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School on Physics, Technology and Applications of Accelerator Driven Systems (ADS)

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Partitioning. Part II

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School on Physics, Technology and Applications of Accelerator Driven Systems November 19th –30th 2007 Trieste, Italy





Outline 2

2

introduction

- some historical background
- *basic principles and data*

break

- *process developments*
- *international networks and collaborations*
- *outlook*



- promote long-term availability of the systems and effective fuel utilization
- minimize and manage the nuclear waste produced (co-recycling of all actinides) and notably reduce the long term stewardship burden in the future, thereby improving protection for the public health and the environment.
- increase the assurance that systems are a very unattractive and least desirable route for diversion or theft of weapons-usable materials.

Change of philosophy

dirty fuel-clean waste instead of clean fuel-dirty waste



European partitioning projects

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	FP3 SCA 1990-1994
FP = Framework Program	<i>FP 4</i> SCA <u>1994–1998</u>
SCA = Shared Cost Action project	FP5 SCA 1998-2002
IP = Integrated project	Ţ
CP = Collaboration project	FP6 IP <u>2002–2006</u>
	<i>FP7</i> <i>CP</i> 2007–2010

HLLW partitioning by means of completely incinerable extractans

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New partitioning techniques for minor actinides, NEWPART

Pyrometallurgycal reprocessing PYROREP New Solvent Extraction Processes for minor actinides, PARTNEW

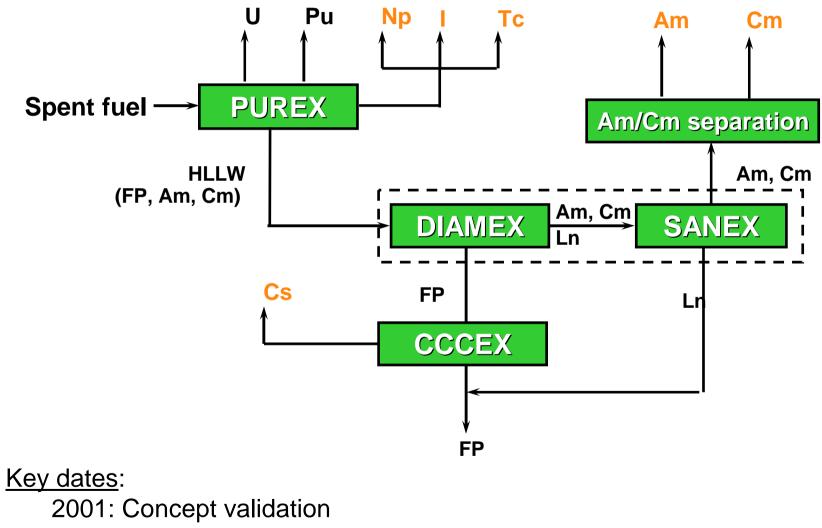
Partitioning of minor actinides from high active wastes issuing from reprocessing of spent nuclear fuels, EUROPART

Actinide recycling by separation and Transmutation **ACSEPT**



Advanced separation french reference flowsheet

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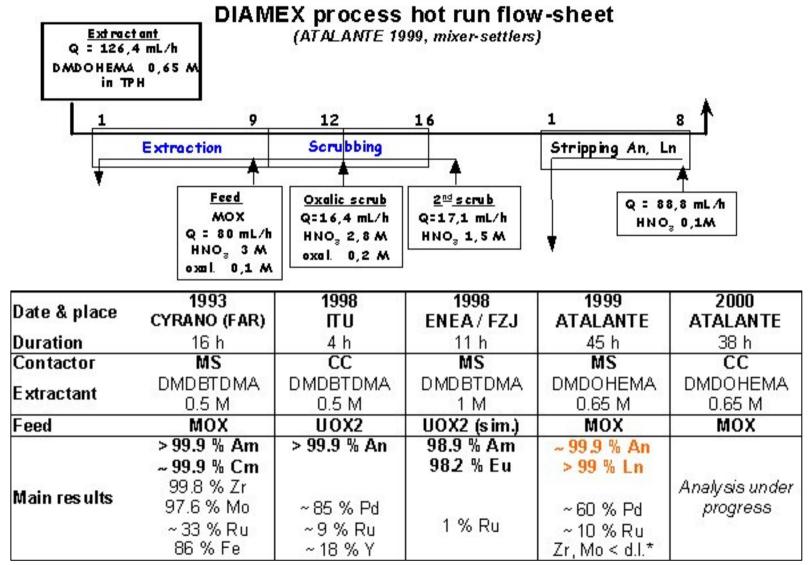


2005: Process definition



DIAMEX process: hot runs

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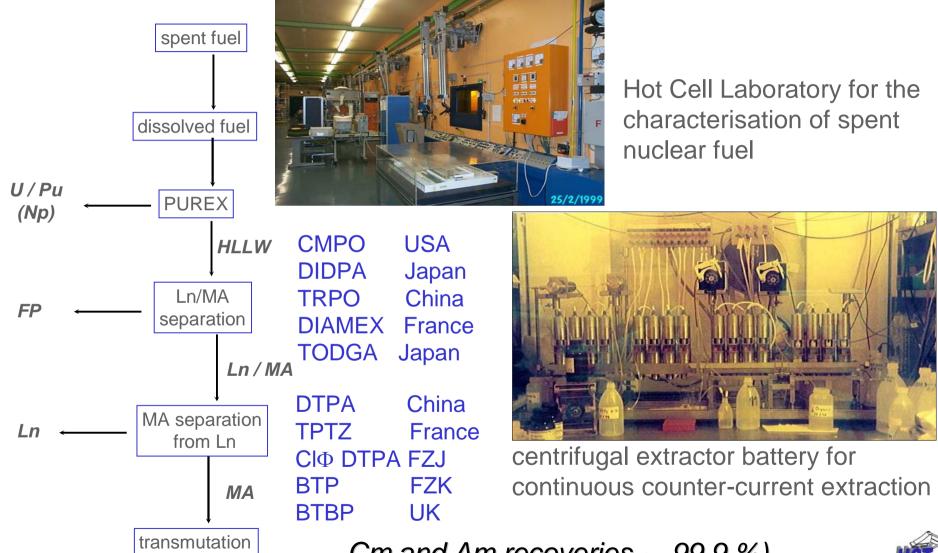
*d.l. : concentration under detection limit

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MA partitioning from genuine fuel by liquid-liquid extraction

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Cm and Am recoveries ~ 99.9 %)

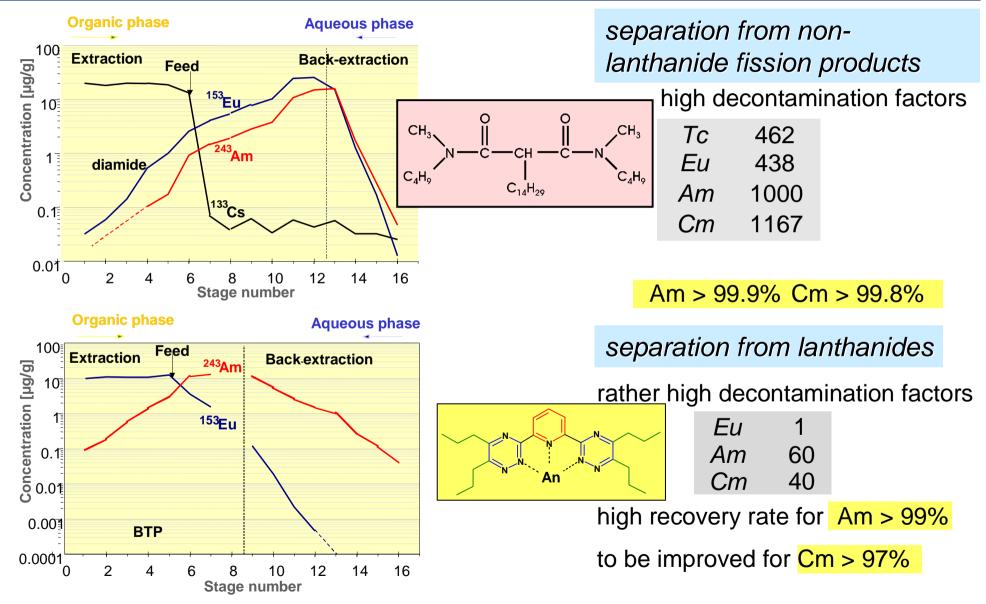




An partitioning from genuine HLLW

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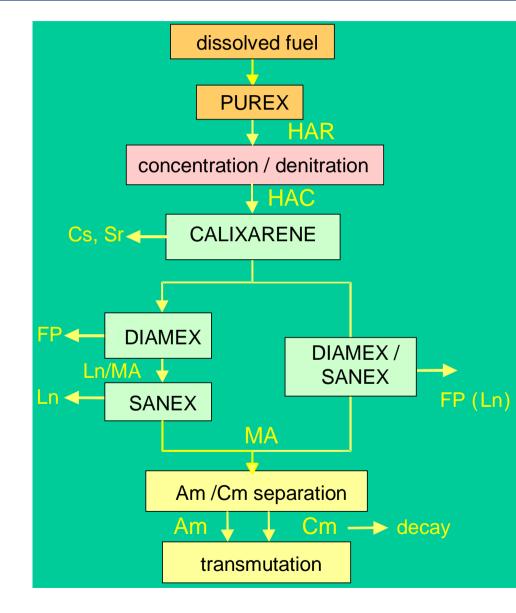
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Advanced PUREX R&D towards industrial application

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Industrial advantages:

- simplified PUREX
- low volumes of feed, waste minimisation and
- compact process

Achievements at ITU:

- demonstration of MA recovery by the DIAMEX/SANEX process combination using HAC (10x concentrated) as feed
- demonstration of direct MA extraction using BTP

European programs: PARTNEW and CALIXPART



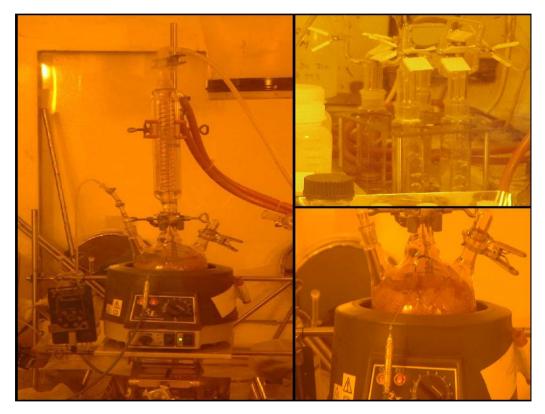
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EUROPART



Concentration/denitration

- Concentration Start volume 4 L Volume reduction 10 times Acidity increase (3.2 to 12 M)
- Denitration formic acid (HCOOH) exothermic reaction



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400 mL High Active Concentrate (acidity 4 M)

Concentration factor 10.2 (without precipitation of MA)



DIAMEX HAC (conc. factor 10) test

	Recovery Good extract	ion and	DF		Recoveries (%) Raffinate An/Ln fraction fraction		
back-extraction properties		Y	85	1.2	98.8		
		Zr	1	> 99.99	< 0.01		
	Dext	Dbext	Мо	1	99.3	0.6	
Am	>20000	>300	Тс	217	< 0.01	2	
Cm	>10000	>2000	Ru	1.1	92	6	
			Pd	1	98.8	0.9	
First successful DIAMEX-HAC			Ln	> 5000	< 0.1	> 99.9	
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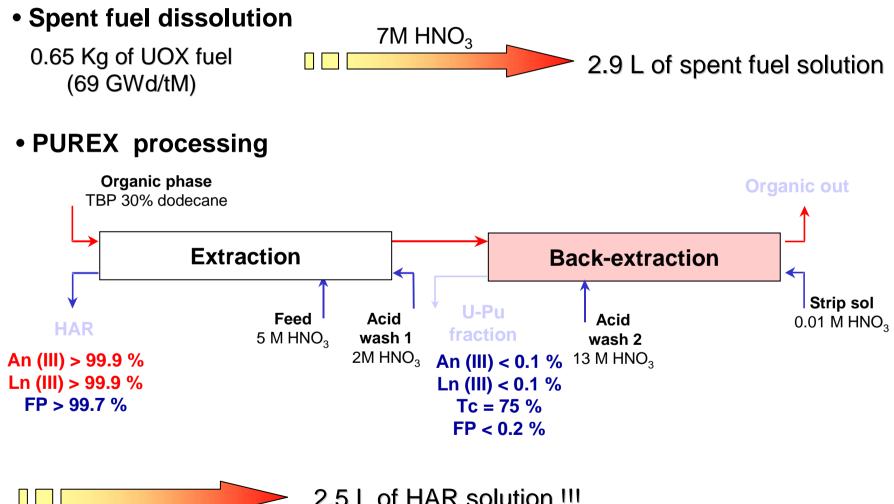
First hot test, a major event in the field of partitioning of minor actinides

Zr	1	> 99.99	< 0.01
Мо	1	99.3	0.6
Тс	217	< 0.01	2
Ru	1.1	92	6
Pd	1	98.8	0.9
Ln	> 5000	< 0.1	> 99.9
Np	50	2	49
Am	20000	0.005	99.7
Cm	10000	0.01	99.9



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Preparing feed for a TODGA-BTBP processes

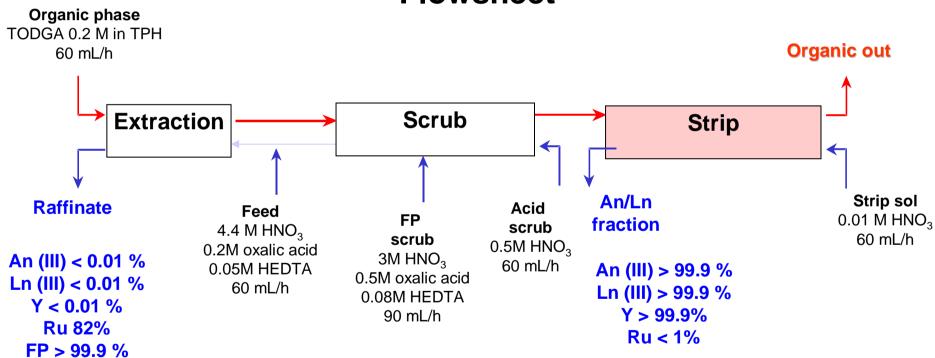


2.5 L of HAR solution !!!



TODGA-BTBP process

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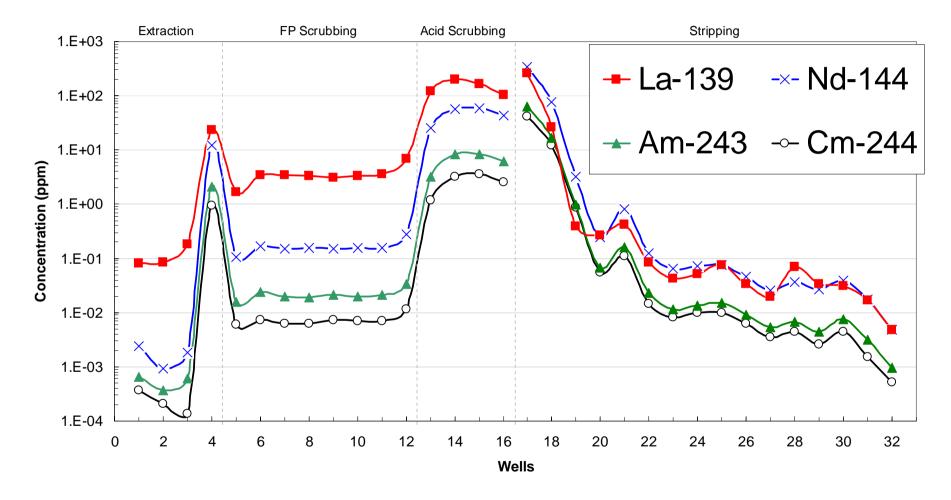


Flowsheet



TODGA-BTBP process

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Ln-An profiles



TODGA-BTBP process

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			Recoveries (%)		
		DF	Raffinate fraction	An/Ln fraction	
	Y	1500	< 0.1	> 99.9	
overy of key	Zr	1	> 99.9	< 0.1	
nents show	Мо	1	> 99.9	< 0.1	
d extraction	Sr	1	> 99.9	< 0.1	
k-extraction	Ru	1.2	82	1	
properties	Pd	1	> 99.9	< 0.1	
	Ln	> 1000	< 0.1	> 99.9	
	Am	40000	< 0.01	99.99	
	Cm	40000	< 0.01	99.99	

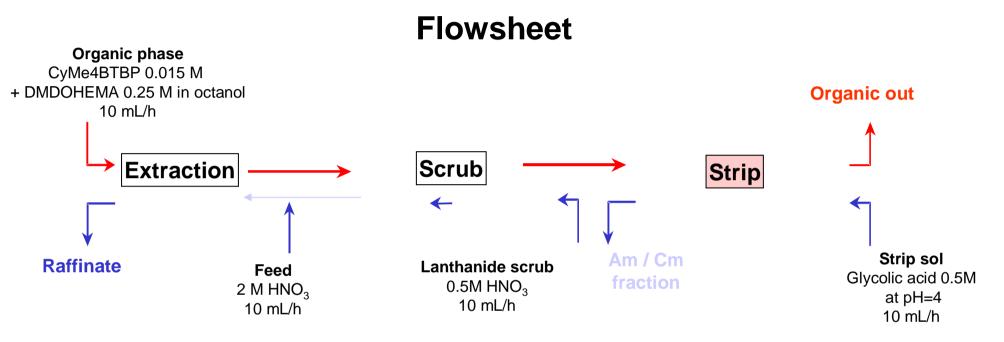
Reco elem

Good and back



TODGA-BTBP process



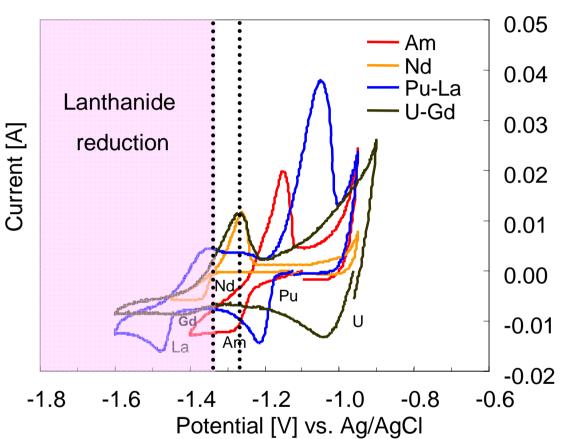


Major problems

- Low kinetics
- Low solubility
- Radiolysis

UROPEAN COMMISSION Cyclic voltammetry on solid aluminium

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✓The difference between MA and Ln reduction is 100 mV.

- ✓ An electrolysis performed at a cathodic potential equal or greater than-1.3 V allows to separate An from Ln
 - ✓ Separation factor of Am from Nd on Al foam

$$SF = \frac{\begin{bmatrix} Nd \end{bmatrix}_{salt}}{\begin{bmatrix} Am \end{bmatrix}_{salt}} = \begin{bmatrix} Am \end{bmatrix}_{Al} \begin{bmatrix} Nd \end{bmatrix}_{salt}}{\begin{bmatrix} Am \end{bmatrix}_{Al}} \begin{bmatrix} Am \end{bmatrix}_{salt} \begin{bmatrix} Nd \end{bmatrix}_{Al}}$$
$$\begin{bmatrix} Am \end{bmatrix}_{Al}$$

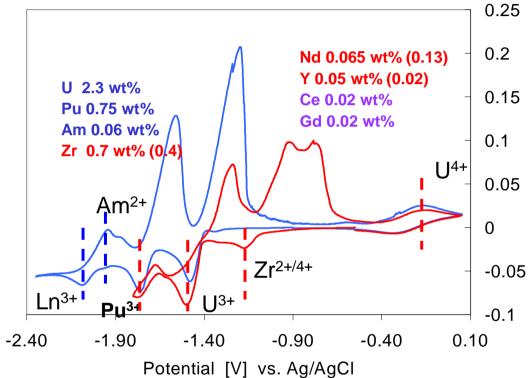
	Nd	Am	Nd	Am	SF
	wt % in salt	wt % in salt	mg in Al	mg in Al	
Before 1 st electrolysis	0.247	0.45			
After 1st electrolysis	0.247	0.12	2.1	125	120
Before 2nd electrolysis	0.249	0.13			
After 2nd electrolysis	0.244	0.03	35	130	40



Current [A]

Cyclic voltammetry of UPuZrAnLn alloy

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LiCl-KCl, $T = 460^{\circ}$ C, v = 100 mV/s, Reference electrode : Ag/AgCl 1 wt%, Working electrode area : ~ 0.2 cm². *Cyclic voltammetry on W in a* solution of An³⁺/Ln³⁺/Zr^{x+} in LiClKCl obtained from U₆₁Pu₂₂Zr₁₀Am₂Nd_{3.5}Y_{0.5}Ce_{0.5}Gd_{0.5} alloy,

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Interpretation of Zr^{+,2+/4+} signal is difficult due to possible multiple valency of Zr,

clear reduction peaks of U^{3+} and Pu^{3+} (most concentrated elements in the salt phase)

Small peak is observed for Am,

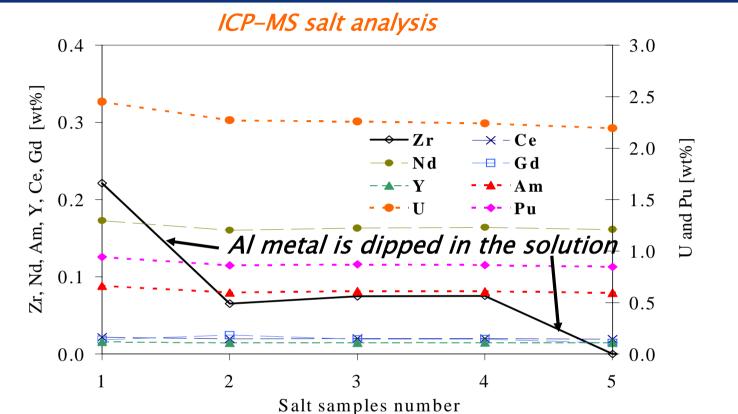
One global peak is observed for Ln.



Extraction of zirconium by Al

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Zr concentration in the salt phase decreases when AI is dipped in the solution. Zr^{+, 2+ or 4+} *ions are reduced by AI metal according to:*

 $Zr^{x+} + Al^0 \longrightarrow Zr^0 \text{ or } Zr - Al_x + Al^{3+}$

Peaks observed by CV attributed to Zr reduction tend to disappear and a signal for Al³⁺ reduction is observed.

EUROPEAN COMMISSION EUROPEAN COMMISSION *batch experiment involving 25 runs (~30g of fuel)* ICTP Trieste 29.11.2007 ADS school

	Wt % in salt					Composition of the dissolved deposit (mg)				
	Before Run 1	After Run 1	Before Run 11	After Run 11	Before Run 14	After Run 14	Run1	Run 7	Run 11	Run 14
U	2.55	2.14	0.25	0.30	0.20	0.30	936	512	223	176
Pu	0.96	1.58	0.65	0.50	0.32	0.27	94.2	243	203	146
Am	0.09	0.17	0.24	0.23	0.19	0.19	4.4	13.5	9.95	8.8
Zr	-	-	0.056	0.096	0.055	0.075	-	-		-
Υ	0.016	0.048	0.074	0.075	0.075	0.078	0.039	0.018	0.015	0.008
Се	0.021	0.047	0.078	0.085	0.089	0.091	0.041	0.026	0.024	0.011
Nd	0.177	0.403	0.59	0.61	0.65	0.65	0.374	0.244	0.183	0.092
Gd	0.025	0.048	0.069	0.073	1.8*	1.8*	0.042	0.025	0.018	0.027
ΑΙ	-	-			-	-	387	301	173	134
	Current density (mA/cm ²)				17	9.3	8.2	6.1		
	Charge (C)		1386	1193	674	598				
* added to the melt as GdCl ₃ Faradic efficiency (%)		cy (%)	91	70	80	70				
	m _{An} /(m _{An} + m _{Ln}) (%)			99.95	99.96	99.9	99.9			
				m _{Am} /m _{Ln}			8.8	43	42	26

• Gd has the lowest electrodeposition potential difference compared to Am

• at Gd conc. corresponding to ~250 runs (300g of fuel) Am/Ln sepation still efficient



Actinide separation from U,Pu,Zr,MA,RE fuels

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- ✓ Selective <u>grouped</u> separation of actinides (U,Pu,Np,Am and Cm) from realistic fuel.
- Adherent and compact deposit are obtained on solid Al with a good faradic yield (~ 90%)
- ✓ Separation from Ln: at a cathodic potential Ec > -1. 2V vs. Ag/AgCl 1 wt% < 2% of Ln are in the An deposit
- separation efficient after 25 electrolyses (> 5g of fuel) of the same salt mixture (more than 6 months of experiments)
- ✓ at Gd (electrodeposition potential close to Am) conc. corresponding to ~250 runs (300g of fuel) Am/Ln separation still efficient





- meet clean air objectives and promote long-term availability of the systems and effective fuel utilization
- minimize and manage the nuclear waste produced (co-recycling of all actinides) and notably reduce the long term stewardship burden in the future, thereby improving protection for the public health and the environment.
- increase the assurance that systems are a very unattractive and least desirable route for diversion or theft of weapons-usable materials.

Change of philosophy

dirty fuel-clean waste instead of clean fuel-dirty waste

EUROPEAN COMMISSION Direct electroreduction of simulated MOX EUROPEAN COMMISSION ICTP Trieste 29.11.2007 ADS school ICTP

PYROmetallurgical processing REsearch Programme

PYROREP

major objective

partitioning of minor actinides (MA) from fuel target or HLW

• participants

CEA – Marcoule, F BNFL – AEA Technology – Harwell, UK CIEMAT – Madrid, E NRI – Rez, Cz ENEA – Casaccia, I CRIEPI – ITU – Karlsruhe, D



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PYROmetallurgical processing REsearch Programme

PYROREP

- consolidate and revive European expertise in pyroprocessing
- obtain basic data to allow conceptual design of processing processes suitable for
 - fuels
 - targets
 - HLLW calcine
- system and performance assessment
- *identify materials usable for industrial implementation*
- asses waste and reactive treatment



- Project full title: EUROpean research program for the PARTitioning of minor actinides from high active wastes issuing the reprocessing of spent nuclear fuels
- ✓ hydrometallurgy (5 WP) and pyrometallurgy (4 WP)
- ✓ duration 4 years; starting date January 2004
- ✓ shared costs
 ✓ total eligible 10.3 mio €
 ✓ EU contribution 6 mio €
- ✓ 25 participants including CRIEPI and ITU
- ✓ co-ordination CEA



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EU EUROPART Integrated Project

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WP N°	Workpackage title	Person- months
1	Partitioning of An from HAR and HAC from UOX and MOX spent fuels (using polyamides, poly N or S molecules and cosan)	233.1
2	Partitioning of An from HAR and HAC from UOX and MOX spent fuels (using calixarenes, or cosan extractants)	165.2
3	Partitioning of An for advanced dedicated fuel cycles (using polyamides, N-S types of molecules and cosan)	99
4	Partitioning of An for advanced dedicated fuel cycles (using calixarene, cosan and calixarene-cosan molecules)	79.8
5	Methods for co-conversion of separated actinides	25.8
6	Pyro Partitioning of An from UOX and MOX wastes	27.6
7	Study of the chemistry of trans-Cm elements in molten salts and definition of pyro processes for advanced dedicated fuel cycles	56.7
8	Study of the conditioning of the wastes issuing pyro processes	45.1
9	Pyrochemical system studies	18
	Total	750.3



METAPHIX irradiation experiment Irradiation of U,Pu,Zr,Ln,MA alloys

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3 capsules;

3 different compositions

under irradiation since November 2003 in the PHENIX reactor in Marcoule

> 1. 90 PEPD 1.7 % burn-up unloaded: 8. August 2004 transport to ITU 25. September 2006 NDE performed

- 2. 3 cycles of 120 PEPD 7% burn-up unloaded: 20. July 2006
- 3. 5 cycles of 120 PEPD 11 % burn-up to be unloaded beg. 2009

work initiated by CRIEPI, Japan; now trilateral project CRIEPI, CEA, ITU







Objectives

- Reprocessing of the metallic fuels U, Pu, Zr with up to 5% An's and Ln's
 - un-irradiated material from the fuel production
 - irradiated METAPHIX target fuels after irradiation in PHENIX reactor
- Study processes on calcined and chlorinated HLW from PUREX type waste
 - Electrorefing
 - Molten salt liquid metal extraction
- Study of direct electroreduction processes for the head-end conversion of oxides to metal

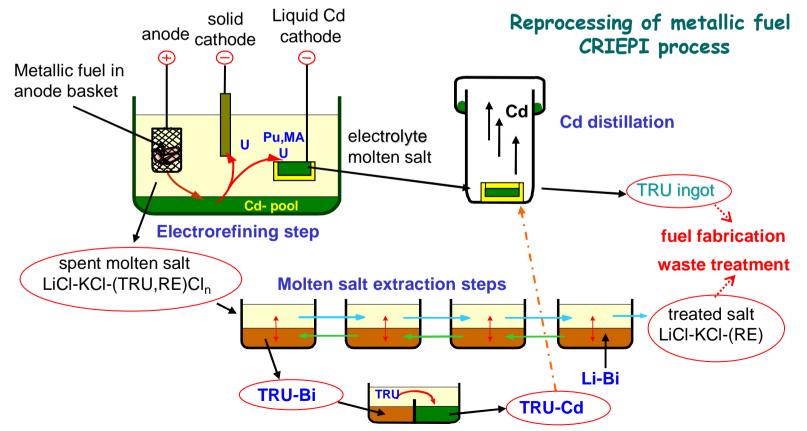




Pyrometallurgical Processes CRIEPI-ITU collaboration

⇒ Main Tasks

- Installation of GBs and hot cell for experiments
- Setting-up of necessary equipment
- Colds test using U, Pu and fresh metallic fuels
- Hot tests using fresh and irradiated fuels (METAPHIX) & HLW
- Optimisation of the process





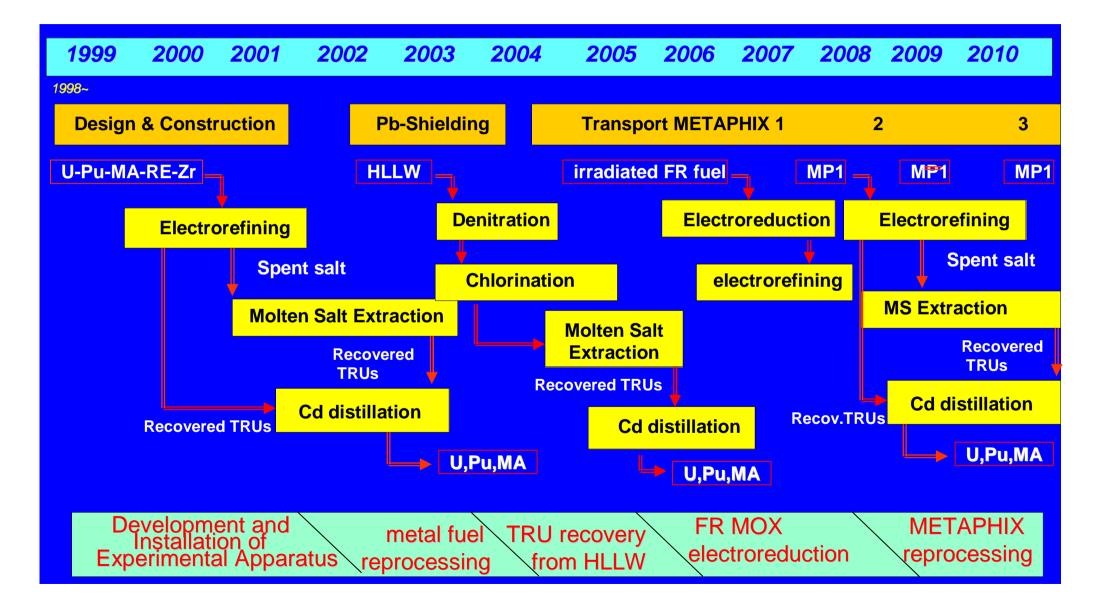
Alternative Fuel Cycles

Pyroprocessing: Tentative Test Plan



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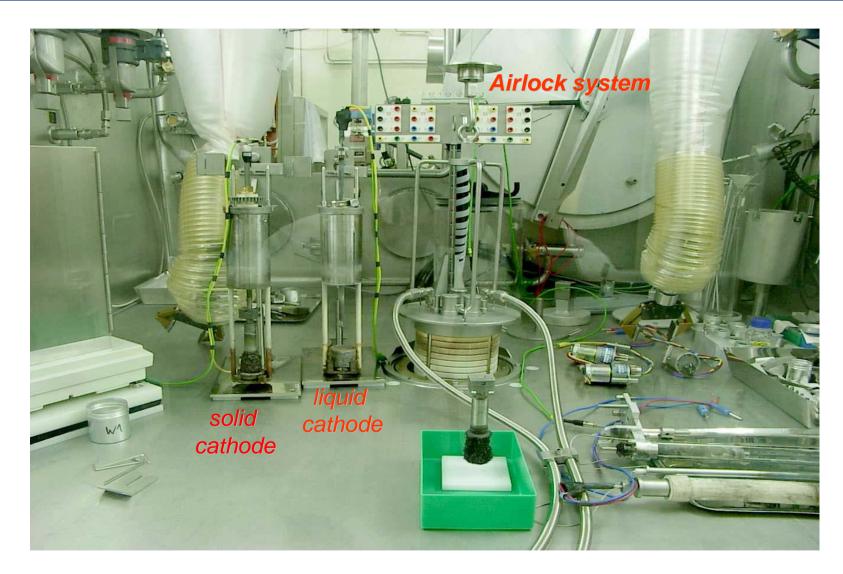
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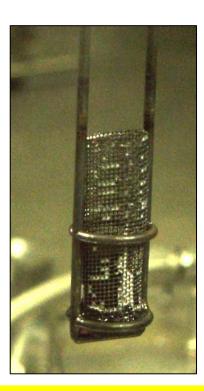


inside view of the electrorefining caisson



Pyrometallurgical Processes CRIEPI-ITU collaboration

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anode basket loaded with U metal (60g)

solid cathode with U dentrite deposit

U electrodeposition





salt sample

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electrodeposition parameters: . constant current: 500 mA . time: 4 hours

. temperature: 773K

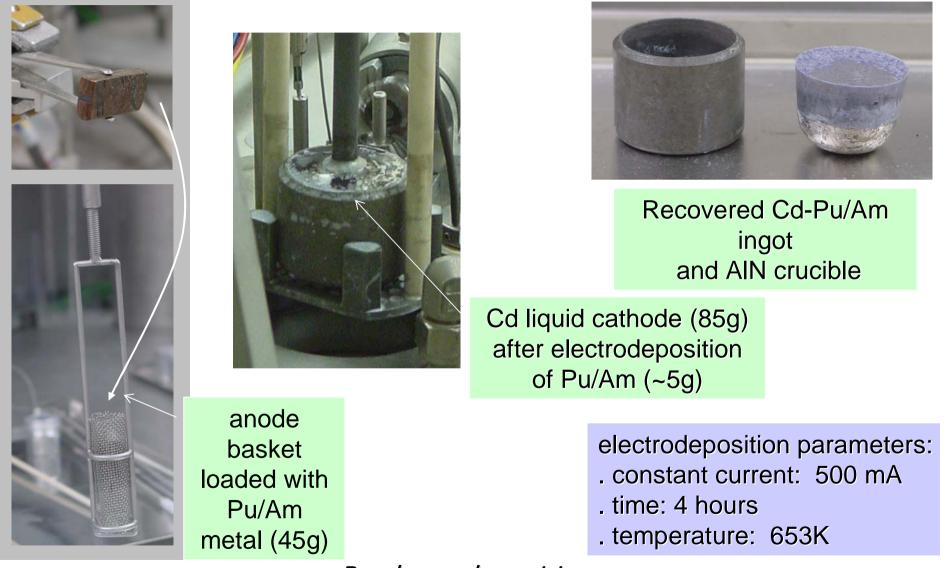


U-Salt sample



Pyrometallurgical Processes CRIEPI-ITU collaboration





Pu electrodeposition



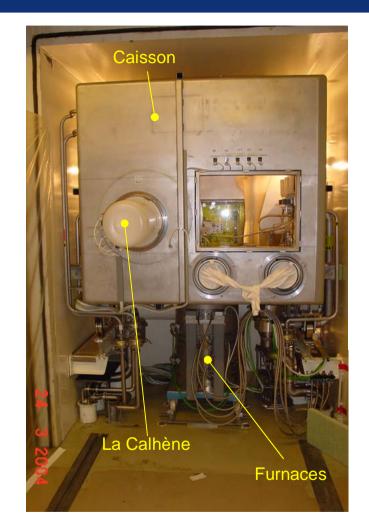
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- ⇒<u>Phase 1:</u> Installation of the equipment in an alpha laboratory
 - * Adaptation & testing of equipment
 - * U & Pu runs
 - * UPuZrMA runs
 - *** Reductive extraction**

Atmosphere in caisson: Ar with O₂ and H₂O concentrations < 10ppm respectively



 \Rightarrow <u>Phase 2:</u> Installation of the equipment in a betagamma laboratory (15cm lead shielding

* Hot tests with HLW & irradiated METAPHIX metallic fuel



Pyro-reprocessing cell

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Installation of Caisson (2003)

Introduction of material through an Ar flushed LaCalhène door (2005)



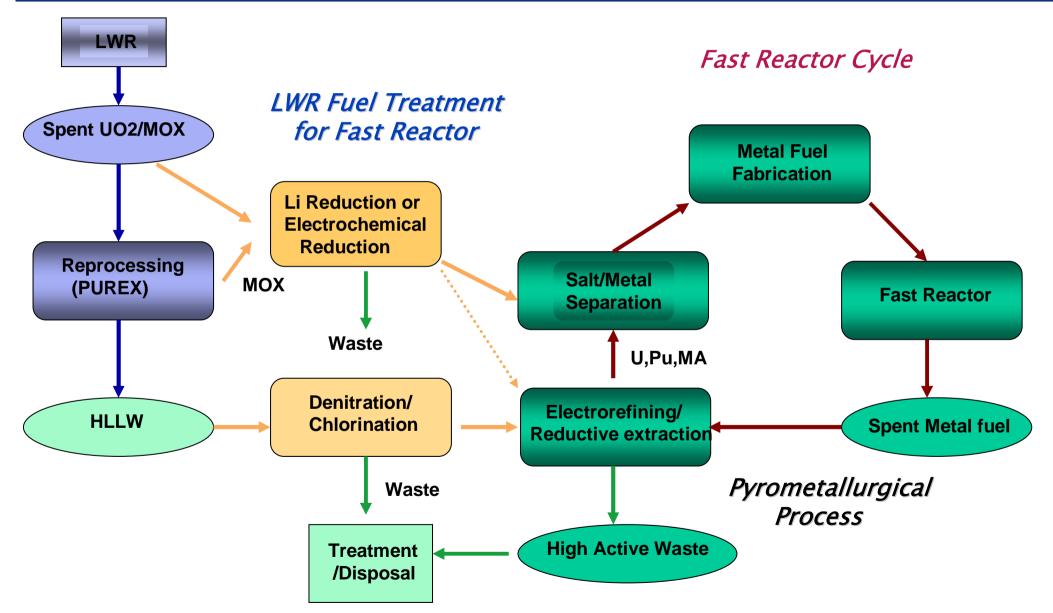
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Combined Oxide-Metal FR Fuel cycle Actinide Recycling

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Raffinate from Purex reprocessing of commercial MOX. • *Pu/(U+Pu)=4.9% before irradiation*

• *Burn up: ~ 30GWd/t*

*To better observe the behavior of Np and Pu, HLW from reprocessing of LWR UO*₂ *fuel and FR MOX fuel were mixed to form the raffinate.*

Final composition

- •*U: 8400 µg/g*
- TRU: 600 µg/g

• Fission products: 2000 $\mu g/g$, including 900 $\mu g/g$ of rare-earth elements





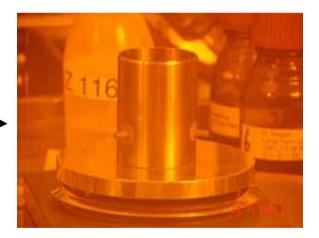
The mixed HLLW was concentrated in a glass flask in an air atmosphere hot cell. Then, the concentrated solution was poured into a stainless steel crucible and heated on a hot plate to remove most of the water.



Concentration of HLLW



HLLW after concentration

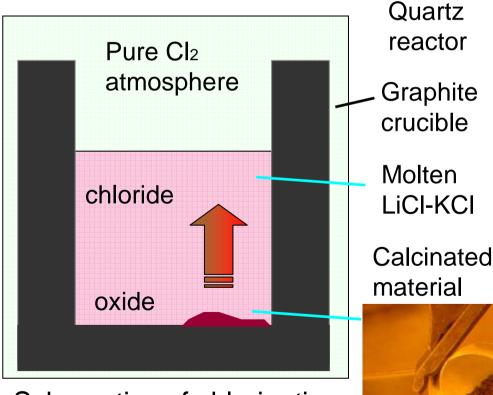


Heating on a hot plate





The calcinated material is converted to chloride under pure chlorine gas atmosphere at 650°C . The chlorination is performed in the argon atmosphere hot cell.



Schematics of chlorination



Quartz reactor for chlorination

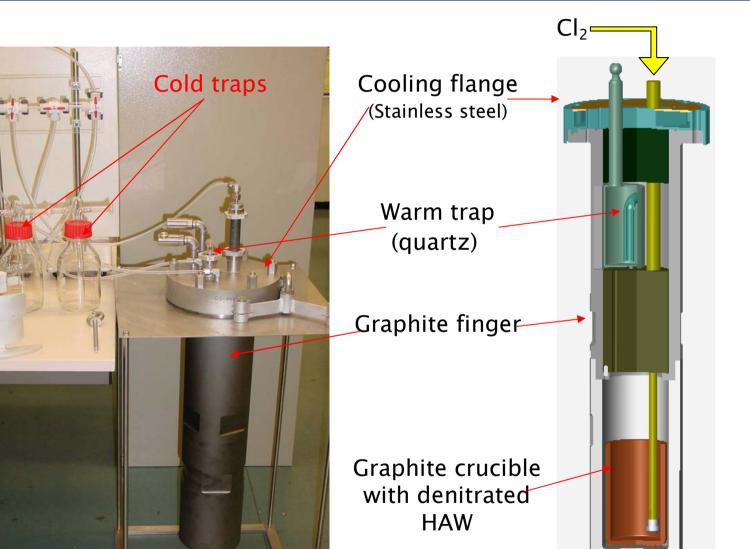


Central Research Institute of Electric Power Industry



Chlorination device



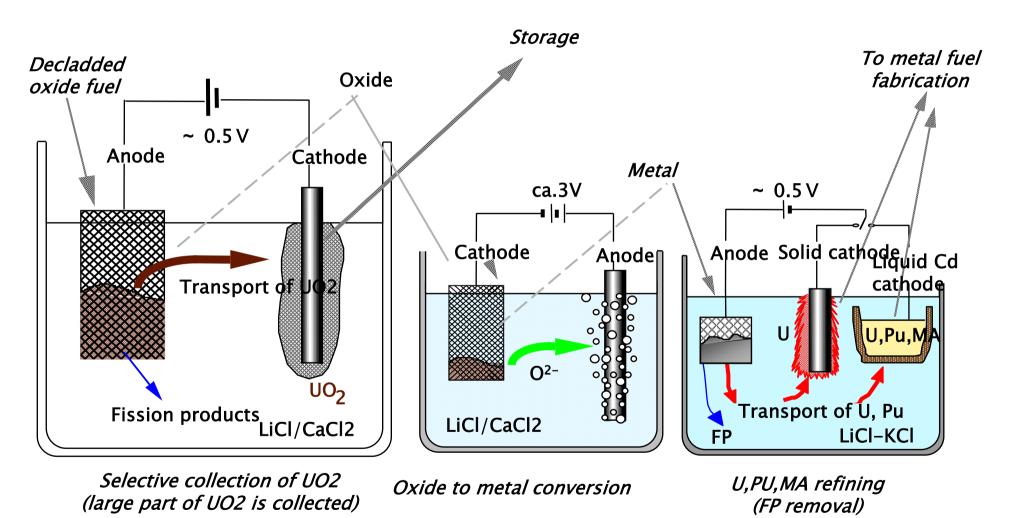




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Spent oxide treatment by pyro-porcess with UO₂ separation



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Lithium Reduction Process

reduction using lithium metal in a molten LiCl salt at 650 °C; major concern in this process is the carryover of unreduced rare-earth oxides into the electrorefiner.

Calcium Direct Oxide Reduction Process (DOR)

used for many years in production of plutonium metal from oxide using calcium metal in molten $CalCl_2$ or $CaCl_2$ - CaF_2 at 850°C; the drawback is the high temperature

Direct electroreduction

new process with a lack of fundamental data for actinide oxides; efficiency for the reduction of unirradiated oxide fuels to be tested followed by the reduction of irradiated oxide fuels. Important parameters to investigate are the influence of temperature, salt bath composition, potentials.



Direct electroreduction of oxide fuels

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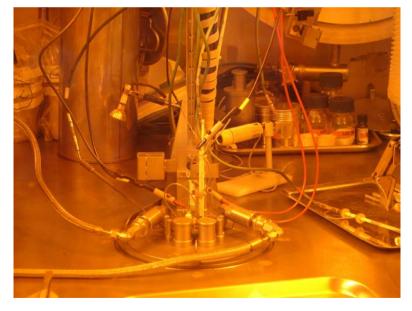


Experiments ongoing using SUPERFACT irradiated fuel, fabricated by ITU: 2 pieces of $(U_{0.74}Pu_{0.24}Am_{0.02})O_2$ and 2 pieces of $(U_{0.74}Pu_{0.24}Np_{0.02})O_2$ Crucible material: MgO Electrolyte: LiCl + Li₂O (T=650°C)





Set of electrodes

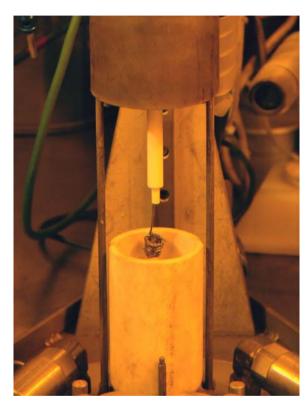


Setup inside the pyro caisson

Direct electroreduction of irradiated oxide fuels



Direct electroreduction of oxide fuels



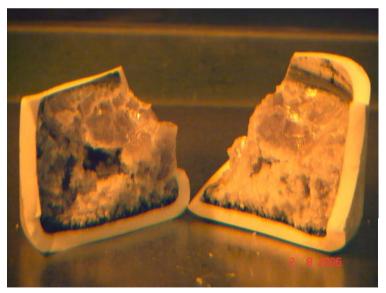


Salt samples taken from the top and bottom (incl. black fuel deposit) of the crucible

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fuel loading

Crucible showing a black reduced fuel deposit on the bottom



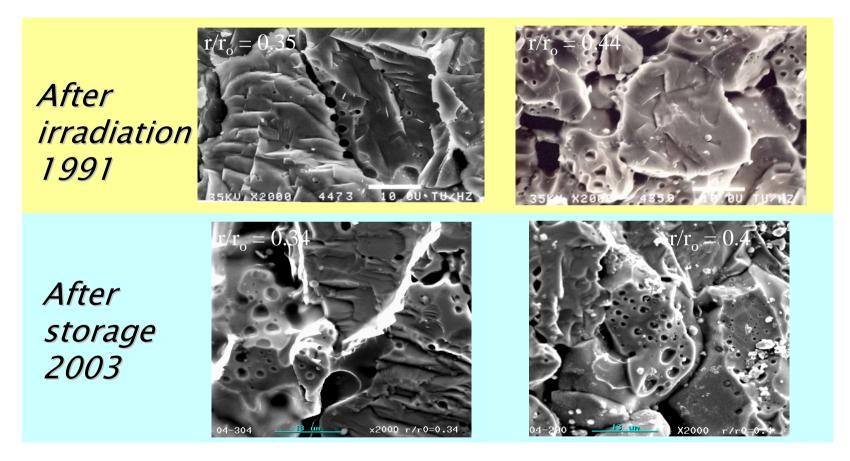
Direct electroreduction of irradiated MOX



Direct electroreduction of oxide fuels

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SEM examination of SUPERFACT fuel showing a strong intergranular porosity due to fission gas release



- mature for PUREX to be extended to advanced aqueous
- development of destructive and non-destructive assay
 techniques for inventory and flow verification purposes, including new type of fuels containing minor actinides, *Np-237, Am-241, Am-242*

IAEA and EURATOM: effective flow sheet verification (FSV) at ITU to update the existing design information as provided for safeguarding U and Pu, and to provide additional information relevant to Np

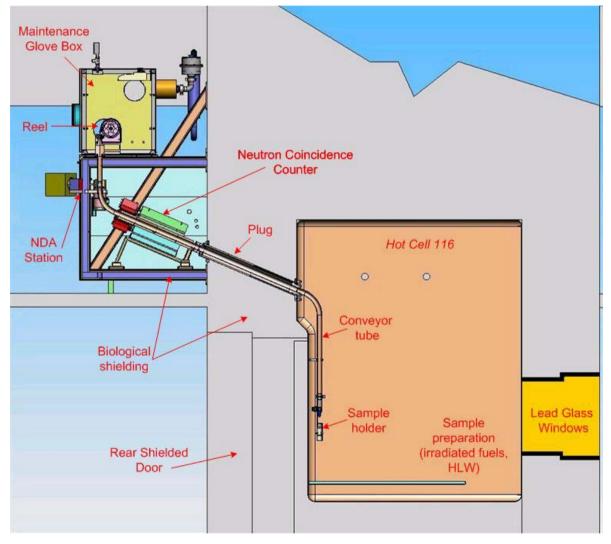
- proliferation and safeguards issues are far less defined • and investigated for pyroprocessing; new schemes, approaches and analytical techniques have to be developed
 - K– X ray Fluorescence (K–XRF)
 - Neutron Coincidence Counting (NCC)
 - High Resolution Gamma Spectrometry (HRGS)
 - Calorimetry



Pyrometallurgical Processes

new analytical equipment

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Objective: to install NDA assay system for a direct non-destructive assay on high active samples

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Advanced NDA assay system connected to a hot cell

Advanced Nuclear Fuel Cycle

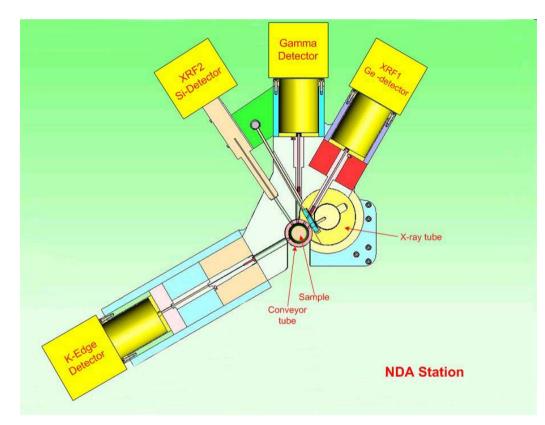
New analytical equipment for Pyrometallurgical Processes



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JRC

EUROPEAN COMMISSION



Advanced NDA assay system detector arrangement



Testing of equipment Status of the project

- installation for testing in A117
- x-ray and detectors to be installed and tested



Operations at the front end or the tail end of the main process, where research and development are also mandatory:

- preparation of the feed stream to ensure compatibility with the core process (e.g., de-cladding of solid fuel, halogenation of oxide fuel, and dissolution in molten salts)
- Purification of the output stream with recycling of contaminants back into the core process and treatment of the process and conditioning of waste materials in accordance with their chemical and radiological properties (e.g., vitrification)
- Decontamination of technological waste (e.g., crucibles) with recycling back into the core process and conditioning of the actual waste materials (e.g., melting of scrap metal)
- Decontamination of all process off-gases with recycling of contaminants back into the core process.





- complete the basic thermodynamic and electrochemical data especially Cm (collaboration with RIAR Dimitrovgrad in an ISTC project)
- further develop head-end and back-end treatment in view of large scale process development
 - techniques for cathode reprocessing
 - direct electroreduction
 - waste treatment
- electrorefining in molten fluoride systemes (expertise Rez)
- ACSEPT project in the European 7th FP

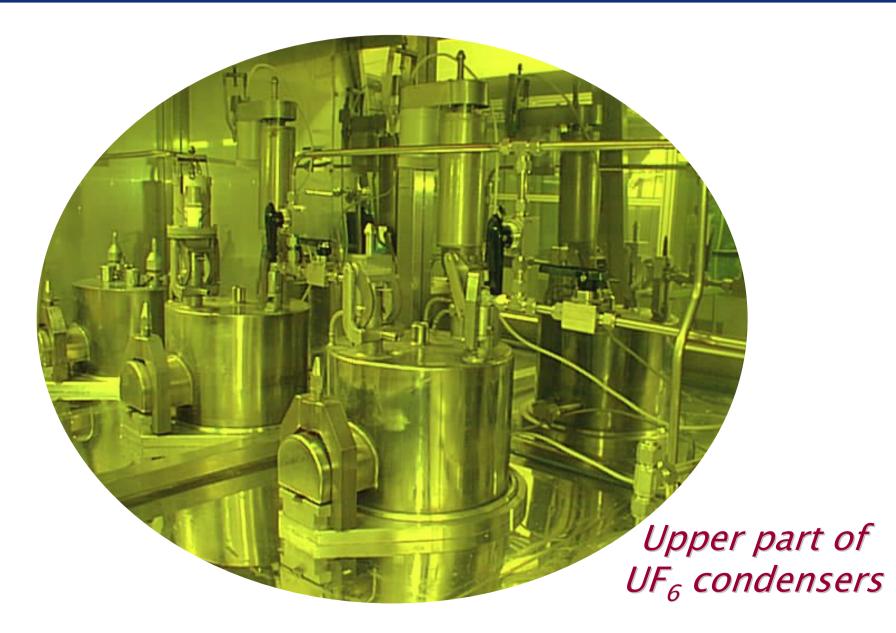


 develop a safeguarding technology for a pyrometellurgical process





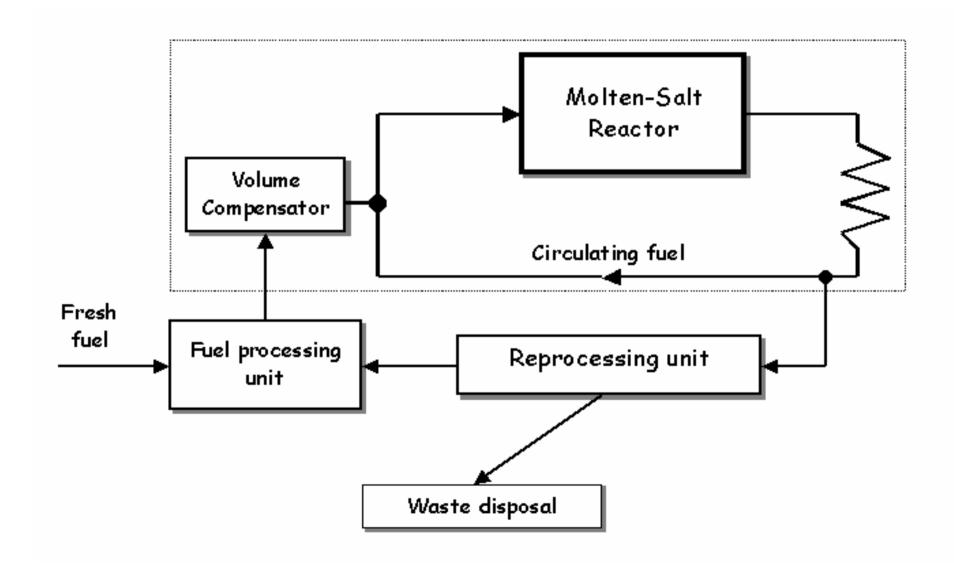
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Main groups of elements, which have to be extracted from the fuel (primary) circuit of the MSTR

- Noble gases (Xe, Kr)
- Noble metals (Nb, Mo, Ru, Sb, Te), which tend to leave carrier molten salt
- Lanthanides (rare-earths) forming stable fluorides in molten salt medium
- Actinides forming stabile fluorides in molten salt medium. (However TRU elements have to be returned back into the fuel circuit)



Research defined in the frame of the European MOST project

- ✓ Molten salt fuel processing (liquid-liquid extraction, fluorination, ...)
- Most of the data are not measured (only calculated) : need of measurement of the static and kinetic parameters (Decontamination factors, Gibbs energy, ...)
- ✓ Reprocessing will be done by small batch during MSR operation
- ✓ One reprocessing unit for one site (3 or 4 MSR) having a capability of 1 to 3 m³ of fuel salt per day.
- ✓ Reprocessing part of MSR has a strong impact on economy of the system





New European Research Area concept N P&T integrated projects EUROPART and EUROTRANS ACTINET network *▶ Increase training, education, exchange of* scientific staff ▶ Pooling of facilities *▶ EURATOM participation in GIF (charter signed on 30.6.2003)* Note: No A develop fuel cycles including safeguards *implified PUREX + advanced aqueous → Mary reprocessing techniques*

development of advanced fuels including inert matrix fuels and targets



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✓ Contract with CRIEPI in support to the start-up of the reprocessing in Rokasho: ✓ successful cold tests on sludge formation upon fuel dissolution, ✓ possible follow-up projects until 2012 ✓ demonstration of the TODGA MA extraction process ✓ collaboration with FZ Jülich ✓ implementation of HFM extraction equipment ✓ successful MA separation from chlorinated PUREX raffinate ✓ extraction by liquid Bi from molten salt

✓ new partitioning project ACSEPT 2008–2012

✓ ITU contributions on hydro– and pyro–reprocessing (~750 k€) ✓ participation in the PATEROS project to prepare the

SNETP platform





Hydro-pyro

 No competition but complementarity between aqueous and pyrochemical techniques



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Advanced and extended PUREX in the LWR cycle for separation of all actinides

Industrial implementation in progress

Pyroreprocessing in the advanced FR cycle for
 grouped actinide recycling

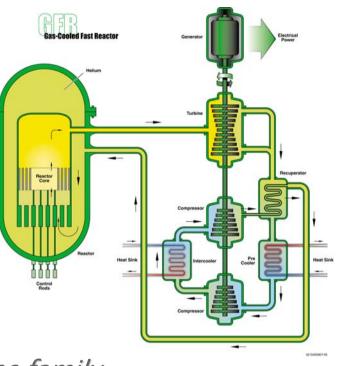
Demonstrated on laboratory scale



CEA R&D on GFR fuel reprocessing

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- Fuel dissolution
 - *k* First tests to dissolve selected materials in aqueous media and molten salts
 - *k* Evaluation of mechanical pre-treatment (burn & crush, sonic waves, ...)
- Grouped Actinides recycling
 - M. Hydro
 - Calculation of a separationscheme to meet required extraction performances
 - *R&D on specific extractant for the actinides family*
 - In Pyro:
 - Hot cell tests of actinides electrochemical recovery in molten chlorides
 - R&D on the liquid-liquid extraction with a metal



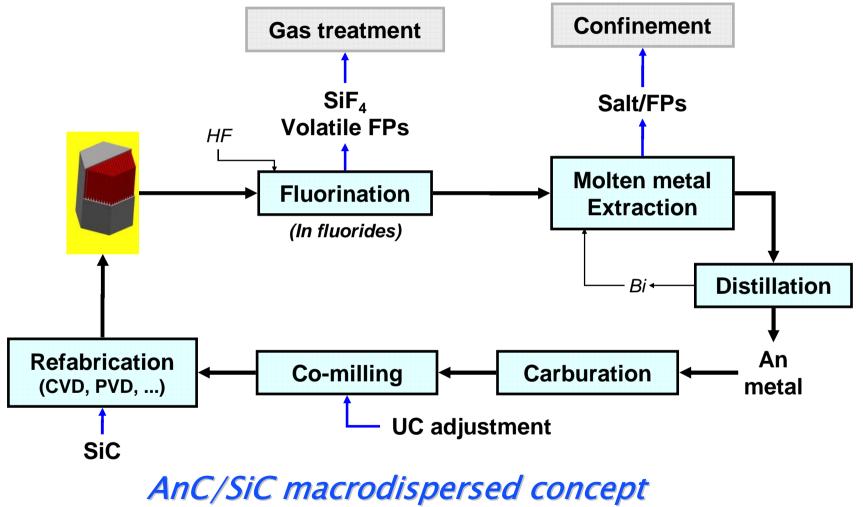


- Mead-end process and fuel dissolution in molten salt difficult, especially in the case of coated particles
- Conversion processes (of e.g. ThO₂ or ThC) if metallic concepts have to be applied
- 231 Pa problem; can possibly be transferred to the waste treatment part of the process
- *▲ difficult fuel fabrication also due to ²³²U*



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Pyrochemical treatment



Performed in close international collaborations, the research work should focus on the following major topics:

- completion of thermodynamic database on actinide containing fuels for molten salt systems
- Testing and evaluating advanced dry reprocessing techniques including scale-up
- Installing new large scale facilities for pyrochemical reprocessing
- Developing supporting analytical techniques in combination with a safeguarding process