industry can accept at the same time as it is on a high scientific level.

Last but not least the ASGARD project has an extensive scheme for training and education of younger researchers. This program comprise not only dedicated lectures and summer(winter)schools, but also funding possibilities for shorter or longer visits to perform work in other laboratories than their own. In addition, it is also possible to receive funding to present ASGARD work at international meetings and conferences thus promoting not only the aspect of broadening the experiences of the younger researchers but also serves as an excellent tool for disseminating the results of the ASGARD project.

# 1 PROJECT SUMMARY FOR THE FIRST YEAR

The first year of any project is in many cases crucial for its success. This is particularly true for projects dealing with radioactive material where work permits and transport of material can take a considerable time. Thus it is good to realise that the ASGARD project is running according to schedule and these parts have been carried out in accordance with what was planned from the beginning. There were some parts of the project that had to rely on the support from different entities and here I would like to express a special gratitude to KIT-INE in Germany for agreeing to support us in the acquirement of americium for the work to be carried out at NRG and Chalmers. This material is crucial for the production of (Pu, Am) bearing fuel which will be manufactured and then its dissolution behaviour will be tested.

The ability to hold on to the time table is also true for the training and education domain. Several funding schemes for training and mobility of younger researchers have been launched and in some cases already granted. During its first year ASGARD has also been co-organising a session in the ATALANTE conference focussed on actinide materials. Thanks to the cooperation between ASGARD, FAIRFUELS and CINCH there has already been a Winterschool in fuel manufacturing prepared for M13 during the first year. This school was held at NRG in Petten in connection to the project meetings of ASGARD, FAIRFUELS and CINCH. Thus it is clear that a good synergy is already established between ASGARD and other relevant EU funded projects. It is expected that in the future other newer projects such as SACSESS will also be included into circle.

In all technical domains there were several basic studies planned for the first year to work as a basis for the future work. These have been carried out well and a good survey of the field now exists within the consortium.

On the practical side some fuel type simulates using zirconium as an analogue for plutonium have already been manufactured and among these are molybdenum based fuel made at NRG and nitrides at KTH. Since the ASGARD project deals with the whole fuel cycle the dissolution behaviours of e.g. MgO pellets has also been studied and described.

Thus, in conclusion it is fair to say that the first year of the ASGARD project has been successful without any real delays or other mishaps affecting the progress of the project. Normal unplanned events occurring during the life time of any large project has been handled efficiently between the Administrative Management Office (AMO) and the Project Coordination Committee (PCC).

Christian Ekberg

ASGARD Coordinator



## 2 DOMAIN SUMMARIES FOR THE FIRST YEAR

### 2.1 DOMAIN 1 – MANAGEMENT, EDUCATION AND TRAINING, DISSEMINATION

The management of the project; sustainable education and training and dissemination of the knowledge achieved within ASGARD project are addressed in Domain 1, in three different work packages (WPs).

The objective of WP1.1 is to assure efficient co-ordination of technical activities of ASGARD project, quality assurance of the project's deliverables and the processes leading to them, effective overall legal, contractual, ethical, financial and administrative management of the project and management of the project's decision-making processes to respect relevant standards. Establishment of internal communication within the partners is also one of the tasks. This goal has already been achieved in the first 6 months, and is mirrored in the: Collection and distribution of the: Grant agreement; Consortium agreement; Distribution of EC advance payment to the partners.

Creation of: the project webpage (internal):<https://pingpong.portal.chalmers.se/login/>.

Creation of templates for: Partner Interim Report, Domain Activity Report, Detailed Work Plan (D1.1.1), Quality Plan – Project Handbook (D1.1.2), templates for minutes of various types of meetings.



Figure 6 ASGARD consortium at Jachymov meeting in June 2012.

As a continuation within the second 6 months, the project has been monitored and documents, web-page and internal project webpage are kept up to date.

The main objective of WP1.2 is to stimulate exchange of knowledge and practical experience among the community and future researchers. Students (BSc, MSc and PhDs) represent the primary target group. Teachers and other members of the community benefit from ASGAR activities and measures in area of education/training and mobility. Deliverable D1.2.1 “Initial training programme” and D1.2.2 “Travel Fund” has been elaborated in order to put the base for the training program. So far, 7 applications were received for the travel fund and 6 were granted for different purposes, like: training courses, conference participation and the Winter School. Also, ASGAR has been co-organizer of a section in ATALANTE conference, in September 2012.

The first Winterschool was planned for January 2013, organized by NRG in Petten, Netherlands.

The main objectives of WP 1.3 are to guarantee dissemination of knowledge of the project and results from the project into the nuclear community. The projects webpage (public) is the first achievement: <http://asgardproject.eu/>. Also, the ASGAR corporate identity, project logo, brochure and leaflets have been produced. A series of at least 13 press releases and 5 conference participations are achieved so far.

A database comprising the stakeholders has been made available on ASGARs website.

A sustained networking effort is conducted towards synergy with other European networks and organizations (ENEN, ENFTP, SNETP, TSO, etc.).

No problems have been encountered, even if one of our DM and WP leaders has been replaced. The process was smooth and the leadership has gradually been transmitted to the next one. No delays have been encountered.

**Project partners**

Chalmers University of Technology – project coordinator	(Sweden)
Forschungszentrum Jülich GmbH	(Germany)
Instytut Chemii i Techniki Jądrowej	(Poland)
National Nuclear Laboratory Limited	(United Kingdom)
Paul Scherrer Institut	(Switzerland)
Nuclear Research and Consultancy Group	(Netherlands)
Karlsruher Institut für Technologie	(Germany)
Commissariat à l'énergie atomique et aux énergies alternatives	(France)
České vysoké učení technické v Praze	(Czech Republic)
Kungliga Tekniska Högskolan	(Sweden)
Evaluation s.r.o.	(Czech Republic)
Westinghouse Electric Sweden	(Sweden)
Institut National de Cercetare-Dezvoltare Pentru Tehnologie Izotopice si Moleculare	(Romania)
University of Leeds	(United Kingdom)
University of Manchester	(United Kingdom)
University of Cambridge	(United Kingdom)

**Project information**

ASGAR is a Large Scale Integrated Project supported by the EU within European Atomic Energy Community's 7th Framework Programme (FP7 2007-2011), EC-Grant Agreement No. 295825. Time Frame: January 2012 – December 2015 (duration 48 months)

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**ASGAR**  
Advanced fuelS for  
Generation IV reActors:  
Reprocessing and  
Dissolution

[WWW.ASGARPROJECT.EU](http://WWW.ASGARPROJECT.EU)

Figure 7 ASGAR project leaflet.

## 2.2 DOMAIN 2 – OXIDE FUELS

Domain 2 on the fabrication and reprocessing of inert matrix fuels has begun and is proceeding to plan for this first 12 months.

As Inert Matrix Fuels contains significant amount of inert matrix (e.g. MgO, Mo) special head end treatments after dissolution are necessary. On the other hand, it is necessary to remove the inert material to simplify the separation of the actinides and to improve the final conditioning of the remaining fission products before vitrification.

Mo pellets are prepared and characterized both in Jülich and NRG. Dissolution in HNO<sub>3</sub> was performed at different acid concentrations and different temperatures. The dissolution rate is strongly dependent on the acid concentration: Higher acid concentrations result in higher dissolution velocities but also in more precipitation. A second series of experiments using ferric nitrate as an additive was performed (iron(III) nitrate is known to significantly increase the solubility of uranyl molybdate in nitric acid).

NRG will use the irradiated (Pu<sub>0.8</sub>Am<sub>0.2</sub>)O<sub>2</sub> in Mo of HELIOS pin5 to perform basic dissolution studies. The dissolution will take place in the G1 (chemical) cell of the Hot Cell Laboratories. First, the safety assessment had to be done. This safety assessment is finished. Cold dissolution (pure Mo pellets) tests were performed. As Pu is very difficult to dissolve, semi hot tests are being performed on PuO<sub>2</sub> (12.2 wt.%) in Mo matrix (pellets from LWR Deputy EU project). The results exhibit a trend in two steps. First the molybdenum matrix should be dissolved by mild nitric acid. Second, after recovering the non-dissolved PuO<sub>2</sub>, a process involving hydrofluoric acid (HF) is used. The full process still need to be optimized before going into the hot cell.

Solutions of isotopically pure <sup>98</sup>MoO<sub>3</sub> in nitric acid were investigated as function of HNO<sub>3</sub> concentration by means of electrospray ionization mass-spectrometry (ESI MS). In strongly acidic media, the formation of dimeric [Mo<sub>2</sub>O<sub>5</sub>OH]<sup>+</sup> is favoured. At high HNO<sub>3</sub> concentrations, pronounced nitrate coordination occurs. Formation of polymeric species (up to 13 Mo moieties) becomes more pronounced at lower HNO<sub>3</sub> concentration while the formation of nitrate complexes decreases. <sup>98</sup>MoO<sub>3</sub> could only be dissolved in 0.5 mol/L HNO<sub>3</sub> over long time scales by heating the solution. ICP-MS measurements of this solution revealed that not all the MoO<sub>3</sub> was dissolved, even if no precipitate was visible. This solution showed the largest polymeric species, reflecting the on-going polymerization processes toward the formation of colloidal species and precipitates.

First measurements with <sup>90</sup>Zr(IV) (as analogue for Pu(IV)) added to the Mo solution were performed. The assignments and interpretation of the spectra is currently underway. These measurements should reveal whether mixed Zr-Mo species are formed, or whether the two metals form separate species.

MgO pellets were made and characterized. Dissolution of MgO pellets was studied as a function of HNO<sub>3</sub> concentration, temperature, and pellet density. In 4 and 7M HNO<sub>3</sub> at 90 °C complete dissolution could be achieved within about 1 h. The average dissolution rate at 90 °C is about ten times as high as the dissolution rate at room temperature.

The dissolution rates of the pellets with densities below 96% are very similar whereas the dissolution velocity at a pellet density of 99.4% is significantly smaller.

Monte Carlo simulations of the composition of oxide fuels dedicated to transmutation of minor actinides has been completed, using the SERPENT code. Two cases were studied: Minor Actinide Bearing Blankets and a CERMET inert matrix fuel. Deliverable 2.2.1 has been completed according to schedule.

In the Conversion from Solution to Oxide Pre-Cursors part the work was focused on the production and characterization of particles. Microspheres containing only uranium and microspheres containing Nd and U were fabricated and characterized. The content of Nd



was increased by an increment of 5 % up to  $\chi(\text{Nd}) = 40$  %. Particles of all fractions were characterized by several methods.

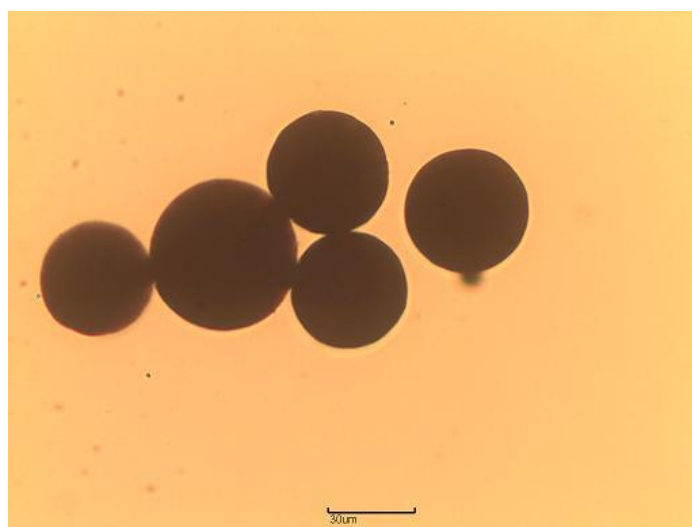


**Figure 8 Photo of fabricated microspheres dried at air.**

Thermogravimetric analysis coupled with differential scanning calorimetry (TGA/DSC) was used to investigate the thermal behaviour of the microspheres (Netzsch “STA 449 C Jupiter”). One can observe a removal of the residual humidity as well as a decomposition of the organic gelification agent up to 300 °C. One can observe a changing effect at the temperature of ca. 625 °C. Samples containing only small amounts of the Nd (up to 17.40 %) transit endothermic, while the other microsphere fractions show an exothermic effect. These transition will be further investigated in the future. Scanning electron microscope (SEM) investigations were performed to observe the surface and the morphology (FEI “Quanta 200F”). Furthermore diameter measurements were done and the chemical composition was investigated by energy-dispersive X-ray spectroscopy (EDX), these observations were done with a EDAX “Genesis 4000” system. X-ray diffraction (XRD) analysis is a long-range method and was performed to define the structure of the crystalline sample.

The lattice parameter  $a$  as well as the unit cell volume of the cubic system were calculated by the use of the Nelson-Riley method. The software Match! (Version: “1.11 f”) by Crystal Impact was used for data analysis. Up to a Nd content of 25 % the expected linear behaviour according to Vegard’s law is evident. The fractions having a higher  $\chi(\text{Nd})$  show a different behaviour which has to be further investigated.

Optimization of the preparation and alkalization of U-ASC-Nd sols and characterization of them (pH, viscosity, UV-Vis, stability in time) was done.



**Figure 9 Spherical particles of U-ASC-Nd gel after calcination.**

The gelation process was optimized as well, preliminary experiments with Internal Gelation to kernels is performed - sols solutions with molar ratio  $\text{UO}_3/\text{ASC}=1$  and doped by Nd (MR  $\text{U}/\text{Nd}=0,1$ ) as surrogates of MA were studied. The gels are characterized by IR spectroscopy, SEM, EDS, and IC-PMS. Drying and calcination of gels is done and characterized by TG DTA analysis.

The basic studies on radiation and photochemical conversion of actinides from solution to solid oxides has been extended from the radiation induced preparation of uranium oxide to the preparation of uranium(IV), thorium(IV) and mixed uranium-thorium oxides.

The preparative method used is based on UV radiation induced formation of solid phase in aqueous solutions containing uranium and/or thorium nitrate and ammonium formate. Irradiation was performed using medium pressure mercury lamp. Changes in solutions' absorption were evaluated by UV-Vis spectrophotometry and the pH was measured.

Finely dispersed solid phase formed under irradiation was separated via microfiltration, washed with deionized water, dried at room temperature and heated at temperatures 200 – 800 °C in air or argon + hydrogen ( $\text{Ar}+\text{H}_2$ ) atmospheres. Solid phase was characterized by thermal analysis under air and  $\text{Ar}+\text{H}_2$  atmospheres, elemental (C, H, N) analysis, X-ray powder diffraction, specific surface area evaluation, X-ray fluorescence and both scanning and high resolution transmission electron microscopy.

Formed solid precursors are amorphous. Results of elemental analysis indicate that there are only negligible amounts of carbon in solid precursor formed from any solution, which makes it possible to prepare crystalline nanomaterials containing U(IV) oxide from (amorphous) solid precursors directly via treatment in  $\text{Ar}+\text{H}_2$  atmosphere without pre-calcination in air.

Subsequent heat treatment under various atmospheres leads to formation of nanocrystalline uranium and/or thorium oxides at minimum temperatures in the interval 300 - 550 °C. In the solid precursors prepared via irradiation of solution containing both uranium and thorium nitrates, heat treatment at temperatures above 300 °C results in formation of solid  $\text{UO}_2\text{-ThO}_2$  solution. Based on the knowledge of lattice parameters of both oxides and the lattice parameter of mixed uranium-thorium oxide, the amount of oxides in solid phase containing both U and Th heated was calculated. The solid phase prepared by irradiation of solution with U:Th ratio 2:1 is  $\text{U}_{0.95}\text{Th}_{0.05}\text{O}_2$  and for U:Th ratios both 1:1 and 2:1  $\text{U}_{0.60}\text{Th}_{0.40}\text{O}_2$  stoichiometry.

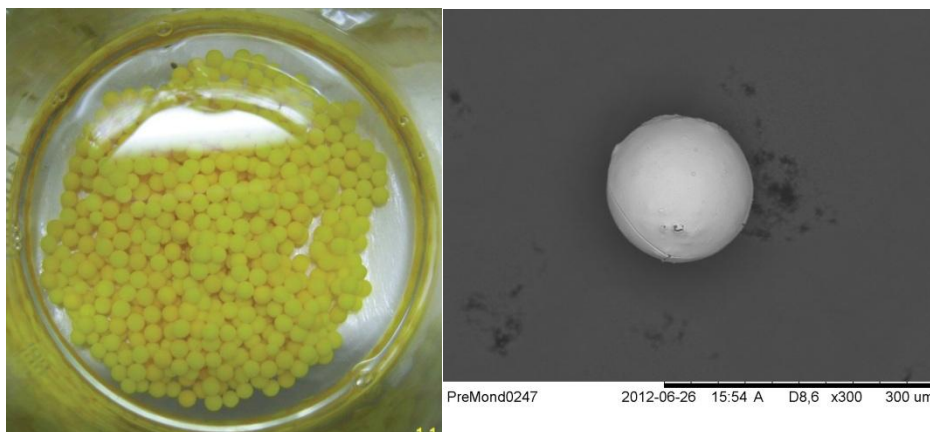
The method yields material with high purity, consisting of nanoparticles from 3 to 30 nm in diameter depending on post-radiation heat treatment.

## 2.3 DOMAIN 3 – NITRIDE FUELS

Cold dissolution tests of ZrN have been made at NRG. The safety assessment for dissolution of irradiated (Pu,Zr)N has been approved.

A method for recovery of N-15, based on hydrolysis of UN has been defined by KTH, and an apparatus for test of this method has been designed.

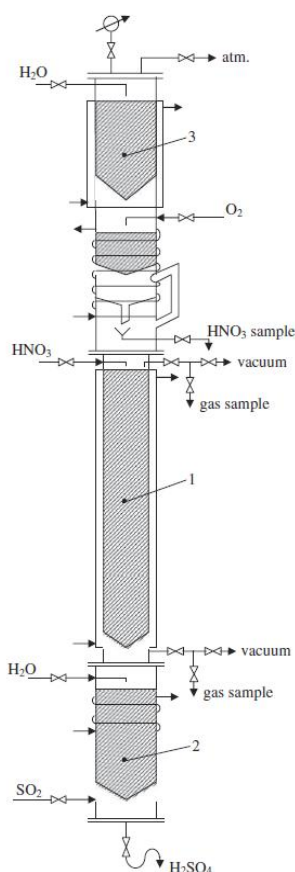
Glove-boxes for manufacture of plutonium bearing nitride fuel have been commissioned at Chalmers, and are now operational. The facility includes a sol-gel-line for manufacture of oxide microspheres with dispersed carbon. IN a greater detail it may be worth to mention that a novel source for the normally used carbon powder has been tested. This is carbon nanotubes which makes it possible to keep the sol suspension more stable for longer time without sedimenting. Solid spheres of uranium nitride and zirconium nitride precursors have been manufactured, see Figure 10.



**Figure 10, Uranium spheres as produced by the sol-gel techniques (left) and a SEM picture of one of the spheres (right).**

The enrichment of N-15 required for manufacture of nitride fuels has been estimated at 99% for a fuel cycle where nitride fuels are used in fast reactors dedicated to minor actinide transmutation.

The experimental facility for N-15 enrichment has been constructed and tested at INCDTIM, where also equipment for concentration of sulphuric acid has been commissioned and tested.



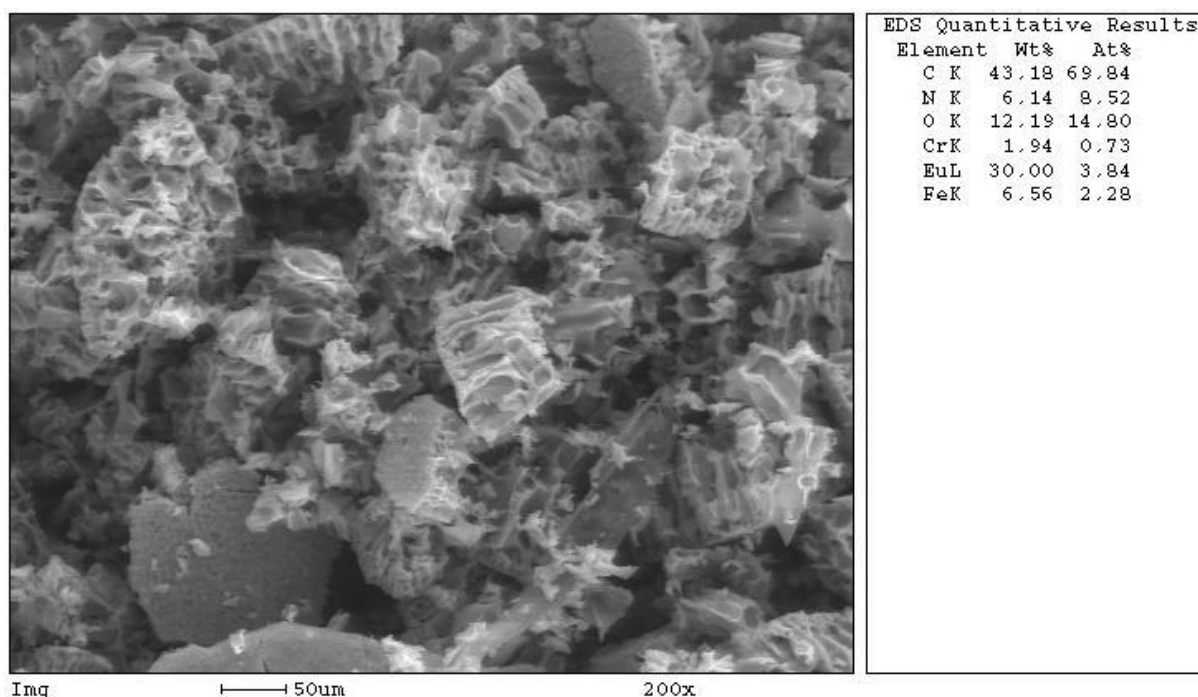
**Figure 10 Schematic diagram of the experimental plant: (1) stainless steel column, (2) hastelloy product refluxer, (3) stainless steel waste refluxer.**

Domain 3 is so far on schedule. Within next reporting period, it is important that fabrication of samples for dissolution tests will start according to plan. For this purpose, additional equipment must be installed and commissioned at Chalmers and KTH.

## 2.4 DOMAIN 4 – CARBIDE FUELS

Carbide fuel pellet design modelling has defined the dimensions and creep stress enhancement material levels resulting in a pellet that is expected to exhibit reduced fuel swelling compared to a reference material by about 50% by controlling the in reactor temperature profile of the fuel pin. The benefits of a gradient density fuel could not be satisfactorily proven and it is decided not to pursue this option. Sphere-pac and fuel pellets will be made according to the results presented in D 4.1.1.

While progress on Task 4.1.3 is achieved with a patent being written in the colloid route, progress has been made for the PAN beads and Ascorbate methods. The work initially planned on UC/SiC interactions (Task 4.1.4) has not started yet and it will be complemented by a study on UC/TiC interactions and this has been approved at the Governing Board 12 month meeting.



**Figure 112 SEM picture and EDAX analysis results of Eu<sub>2</sub>O<sub>3</sub> in graphite matrix obtained by graphitization of material prepared by infiltration procedure.**

Pyrophoricity studies continue. Studies of direct dissolution of UC in nitric acid started during this period showing a quick dissolution leading to organic rich dissolution liquors. Characterization of these organic compounds are in progress with previously unreported organic compounds identified, although a large proportion of the organics are yet to be characterised.

The treatment of UC material with CO<sub>2</sub> has been proven to give a UO<sub>2</sub> product that is soluble in nitric acid without the formation of organics in solution. This will now be applied to unirradiated (U,Pu)C. Analysis of irradiated (U,Pu)C fuel by the InVAP method will also

reveal the major volatile fission products that will be released during high temperature treatments.

The influence of organics, known to form during carbide dissolution (e.g. oxalic and mellitic acid), on the behaviour of iodine under dissolver conditions is complete. No significant increases in organo-iodides were detected and these experiments will now be repeated in the presence of UC.

Molten salt processing of carbides is an alternative method to oxidative pre-treatment and has been demonstrate simply using SiC and Ca-Cl<sub>2</sub> + oxygen donor materials and will be the focus of the work over the next 6 months.



## CONCLUSIONS

The purpose of this document is to provide the broad public with short summary of work carried out by ASGARD project during the first year of its implementation. The document uses the information provided by the project Beneficiaries within their internal project reports (Project Interim Reports and Domain Activity Reports). General introduction of the project is provided in the beginning of the report.

Publishable summaries for each ASGARD domain are completed with short executive summary at the project level prepared by the Coordinator.

This report is public and shall be made available for unrestricted download on the ASGARD webpage [www.asgardproject.eu](http://www.asgardproject.eu).