EUROPEAN COMMISSION
Community Research

SEVENTH FRAMEWORK PROGRAMME

CARBOWASTE

Treatment and Disposal of Irradiated Graphite and Other Carbonaceous Waste

Grant Agreement Number: FP7-211333

T-3.1.1 & T-3.1.2

Sample Management

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Document Number: CARBOWASTE-1306-T-3.1.1-2
Date of issue of this report: 01/06/2013

Start date of project: **01/04/2008** Duration: **60 Months**

Project co-funded by the European Commission under the Seventh Framework Programme (2007 to 2011) of the European Atomic Energy Community (EURATOM) for nuclear research and training		
Dissemination Level		
PU	Public	
RE	Restricted to the partners of the CARBOWASTE project	
CO	Confidential, only for specific distribution list defined on this document	**X**

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CARBOWASTE		
Work package: 3 **Task:** ^1.1	**CARBOWASTE document no:** CARBOWASTE-1306-T-3.1.1-2 (e.g. May 2008 as date of issue: 0805)	**Document type:** T = Technical
Issued by: Ciemat (SP) **Internal no.:** CW1304-Technical Report 3.1.1-2		**Document status:** Final

Document title

Sample Management

Executive summary

This document presents the Sample management plan and the results within the project joint in the same document the CW-T-3.1.1 and T-3.1.2.

In the Sample management plan it is included the strategy to establishing the availability of samples from different suppliers and reactor types as well as it is compiled the virgin and irradiated graphite samples assayed within the WP3 and 4 and the original reactors classified by reactor types: UNGG. MAGNOX, RBMK, MTR or HTR and the users by each partner.

It is adopted the decision to distribute samples from three reactors reactor core of Saint Laurent A-2 (UNGG) by EDF, reactor core of Oldbury-2 (Magnox) by NDA and graphite from Thermal Column of TRIGA-Pitesti (MTR) by INR. Additionally the samples of CW-RRT were wide distributed graphite within Carbowaste and also are considered, these comes from sleeves of Vandellós-1 (UNGG) supplied by ENRESA.

A description of the reactors and the samples used in the project is also included in section 4.

Management procedures for the selected samples are detailed in section 4. In some cases with the chronology and the transport requirements. Some examples of sample management at the Labs and topics to be covered in the standard procedures are given in order to improve the reliability of the results as leason leaned.

The use of the samples in the different tasks of WP4 and WP5 is also detailed by reactor and beneficiary.

Finally main achievements in the Sample Management are listed in section 5.

Revisions

Rev.	Date	Short description	Author	Internal Review	Task Leader	WP Leader
00	dd/mm/yyyy	Issue	Name, Organisation *Signature*	Name, Organisation *Signature*	Name, Organisation *Signature*	Name, Organisation *Signature*
01	01/06/2013	First issue	G, Piña Ciemat	E. Magro Ciemat	G, Piña Ciemat	G, Piña Ciemat
02	dd/mm/yyyy	2nd Issue				

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1. INTRODUCTION

1.1. Objectives of Task

The sample management in the WP3 and for the other WPs was use for the establishment links between partners, taking into account the natural relationships between suppliers and characterisation labs for shifting radioactive samples in the most effective and inexpensive way

1.2. Scope

The sample management cover both domestic and inter-partners distribution of samples to rationalise synergetic approaches and trying to cover the maximum type of reactors graphite. In this propose graphite from MTRs, HTR, RBMK, UNGG and Magnox reactors. Aditionally the distribution of samples from the inter-comparison Test (CW Round Robin Test) can be involved in the scope of this task.

1.3. Structure

The document is divided in order to describe:

- The organisation of samples transfer (Section 2)
- Streams and original reactors and sampling methods (Section 3)
- Management Protocol and some examples of Management and in-lab methodologies for sampling, packaging, delivery, and sample reception, interim storage and preparation are given. (Section 4)
- Use of the samples tested in WP3 and WP4 (Section 5)

2. Samples Management Plan

For the experimental work on WP3, WP4 and WP6 of Carbowaste it is necessary to establish a management of the virgin and irradiated graphite samples from the reactor within the project for both:

To obtain detailed knowledge of the graphite to be manage for any phase of the waste life cycle from retrieval, treatment, conditioning, storage or final disposal for the establishment inventory. This data are useful to establish the retrieval strategy, to demonstrate the treatment capabilities and to feed with reliable data the safety assessment of the interim or final disposal of the i-graphite. This first objective use to be cover by the experimental groups within the graphite sampling performed in the domestic reactors of any State Member.

On the other hand Carbowaste give an opportunity to develop and to put in common the developed technologies for any stage of the life cycle, to contrast and to review and to improve on the accuracy and effectiveness of these methods. For this reasons it was impulse the interchange of different graphite grades among the beneficiaries in order to inter-compare results, go into deeper knowledge of their characteristics and behaviour at different conditions.

This particular and global knowledge of the graphite give elements for waste management strategies and options (WP1) and obviously are useful for retrieval technologies (WP2) and reuse of recycling the carbonaceous waste streams (WP5)

Therefore an effort to equilibrate the knowledge of graphite streams within the project was done in order to compile the data of the distribution of samples and to complete and help the searing of samples among the beneficiaries with experimental work involved.

In order to know the availability of samples and the natural suppliers and receptors a questionnaire was distribute among WP3 and 4 members (Appendix 1) to build up a double entrance table.

Table 2.1. Samples distribution

REACTOR		ORIGIN	SUPPLIER		FZJ	CEA	CIE	ENE	FTMC	INR	SCK	JRC	UOM	RRT
UNGG	SLA-2	FR	EDF	V		X	X							
				I	X	X	X							
	G2	FR	CEA	I										
	VD-1	SP	ENRESA	V			X							X
				I			X							X
MAGNOX	OLDBURY-2	UK	NDA	I	X	X	X	X					X	
	WILFAX	UK	NDA	I										
	LATINA	IT	SOGIN	I		X		X				X		
RBMK	IGNALINA	LIT	IGNALINA NPP	I	X				X					
MTR	TRIGA	RO	INR	V			X			X	X		X	
				I			X			X	X		X	
	JEN-1	SP	CIEMAT	V			X							
				I			X							
	BEPO	UK	UKAEA	I									X	
	MERLIN	GE	FZJ	I	X							X		
	DIDO	GE	FZJ	I	X									
	BR-1	BE	SCK	I							X			
HTR	AVR	GE	FZJ	I	X							X		

REACTOR		ORIGIN	SUPPLIER		ENS	PBMR	NECSA
UNGG	SLA-2	FR	EDF	V	X		
				I	X		
MAGNOX	OLDBURY-2	UK	NDA	I	X		

Besides the natural flow of samples It was agreed that the graphite coming from a UNGG reactor, a Magnox reactor and a MTR reactor had to be spread among the maximum lab receptors due to the importance in terms of mass/volume and the availability.

The selected UNGG reactor was Saint Laurent A2. EDF supplied, through CEA, samples from the core as is detailed in the section 3. (CEA, FzJ, ENS, and CIEMAT)

Magnox Oldbury-2 i-graphite is the one that was supplied to the interested partners.(UoM, FzJ, CEA, ENEA, ENS and CIEMAT)

MTR graphite selected was the TRIGA reactor at INR Pitesti and was delivery at

Triga reactor from INR Pitesti was the MTR type i-graphite selected for sharing among partners interested. The partners that studied this samples were CIEMAT, UoM, SCK·CEN and INR

RBMK Ignalina reactor graphite was studied at FzJ and FTMC.

The following sections described the reactors and samples studied and the procedures to manage the sample inside the lab as a guidance.

3. Samples investigate

3.1. SAMPLES OF material testing reactors (MTR)

3.1.1. TRIGA Reactor

The thermal column of TRIGA reactor is a graphite block with dimensions 1716 x 1144 x 710 mm; this is formed by 96 rectangular graphite cells (12 rows x 8 bricks) in aluminium cladding, with dimensions 144 x 144 x 770 mm, placed in the reactor pool. A number of graphite cells are already removed from the thermal column and stored.

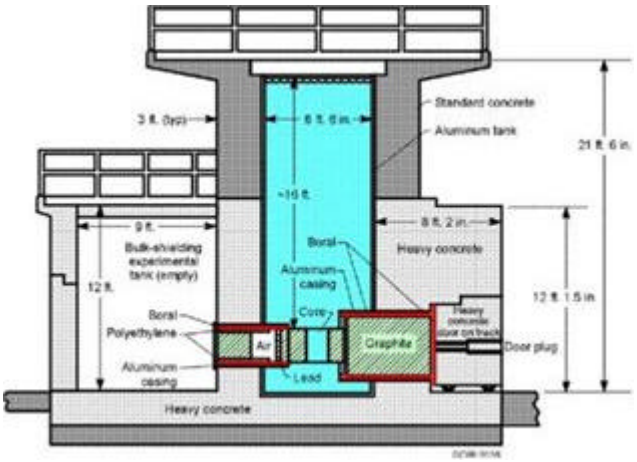

Figure 3.1 Triga Reactor

INR investigated samples of i-graphite arising from thermal column of material testing TRIGA reactor. In the first stage INR has investigated both sample of virgin and irradiated graphite. For the characterization of virgin graphite, polished samples have been mechanically prepared for elemental analysis and morphology investigations. In order to characterize the i-graphite, into a sealed niche the powder samples were mechanically prepared from different locations of a cell removed from thermal column.

Figure 3.2 shows a cross-section of the graphite cell used in TRIGA reactor. P1 – P8 represent the locations selected for i-graphite samples preparation in view of characterization.

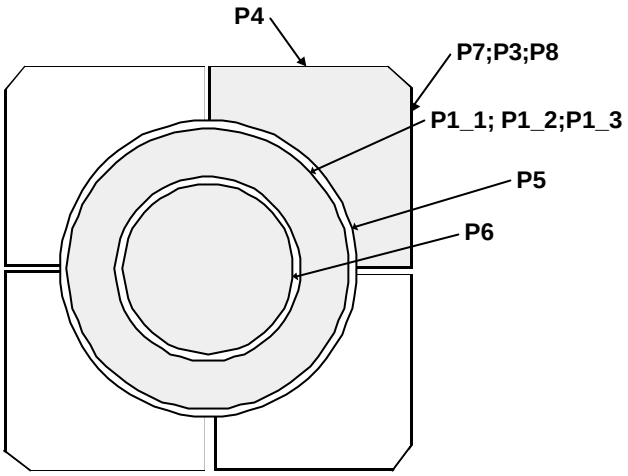

Figure 3.2: A cross-section of TRIGA reactor graphite cell

In this graph we see that a cell is constituted from a central cylindrical piece, followed by a pipe piece and finally four identically components, one for each cell corner. These four components have at outside a rectangular form and at the inside a concave form.

The pieces selected for preparation of irradiated samples were: a central cylindrical piece, pipe piece and one of the four pieces located in the cell corner were chosen. For the characterization of ŧgraphite from these three main components of the cell the following samples was prepared:

- One sample from central cylindrical piece (the sample codified P6);
- Three samples from external surface of pipe piece; the height of pipe was divided in three equal parts and from the upper part sample P1-1 was prepared, from the middle part sample P1-2 was prepared, and from the bottom part sample P1-3 was prepared;
- Five samples from the piece located in the cell corner as follow:
  - One from the middle part of cylindrical inside surface: sample P5;
  - Three from external surface of the piece: sample P7 from upper part, sample P3 from the middle part and sample P8 from the bottom part;
  - One from another external surface of piece: sample P4.

Powder form of these nine radioactive samples was chosen on account of its homogeneity..

A block of irradiated (low activity content) graphite from TRIGA reactor from Pitesti (Romania) was received at **CIEMAT** as part of the shearing samples in the project.

Figure 3.3 Sample of irradiated graphite from Triga

Table 3.1. Samples of MTR reactors

Reference	Origin	Dose rate (µSv/h)	Mass (g)	Remarks
INR-iG	INR	-	100	Rectangular Prism 9 x 6 x 4,5
JEN-I-iG	Ciemat	33	600	Irregular pieces

3.1.2. MERLIN (FRJ-1) and DIDO (FRJ-2)

At the research centre of Jülich (**FzJ**) two types of material test reactors were in operation: the MERLIN reactor or FRJ-1, a light water moderated swimming pool reactor, and the DIDO reactor or FRJ-2, a heavy water moderated reactor. During the dismantling process of the MERLIN reactor and in preparation of the dismantling of the DIDO reactor graphite samples of the thermal column (see Figure 3.4 left) and of the graphite reflector (see Figure 3.4 right) have been taken.

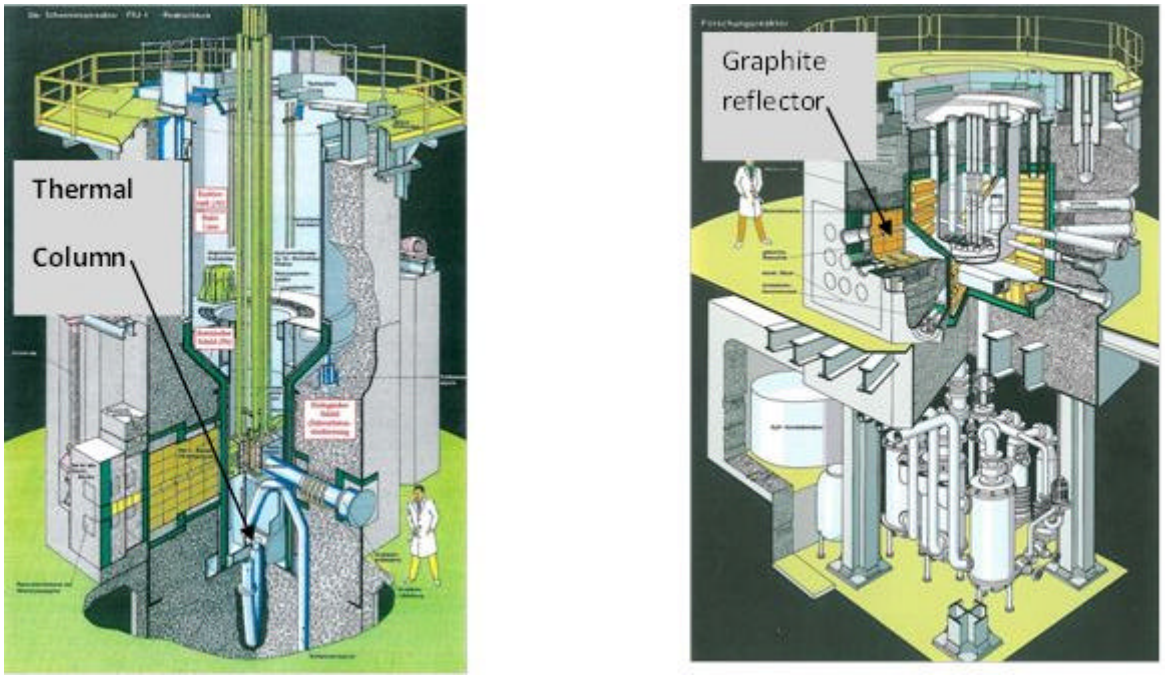

Figure 3.4 Schematic view of the reactors MERLIN (FRJ-1) and DIDO (FRJ-2)

3.1.3. BEPO Reactor

The British Experimental Pile Zero (BEPO) Energy Reactor was commissioned in 1948 and closed in 1968. BEPO was graphite moderated, air cooled and initially fuelled with natural uranium metal in aluminum cans. Two grades of enriched uranium fuel were used at a later date within the reactor. BEPO was initially used for the production of plutonium, but later this function was transferred to the Windscale piles with BEPO becoming a research reactor[3]. BEPO's role as a research reactor included isotope production, studying the irradiation behaviour of graphite and it was also used in studies for coolant compositions for the Magnox and AGR reactors. Figure 3.5 shows a photograph taken of the BEPO reactor.

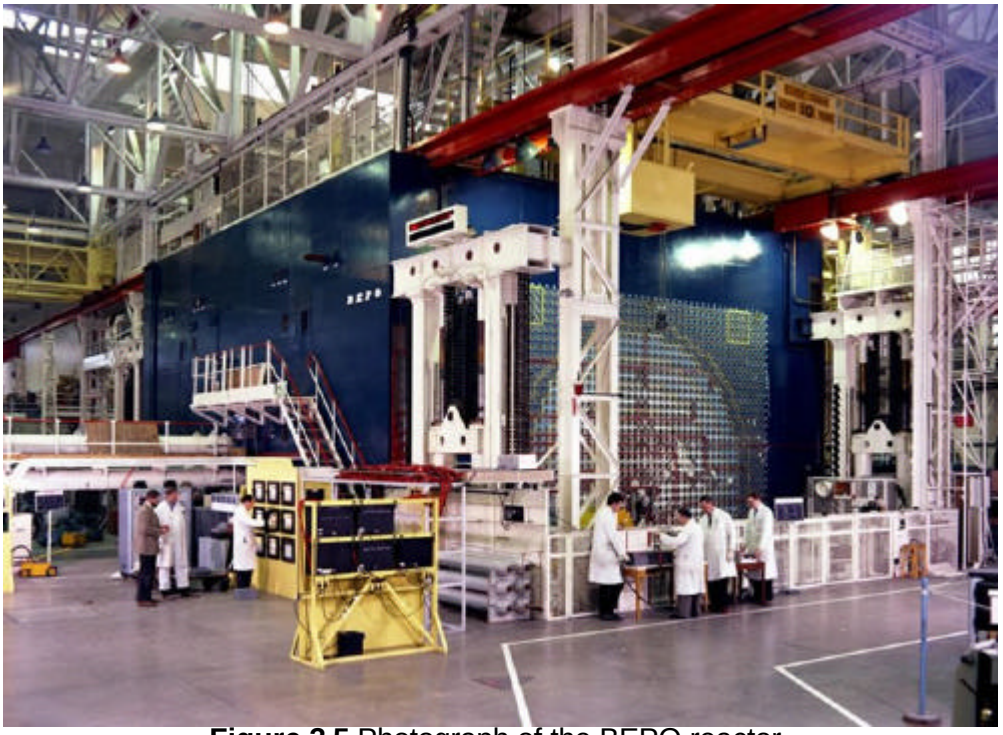

Figure 3.5 Photograph of the BEPO reactor.

In 1975 a four inch cylindrical sample was trepanned from the BEPO core, through the control face of the pile, steel shielding, concrete bioshield and the 20 columns of graphite blocks [95]. From this core the samples used for analysis at The University of Manchester were taken. Sampling was initially carried out in 1975 to provide radioisotope data on the graphite, steel and concrete, and to evaluate the Wigner energy. It was retrieved using a diamond-tipped hole cutter using no coolant or lubricant. After retrieval the core was re-sealed and the reactor remains this way at Harwell to date [4]. Figure 3.6 shows the fluence of neutrons experienced by each channel [4].

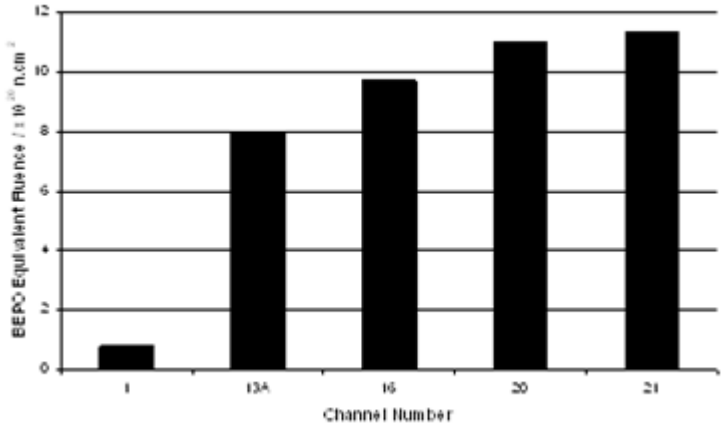

Figure 3.6 Graph of BEPO neutron Fluence against channel number.

The sample number indicates the channel from which the graphite sample was taken, therefore sample 1 (channel 1) was furthest away from the centre of the core and sample 21 (channel 21) was at the centre of the BEPO reactor core. Figure 3.6 shows graphically the total activity measured for each sample data provided by Nirex [5].

These two sets of measurements allow an overall view to be formed of how active each BEPO sample is 40 years after reactor shutdown. A clear observation would be to note the very low activity associated with BEPO 1 samples compared to the other channels which are further into the centre of the reactor core. Whenever possible a sample from channel 1 has been used as a virgin comparison to compare the other more irradiated samples to.

BEPO reactor shut down in 1968 resulting in 43 years of decay to date. Upon receiving this material into the university significant quantities of activity have already decayed leaving behind the long lived isotopes commonly found in all graphite. This helps with risk and COSHH assessment and carrying out many of the experiments on this material as a significant quantity of the material present is beta decay which is stopped by 6mm thick Perspex shielding which can be placed in front of most equipment protecting the user.

The availability of this material was also a high priority and its reduced isotopic inventory made it can ideal choice for use prior to receiving the significantly more active Magnox material at **UoM**.

3.1.4. JEN-1 Reactor

CIEMAT and **ENRESA** performed experiments on samples coming from the JEN-1 reactor. JEN-1 is a 3-Mw pond-type reactor. The fuel elements are MTR type; aluminium flat plates 19.8% U-235 enrichment with 10.5% burn up. Boral sheets are used as control elements. JEN-1 is placed inside the Ciemat research centre in Madrid. The reactor operated from 1959 and shutdown in October 1984, up today is dismantled. The reactor was cooled by heavy water and it had 8 tons of graphite thermal shields.

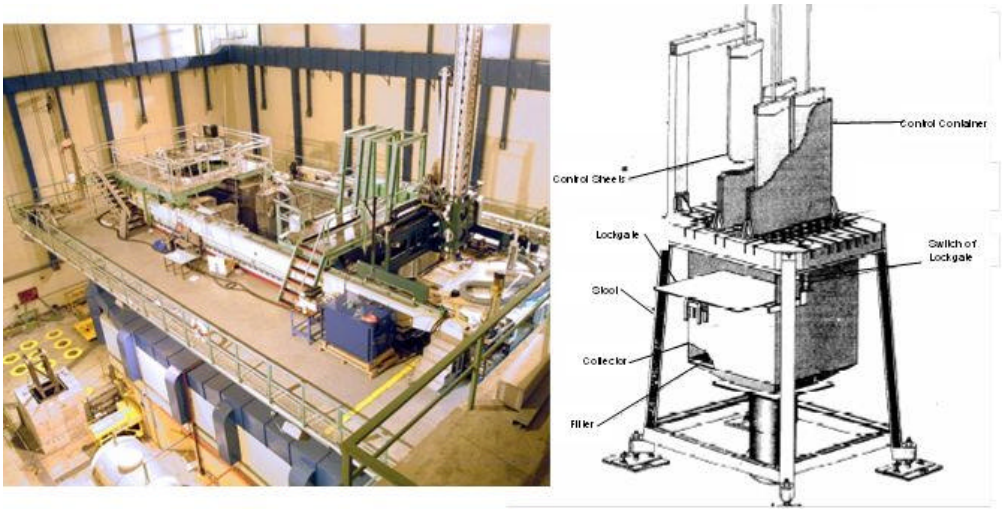

Figure 3.7 General view and scheme of JEN-1 (MTR) reactor

The samples taken from JEN-1 are used for inventory of the material and also for tuning-up the investigation procedures for radio-analytical determination and structural determination

methods. Aliquots are taken for 2 samples (virgin and irradiated graphite) received from dismantling department without information of their coordinates in the moderator.

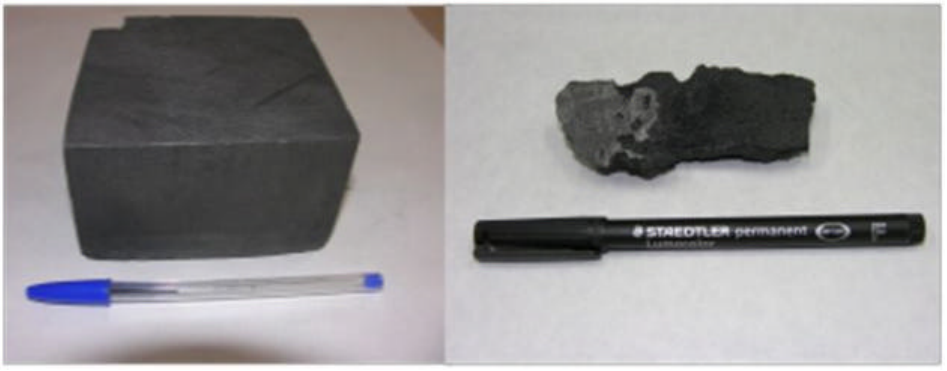

Figure 3.8. Samples of Virgin and irradiated graphite from JEN-1

3.1.5. BR1 Reactor

The BR1 reactor is the first research reactor built in Belgium. It reached its first criticality in May 1956 and it has been working till these days. BR1 is placed at the facilities of SCK.CEN located in Mol (Belgium).

BR1 is a natural uranium reactor moderated by graphite and cooled by means of air which flows inside the fuel channels. The moderator is graphite class A, its density is 1.72 g/cm^3 and includes 0,4 ppm of boron traces. Beyond a radius of 2.6 m graphite of class B is placed as a reflector. ts density is 1.62 g/cm^3 and contains 1.6 ppm boron traces and other impurities such as Fe, Al, K, Ca, Ni and V. The total mass of graphite in the BR1 reactor is about 500 tons.

Figure 3.9 View of the BR1 reactor

Figure 3.10 shows a cross section of the reactor, showing the different parts of the reactor.

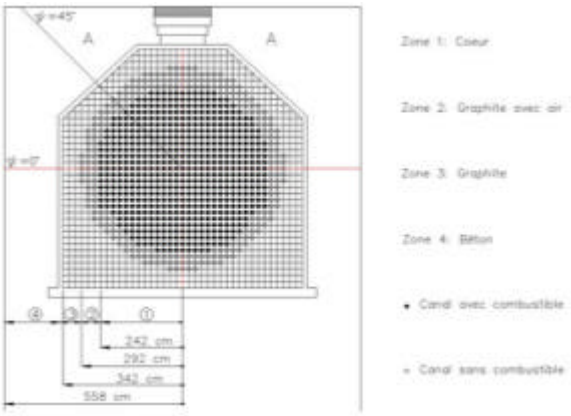

Figure 3.10: cross section of the BR1

3.2. **Samples of UNGG Reactor (Uranium Natural Graphite Gas)**

EDF (Électricité de France) operated in France six gas-cooled reactors, all shutdown now for at least fifteen years. These reactors are of so-called in French, “UNGG” reactor type (Uranium Naturel Graphite Gaz). They were graphite moderated, cooled by carbon dioxide and fuelled with natural metallic uranium.

The design of UNGG reactors is, in its general principle, very close to that of the British Magnox reactors, that was developed independently at the same period in United Kingdom. In the absence of uranium enrichment, graphite was used as a moderating material with a very high level of purity due to the necessity of the highest transparency to neutrons. Graphite has been also chosen as a mechanical support of the fuel cartridges (graphite sleeves) and as a biological shield in some reactors. A scheme of the main possible uses of graphite in UNGG reactors is shown in Figure 3.11. The irradiated graphite from the pile or from the biological shield still lies in the reactors. The graphite sleeves that are not already shipped to the final repository are stored in silos.

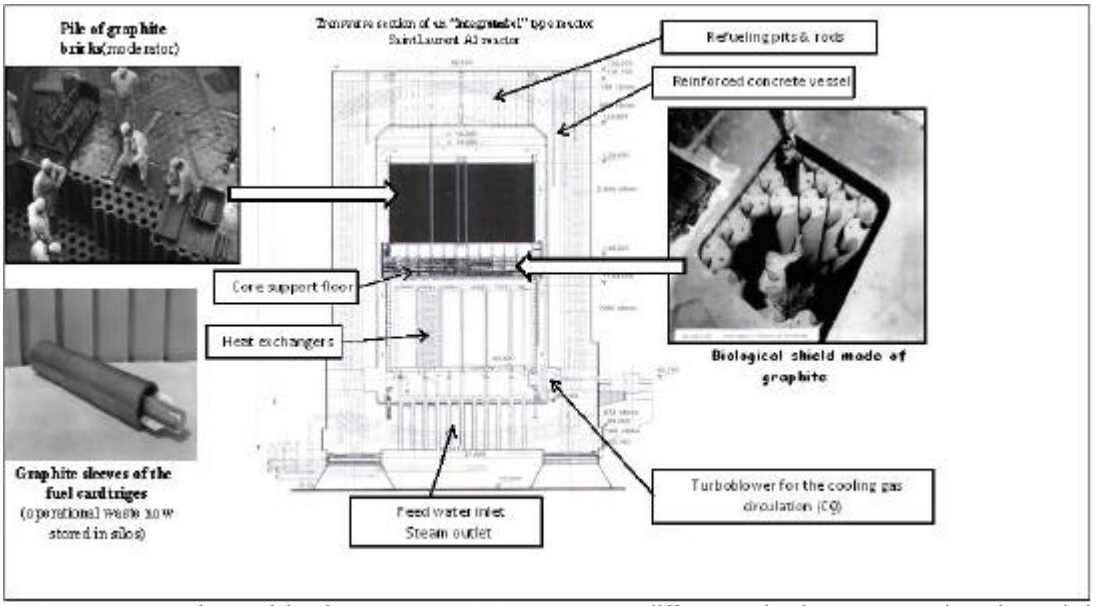

Figure 3.11 Uses of graphite in EDF UNGG reactors; different designs were developed, in this scheme an "integrated vessel" type reactor is presented (approximate external dimensions: 50 m height / 28.5 m diameter)

3.2.1. Saint-Laurent A2 reactor

The Saint-Laurent A2 (SLA2) reactor was commissioned in August 1971. The pressure vessel of this reactor is built of pre-stressed concrete which also acts as biological shielding. The coolant is CO_2 which circulates from top to bottom at a pressure of 29 bar in the space between the graphite and the Mg-Zr alloy fuel cladding. The operating temperature ranges between 240 °C (top) and 470 °C (bottom). The thermal power is 1,700 MW. The gross electrical power is 530 MWe and the net electrical power in nominal operation is 515 MWe. The thermal neutron flux (from 10^{-5} eV to 0.5 eV) in the central zone of the core and in the maximum flux plane is $3.12 \cdot 10^{13}$ n cm^{-2} s^{-1}.

It should also be noted that this reactor was shut down between March 1980 and September 1982 following an incident which led to the melting of two fuel elements at the bottom of the F5 M19 C14 channel (loss of cooling of the channel as a result of its obstruction by a metal plate, leading to overheating of the fuel elements).

The SLA2 reactor was finally shutdown on May 25, 1992. It was operated during 11 years full equivalent operating power. It has been de-fuelled and has been placed in a safe enclosure mode (ventilated by air with a moisture content limited to 50 %) until the final dismantling is performed.

EDF received the authorization to fully dismantle the SLA2 reactor by decree 2012-510 of May 18, 2010. All non-nuclear parts of SLA2 nuclear unit have been dismantled (dismantling IAEA level II achieved – figure 3.12). Up to now, final dismantling (level III) is planned by 2030-2040.

Figure 3.12 Saint-Laurent A1 (left) and Saint-Laurent A2 (right) UNGG reactors (current state 2012)

The graphite stack of the SLA2 reactor has the form of a cylinder with a vertical axis 15.73 m in diameter (13.43 m of moderator surrounded by a 1.15 m thick reflector) and 10.2 m high. The total mass of graphite is 2,440 tons including 1,580 tons of moderator and 860 tons of reflector (Figure 3.13).

The stack's network of graphite bricks is a hexagonal mesh with a pitch of 225.16 mm. The elementary graphite blocks are prismatic bars with a hexagonal base whose distance between two opposite faces is equivalent to the network mesh. The side-by-side juxtaposition of 4,429 bars forms a bed, the SLA2 stack consisting of 8 superimposed beds (bed No. 1 is that at the bottom of the stack). Thus, the bars are superimposed and the stack may also be regarded as the juxtaposition of 4,429 columns. The graphite stack comprises:

- The lateral reflector consisting only of 828 solid columns surrounding the core;
- The moderator (or core) consisting of 3,601 columns: 345 solid columns, 181 bored to 84 mm for the control rods and 3,075 columns bored to 140 mm (basically for the fuel elements).

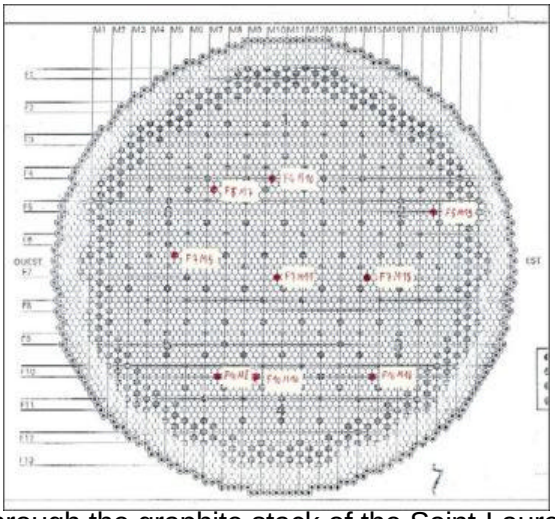

Figure 3.13: Section through the graphite stack of the Saint-Laurent A2 reactor - Juelich graphite cores were sampled from the F4M10 (hexagonal cell) C19 (fuel channel)

The graphite of SLA2 stack was manufactured by the Pechiney/SERS company between May 1966 and November 1967. This graphite was made from Lima coke, underwent one impregnation and was purified using MgF_2. During its manufacture it was tested particularly with regard to effective cross-sections, density measurements and other properties described in the above table.

Ashes (ppm)	B (ppm)	Li (ppm)	Co (ppm)	Cl (ppm)	Thermal neutron absorption cross section (mbarn)	density
98	0.11	0.07	0.05	-	3.76	1.68

Concerning radionuclide inventory of the irradiated SLA2 graphite from the pile, it has been determined as described in Carbowaste report T-3.4.3 and corresponds to the following values for the main radionuclide of interest:

^{3}H	^{10}Be	^{14}C	^{36}Cl	^{41}Ca	^{60}Co	^{63}Ni	^{137}Cs
$8.9\ 10^{4}$	19.1	$7.7\ 10^{4}$	37.9	83.7	$4.0\ 10^{3}$	$3.5\ 10^{4}$	47.0

These values are in Bq/g and related to January 2012. They take no account of the influence of leaching phenomena of the underwater dismantling process. They are over-estimated mean values as described in Carbowaste report T-3.4.3.

The radionuclide inventory for Cesium-137, a trace element for the fission reactions, shows that the decontamination operations adopted following the fuel element melting have been effective. In fact, this incident has no detectable effects in terms of contamination of the graphite. The levels of Cesium-137 are equivalent in all the EDF reactors whether fusion of fuel elements has occurred or not. The presence of Cesium-137 in very low quantities and the heavy nuclei produced are explained by the fission of traces of uranium present in the original graphite. These traces were identified in the analyses performed on the graphite at the time of their manufacture and now evidenced by the identification calculation-measurement method used for the radionuclide inventory assessment.

EDF delegated the CEA Cadarache LARC for the sampling, conditioning and the transport of these samples.
CEA received samples of 10 core samples from St Laurent A2 were chosen to calculate the activity of the radionuclides. The following Table 3.2 shows the position of the samples in the reactor. Some part of the sample was also used by ENS.

Table 3.2 Samples reference from St Laurent A2

Ref.	Channel	Height [mm]	Mass [g]
SLA2-103	F4M10C19	1.680	18.5446
SLA2-105	F4M10C19	2.460	21.6944
SLA2-110	F4M10C19	5.120	21.2202
SLA2-116	F4M10C19	7.880	21.5788
SLA2-119	F4M10C19	9.260	22.0051
SLA2-63	F5M7C17	1.680	23.761
SLA2-65	F5M7C17	2.460	18.7483
SLA2-71	F5M7C17	5.510	23.1647
SLA2-76	F5M7C17	7.880	23.6531
SLA2-79	F5M7C17	9.260	21.9857

The dimensions of these core samples are around 37-60 mm long by a diameter of 19 mm, or around 20 g (Figure 3.14).

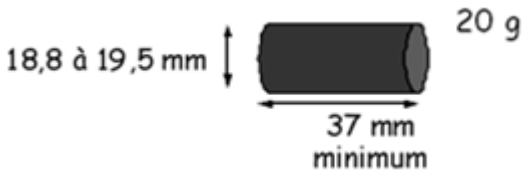

Figure 3.14: Dimension of SLA2 core samples

5 Samples were sent to **Fz Juelich** coming from F4M10 cell C19 channel. They were chosen as being distributed over the height of the fuel channel in order to correspond to the operating temperature range in SLA2 reactor, as shown in the following table:

Samples from St Laurent A2 were received at **CIEMAT** from CEA Cadarache for Characterisation program inside Carbowaste project. The description and the location of the samples are already described and in table 3.3 is collected the main characteristics of these samples. A picture of the sample (aliquots already took) is showing in figure 3.15

Table 2.4. Samples of Graphite pile from St Laurent A2

Reference	Origin	Dose rate (µSv/h)	Mass (g)	Remarks
SLA2-63	Cadarache	158	23,7610	Cylinders ~3 cm ?, ~ 10 cm height
SLA2-65		60	18,7483	
SLA2-71		90	23,1647	
SLA2-76		100	23,6531	
SLA2-79		28	21,9857	

Figure 3.15. Sample of SLA-2 graphite

3.2.2. Reactor of Vandellós-1 NPP

NPP of Vandellós 1 is a Uranium Natural Graphite Gas reactor (UNGG), it was graphite moderated, cooled by carbon dioxide, and fuelled with natural uranium metal:It was a power of 460 MWe, being 56000 GWh from 05/06/72 to 10/19/89. It Was operated from 1971 and Shutdown in October 1989. In 2008 HIFRENSA transfer to ENRESA the plant for dismantling and decommissioning operations and the decommissioning at level 2 including the safe enclosure period of 25 years was performed from 1998 to 2003. An Scheme of this reactor is in figure 3.16.

The graphite material from this reactor was manufactured by Pechiney SA in France. The manufactured method was not well defined; Pechiney graphite was prepared by baking a paste made of oil coke and pitch, graphitized by electrical heating.

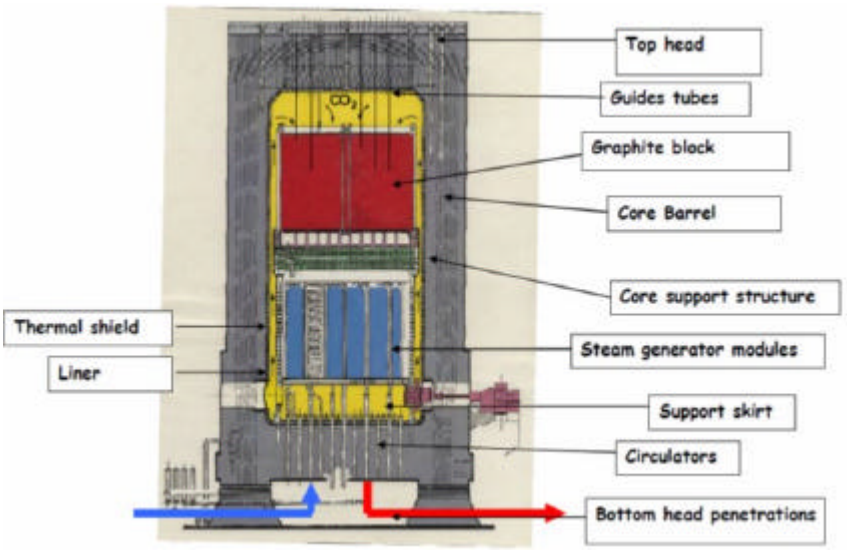

Figure 3.16 Vandellos 1 vessel

There are 186000 used graphite sleeves (1100 tons). The radioactive waste management, during the process of the plant, consisted of the direct dropping of the wastes inside concrete silos. The carbonaceous radioactive waste contained in the silos was graphite sleeves and graphite cylinders (rondins). Additionally two spent fuel elements, with its graphite sleeves, were accidentally dropped inside silo 1. These wastes were grinned, shorted and packaging.

Figure 3.17 Vandellos 1 sleeves virgin and i-graphite

Powder samples (22 listed in table 3.5) took from the filter system used in the grinned procedure were sending to **CIEMAT**. More than 200 aliquots were taken in order to determine the main radiological characteristics of the samples.

Table 3.5. Samples of Graphite Sleeves from Vandellos 1

ID	Container	Origin	ID	Container	Origin
G-01	V-038	Silo 2	G-107	M-010	Silo 1
G-07	S-009	Silo 3	G-114	S-077	Silo 1
G-33	S-033	Silo 3	G-125	S-064	Silo 2
G-43	V-047	Silo 3	G-142	S-037	Silo1/2
G-50	V-079	Silo 2	G-153	M-039	Silo 1
G-81	S-058	Silo1/2	G-175	S-072	Silo1/2
G-82	S-061	Silo 1	G-176	M-006	Silo 1
G-92	V-067	Silo1/2	G-187	V-044	Silo 2
G-93	V-059	Silo1/2	G-203	S-075	Silo 3
G-94	V-055	Silo1/2	G-213	S-004	Silo 2
G-105	S-052	Silo 2	G-219	S-016	Silo1/2

Vandellós 1 moderator graphite pile is a vertical cylinder, 10.2 m height and 16.6 m diameter with a total weight of about 2680 tons.

Previously to the end of the dismantling activities until Level 2, a drilling procedure was used for obtaining 58 cylindrical samples (50 mm height and 20 mm diameter) from graphite pile channels. Approximately all the samples were taken from channels located in the same semi-plane (between the central axis and the west part of the moderator graphite pile).(figure 3.18)

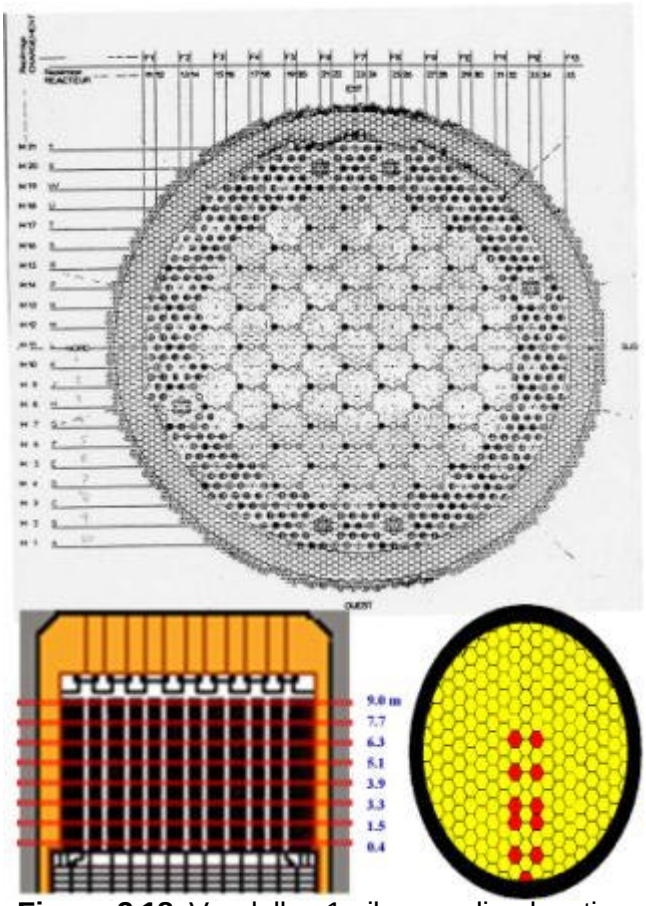

Figure 3.18. Vandellos 1 pile sampling location

Table 3.6 Samples of Graphite pile from Vandellos 1

Height (m)	Approximate distance from the graphite pile axis (D = moderator graphite pile diameter)										
	0.0 D		0.1 D		0.2 D		0.3 D		0.4 D		0.5 D
9.0	G-3	G-6	G-14	G-22	G-30	G-38	G-41	G-44	G-47	G-58	G-50
7.7			G-13	G-21	G-29	G-37					G-57
6.3			G-12	G-20	G-28	G-36					G-56
5.1			G-11	G-19	G-27	G-35					G-55
3.9	G-2	G-5	G-10	G-18	G-26	G-34	G-40	G-43	G-46	G-54	G-49
3.3			G-9	G-17	G-25	G-33					G-53
1.5	G-1	G-4	G-8	G-16	G-24	G-32	G-39	G-42	G-45	G-52	G-48
0.4			G-7	G-15	G-23	G-31					G-51

3.2.3. G2 UNGG reactor

CEA took five core samples distributed over the lower half of the reactor were chosen to carry out studies on micro structural characterisation, behaviour of ^{36}Cl during leaching, and to determine the activities of the radionuclides: four in the moderator (No. 27, 32, 36 and 42), and one in the reflector (No. 46). The five core samples are equidistant from each other, as shown in Figure 3.19.

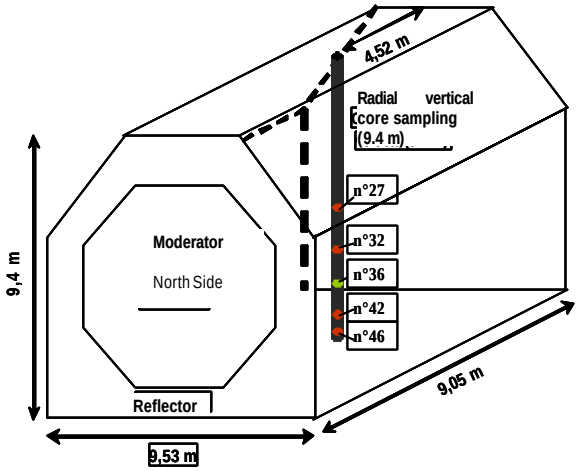

Figure 3.19 Representation of the G2 reactor with the vertical core sampling and the core samples selected

The dimensions of these core samples are around 130 mm long by a diameter of 63 mm, or around 650 g (Figure 3.20).

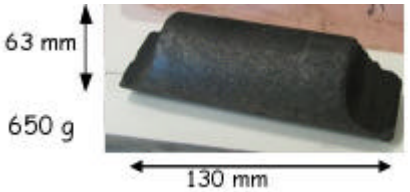

Figure 3.20 Dimension of G2 core samples

Table 3.7 contains the sampling levels and the functioning temperature of the reactor at the level of the core samples selected.

Table 3.7 Selected G2 samples (temperatures correspond to an estimated average for graphite)

Sample No.	Position	Assumed initial coke	Sampling level [m]	Temperature [°C]
G2-27	Moderator	Special A coke	13.60-13.80	327
G2-32	Moderator	Special A coke	14.60-14.80	320
G2-36	Moderator	Special A coke	15.40-15.60	314
G2-42	Moderator	Special A coke	16.60-16.80	309
G2-46	Reflector	Lockport Coke	17.40-17.60	284.7

For the purposes of the leaching tests, and in order to have several sampling tubes, the core samples were split into several samples. Table 3.8 shows the different samples analysed.

Table 3.8 G2 samples analysed

G2-27 G2-32 G2-42 G2-46	Powders arising from splitting operations representing an average core sample	3 samples taken from the ends and the middle of the core sample
G2-36		Removal of 11 samples along the core sample

3.3. Samples of Magnox Reactors

3.3.1. Oldbury-2 Magnox Reactor

Oldbury Power Station is a 434MW, twin nuclear reactor station. It is the world's oldest operating nuclear power reactor and the UK's first nuclear power station to use pre-stressed concrete pressure vessels instead of steel.

The plant entered service in 1967. Magnox Electric is the licensee and is responsible for the day-to-day management and operations of the power station. The Nuclear Decommissioning Authority (NDA) is the owner.

The plant is built on a 175 acres site located on the south bank of the River Severn in South Gloucestershire. The nearest village / town, Oldbury-on-Severn, Bristol, is located 24km away from the power station. The area around the plant site has been specified as a Site of Special Scientific Interest (SSSI) and a Special Protection Area.
Decommissioning the UK's first nuclear power station

The plant was originally scheduled to be decommissioned by 30 December 2008. The deadline, however, was extended twice. Reactor Two got an extension for two and a half years, while Reactor One got four years of extension.

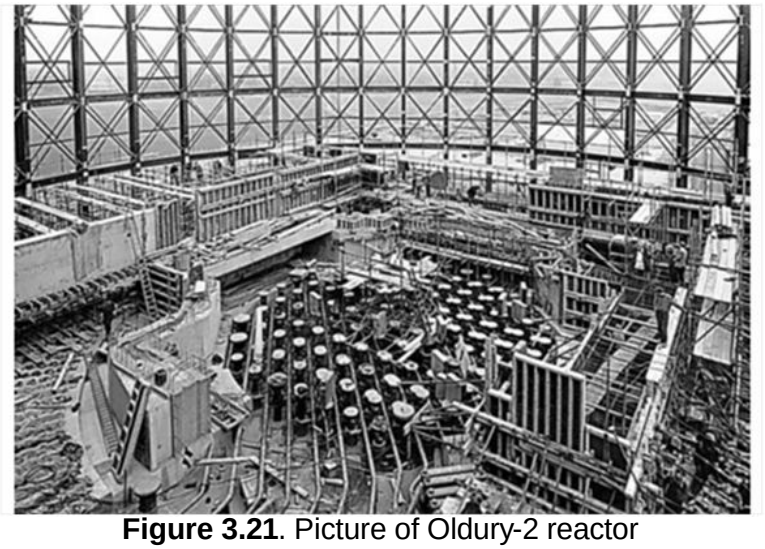

Figure 3.21. Picture of Oldury-2 reactor

Reactor Two was finally decommissioned on 30 June 2011, after 43 years of operations. It produced more than 65TWh in its lifetime. Reactor One will continue to be operational until December 2012.

The Oldbury Nuclear Power Station decommissioning project is estimated to cost £954m. It is expected to take approximately 90 years to attain final site clearance. Defueling of the reactors will take two years. Care and maintenance preparations will start in 2014 and end in 2023.

All the spent fuel will be transported to Sellafield for reprocessing through the Cooling Pond. Reprocessing will remove more than 99% of the entire radioactive content.
Oldbury power plant history

The consent for the construction of Oldbury Power Station was given by the Minister for Power in 1960. Construction commenced one year later in 1961. The first electricity from Reactor One was produced in November 1967.

Reactor Two began production in April 1968. The station was officially opened in June 1969 by the then Minister of Technology, Anthony Wedgwood-Benn.

Buildings of the main power station were constructed by a consortium, The Nuclear Power Group (TNPG). Most of the remaining buildings were constructed by Sir Lindsay Parkinson & Co. (acquired by Fairclough Construction Group in 1974). TNPG also supplied the reactors. Associated Electrical Industries and C.A. Parsons and Company supplied the turbines. Alfred McAlpine, acquired by Carillion in 2008, was the main civil engineering contractor.
Oldbury nuclear power plant details and specifications

Oldbury Power Station was originally designed for 600MW of electrical output. The operating gas temperature had to be reduced soon after the start of operations due to steel corrosion problems caused by the hot CO2 coolant within the reactor.

The net output came down to 400MW, as a result. It was brought up to 416MW in 1975 after making alterations in the main steam turbines. The net output increased to 434MW by making improvements to thermodynamic efficiency in 1982. The output was increased further to 452MW after installation of new HP turbine modules.

The plant uses two CO2 cooled, graphite-moderated Magnox reactors which were originally fuelled with natural uranium. Enriched uranium was introduced in August 1998. The plant has two turbo generators.

The number of fuel channels per reactor is 3,308 and there are eight fuel elements per channel. Natural and enriched uranium are the fuels. Cooling water for the reactor is supplied from the River Severn. The plant is currently served by 480 employees.

The description of the main characteristics of the samples delivered is in the Table 3.9 and in Figure 3.22

Table 3.9 Samples of Magnox Graphite

Reference	Origin	Dose rate (µSv/h)	Mass (g)	Remarks
634-BLOCK-2-4-2	OLDBURY-2	-	10	Circular Sector ~1,5 cm height
634-BLOCK-2-4-3		23	9	Circular Sector ~1,5 cm height
676-BLOCK-4-1		750	63	Disc ~10 cm ? ; ~ 1,7 cm height

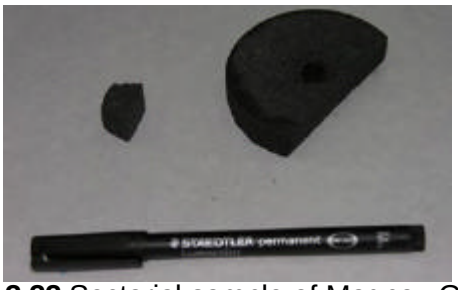

Figure 3.22 Sectorial sample of Magnox Graphite

3.3.2. Wylfa Magnox Reactor

Wylfa Magnox reactors were commission in 1971 and consist of two reactors with a combined capacity of 980 MW. Wylfa reactors were the largest and last of the Magnox reactors to be built in the UK see Table 2.10 in the introduction section. Each Wylfa core has 6,156 vertical fuel channels and contains over 49,248 natural uranium Magnox-clad fuel elements; these large graphite moderated reactors typically supply 23 GW h of electricity per day. The primary coolant used in carbon dioxide.

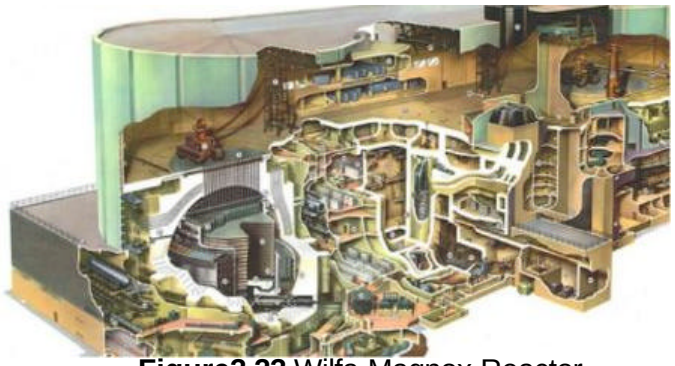

Figure3.23 Wilfa Magnox Reactor

The samples issues to UoM for this research where trepanned from the fuel channel wall of reactor 1 channels 1413/02 and 1319/12 in April 2007. Figure 24 shows the location of these two sampling points. There are twelve bricks per column in reactor 1 and they are numbered from the bottom of the core. Sample 1413/02 9U comes from an upper position in brick nine and sample 1319/12 8U comes from an upper position in brick eight. Table 3.10 details the irradiation history of these two samples. Sample 1413/02 was used for leaching experiments and sample 1319/12 was used for Raman analysis.

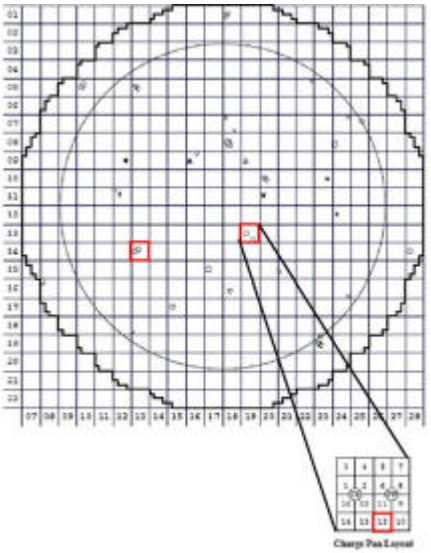

Figure 3.24 Highlighted in red are the samples taken in 2007 from the Wylfa reactor 1 used in this research

Table 3.10 Irradiation conditions of Wylfa samples used in this study

Irradiation conditions	1413/02 9U	1319/12 8U
Irradiation temperature (°C)	352	342
Adjacent fuel dose (MWD/t)	34973	37319
DIDO equivalent dose at channel wall (10^{20} n/cm^2)	34.21	37.11

Along with the irradiation history of the two trepanned Wylfa Magnox samples provided to the University of Manchester the isotope inventory detailed in Table 2.11 and shown in Figure 3.25 was also provided, graphically the vast difference in quantities between each isotope present is observed.

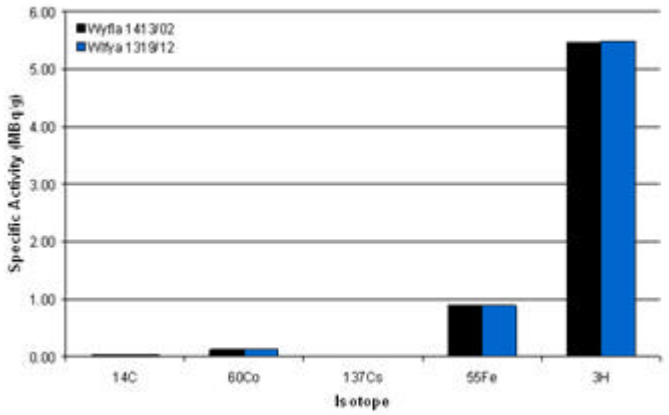

Figure 3.25 Graph showing the isotopic inventory present in the trepanned samples taken from the Wylfa reactor core in April 2007

3.3.3. Latina NPP Reactor

The Latina Reactor is Graphite moderated with fuel constituted by ^{235}U and coated with Magnesium Oxide (MagnOx). The maximum burn-up was 5000 MWD/t. The operating period

of the plant was 1963-1986, the starting nominal electrical power was 200 MW, but the value was reduced to 160 MW since 1969, with a cumulative output of about 26 TWh.
The maximum operating temperature of the fuel was 450 °C. The core consists of a 24-sided vertical prism made of graphite blocks in layers, assembled to form vertical cylindrical channels for fuel, control rods and neutron absorbers. The coolant was CO_2 in closed circuit. The graphite structure is divided into two parts: the central one (moderator or active core that hosts fuel and interstitial channels) and the lateral one (reflector). The floor bricks act both as reflector and moderator.

The Nuclear graphite, specifically manufactured for use as a moderator or reflector within nuclear cores, consists of graphitic carbon of very high chemical purity to avoid absorption of low-energy neutrons and the production of undesirable radioactive species.

Besides the absence of neutron-absorbing impurities, reactor graphites are also characterized by a high degree of graphitization and no preferred bulk orientation. Such properties increase the dimensional stability of the graphite at high temperatures and in presence of high neutron flux.

The production of nuclear graphite consists in two steps: a carbonization or pyrolysis step and a graphitization step. During this graphitization process the graphite is purified with chlorine and hydrofluoric acid, and then pressed.

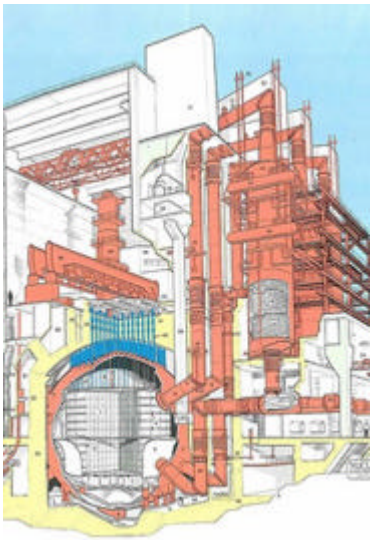

Figure 3.26 Latina NPP reactor

Nuclear graphite for the Magnox reactors were manufactured from petroleum coke mixed with coal-based binder pitch heated and extruded into bricks, and then baked at 1000 °C for several days. To reduce porosity and increase density the bricks are impregnated with coal tar at high temperature and pressure before a final bake at 2800 °C. Individual bricks were then machined into the final required shapes.

As part of an agreement between ENEA and So.G.I.N., ENEA received 15 cylindrical samples from Latina NPP. They were taken from the drilling of the core in two different radial positions (channel 7 and 8): from each sample were removed both the surface layer exposed to the fuel channel and the innermost layer; the approximate mean weight is 5 g.

The removal of the layer exposed to the channel ensures that the activity present in the sample is representative only of the contribution due to neutron activation.

For each sample a complete radiological characterization is scheduled by means of destructive and non-destructive measurements before and after the treatment. In table 3.11 the masses of the samples, after the outer layer removal, are reported; the mean diameter is 1.7 cm and the mean height is about 1.0 cm (see Figure 3.27).

Table 3.11 Irradiated-Graphite Samples weights in grams (g)

08F08							
A1/C3	A1/C4	A1/I2	A1/I3	A1/S1	A1/S2	A1/S3	
3.5476	4.4188	4.1437	4.2189	2.0567	3.9708	3.5290	
07S07							
G2/C3	G2/C4	G2/I1	G2/I2	G2/I3	G2/S1	G2/S2	G2/S3
3.3065	3.7149	3.3451	3.6853	3.5436	4.1867	4.3726	3.9471

As mentioned, 7 samples came from the Channel 8 (08F08), while the other 8 samples from Channel 7 (07S07); For each of these 2 groups, the samples came from different level origins named: *S,* Superior, *C*, Central and *I*, Inferior.

Figure 3.27 One of the i-Graphite samples from Latina NPP

3.4. **Samples of RBMK Reactors**

The graphite samples from a fuel channel of the RBMK-1500 reactor of Ignalina NPP have been taken to represent top, central and bottom parts of a graphite sleeve. The graphite of the particular fuel channel of 10,700 MWd burn-up, which corresponds to 11.6 years of irradiation at the average power, has been selected for sampling.

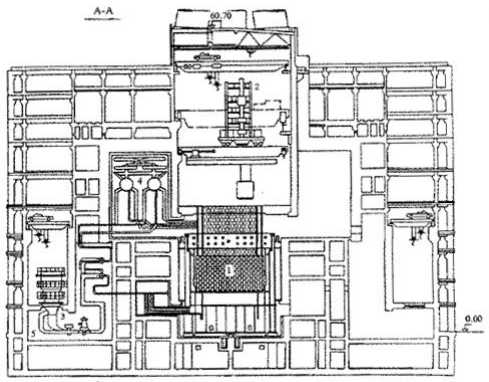

Figure 3.28 Ignalina Plant Reactor section

Two irradiated samples Gr.13 and Gr.14 have been taken from the central part (at the active height of ~375 cm) of the graphite sleeve of the fuel channel and two other samples Gr.18 and Gr.16 from the top (at the active height of 28 cm) and the bottom (the plug fragment) of the fuel channel graphite (see Figure 3.29).

The biggest sample Gr.16 (146 g) taken from the bottom part of the fuel assembly is presented in Figure 3.30. The other samples weighted 2 g each. The original graphite sample Gr.16 had a shape of a cone. The samples for gamma spectrometric analysis have been scraped from the inner and outer surfaces of the graphite sample Gr.16.

To determine the level of homogeneity of activity distribution of gamma-emitters in the graphite the scraped samples from inner and outer surfaces of sample Gr.16 have been divided in three sub-samples from each surface.

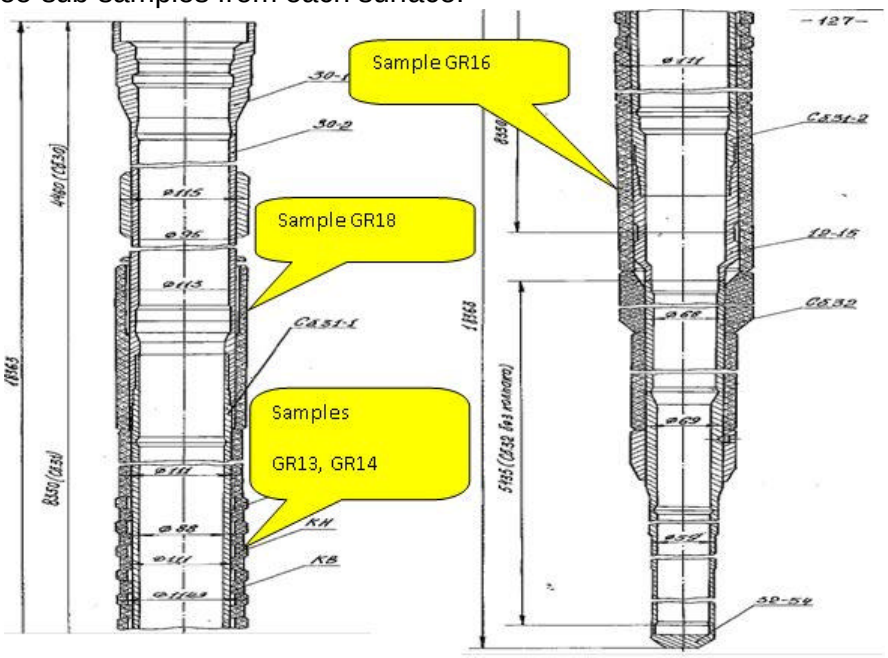

Figure 3.29 Top (on the left side) and bottom parts of the fuel channel

Figure 3.30 The biggest sample (Gr.16) of irradiated graphite taken from the bottom part of the fuel assembly

3.5. Samples from High-Temperature Reactors (HTR)

At the research centre of Jülich a new type of high temperature reactors was developed. The operator was the Experimental Reactor Consortium (Arbeitsgemeinschaft Versuchsreaktor, AVR). The core of this reactor consisted of a pebble bed in which the fuel was embedded in graphite pebbles with a diameter of 60 mm. In these pebbles the fuel was dispersed as coated particles.

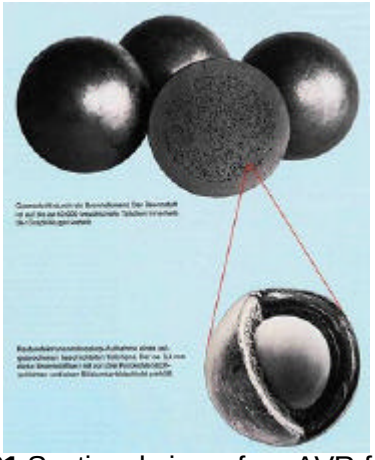

Figure 3.31 Sectional view of an AVR fuel pebble

The fuel zone of one pebble had a diameter of 50 mm and was enclosed by a 5 mm thick fuel-free graphite shell (see Figure 3.31). Flakings of this outer shell were used for the WP3 and 4 experiments.

The AVR reactor was a graphite-moderated gas-cooled high temperature reactor. Also the reflector was graphite (see Figure 3.32). In preparation of the dismantling of the reactor samples of the reflector have been taken.

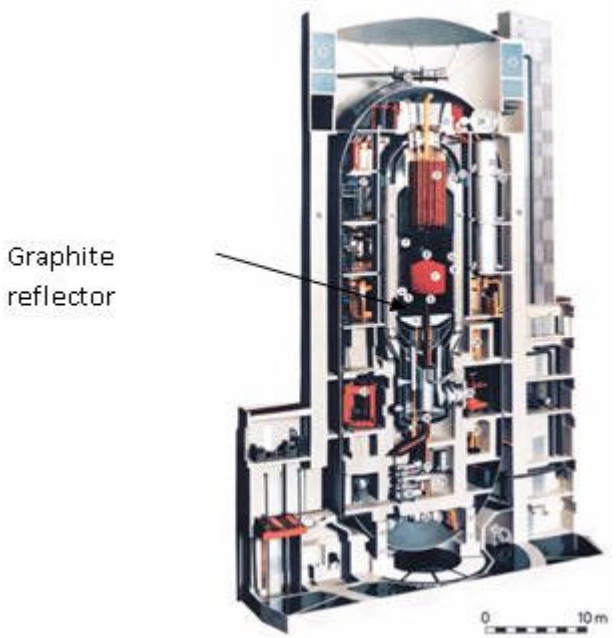

Figure 3.32: Schematic view of the AVR reactor

4. SAMPLE MANAGEMENT PROCEDURES

Establish and standard of waste sample management procedure is a difficult labour due to the regulatory conditions of each state and specific safety, health physics, security and particular capabilities of the origin and reception facilities.

In the case of irradiated carbonaceous waste is not different, but taking into account, the successful management achieve in CARBOWASTE project thanks to the hard work of the actors involved a general guidance can be done and illustrate it with some data of specific examples within the project.

The main steps of the Sample management can be summarized:

- Management strategy ←→Characterisation/application strategy
- Availability of the sample source
- Identification of the samples as a function of:
  - Coordinates in reactor and neutron history
  - Mass needed in function of the final studies to be use
  - Representativeness → Final user of data
- Sampling technology: Radiation protection protocols
- Storage and QA/treazability of samples
- Main Characteristics determination: Aggregation state, size and shape, preliminary data of activity and nuclides content, dose rate, etc.
- Receptor duties: Disclosure agreement, Reception authorization, licenses, etc.
- Transport concept: according to the ADR in Europe.
  - Transport media and authorised companies
  - Packaging
  - Transport documentation Import/Export protocols

  - Delivery : Date and documentation ➔ Certificates of absence of radioactivity of containers and transport media
- QA at the reception:
  - DR, SC and other physical and RP data need
  - Storage
  - Characterisation prior to assays: Reliability of the results
  - Sample preparation for testing

Examples of the main events and features of the management procedures applied on the samples widest distributed among the beneficiaries are done in the following subsections. These cover, in particular, samples from UNGG reactor core SLA-2 (EDF-CEA), samples from Magnox reactor core OLDBURY-2 (NDA), from thermal column of TRIGA reactor at Pitesti (INR) and graphite from the sleeves of UNGG Vandellós-1 reactor (ENRESA-CIEMAT).

Sampling procedure is operationally different but all the sampling methods response to a sampling plan within a management strategy which covers the complete map of variability of graphite in concern and taking into account the sampling technology available and the applicable safety procedures.

4.1. MTR Triga Pitesti Samples

Virgin and irradiated graphite samples from the thermal column from TRIGA reactor were sent by INR at request of:

- CIEMAT – Spain
- University of Mancheste – UK
- SCK.CEN - Belgium

NECSA from South Africa also requested MTR samples but due to the special transport conditions and formal procedures, NECSA canceled its demand.

Blocks of virgin graphite with the same origin as the one used in the termal column of TRIGA reactor were sent on 22nd February 2010 by DHL Express in the requested quantities as it follows:

- CIEMAT: 200g
- UoM: 200g
- SCK CEN: 50g.

For physical protection, each sample was encased in sealed plastic box placed in a cardboard box filled with polystyrene.

The cost of virgin graphite transfer was covered by INR (281.14 RON/transfer)

Irradiated graphite samples sent to CIEMAT, UOM and SCK.CEN arise from the same piece of graphite extracted from the thermal column. The exact position and irradiation history were unknown.

Due to the radionuclide content and activity, elaboration of Material Safety Data Sheet for each sample was needed. According to international norms, samples were included in the excepted materials category, and no special packaging conditions were requested for this

transfer. Consequently the same packaging as for the virgin graphite was used. Quantities transferred are:

- CIEMAT: 100g
- UoM: 100g
- SCK CEN: 100g.

All samples were in solid state to reduce the risks associated to the graphite powder state. Samples characteristics are grouped in the Material Safety Data Sheet (annex 1).
The transfer was provided by MASTER MIND SRL

for the following costs, covered by each beneficiary as it follows

- Bucharest (OTP) - Manchester: 281.50 EURO
- Bucharest (OTP) – Madrid: 314.22 EURO
- Bucharest (OTP) - Mol: 324.22 EURO;

4.2. UNGG Saint Laurent–A2 Samples

The SLA2 reactor stack was subjected to core drilling in 2005 during which 180 cores were extracted. Remote controlled tools introduced into the fuel channels of the stack were used. This technique is the same to the technique implemented for monitoring the wear of the graphite during operation. The remote-control tool (Figure 4.1) was introduced from accesses through the re-fuelling pits at the top of the reactor into the channels, and performed coring on either side of the graphite bricks (Figure 4.2) in the direction perpendicular to the channels with a diameter of about 20 mm.

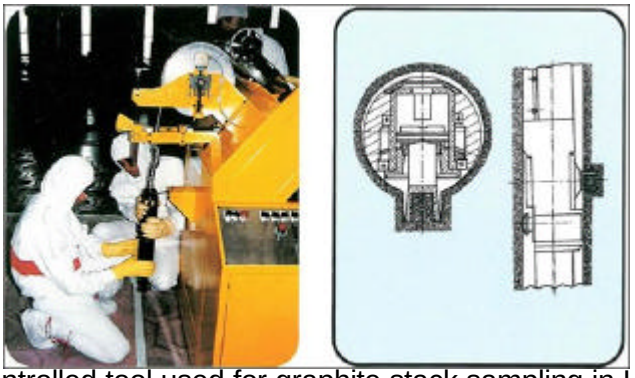

Figure 4.1 Remote controlled tool used for graphite stack sampling in UNGG reactors

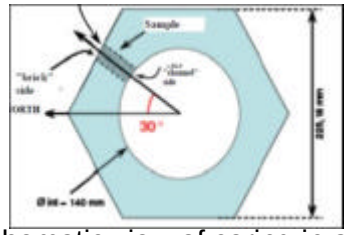

Figure 4.2 Schematic view of coring in a graphite brick

Samples of the reactor core of SLA-2 were distributed by EDF through CEA according to Table 4.1. A preliminary agreement was send prior to transport from origin (appendix 2).

Table 4.1 UNGG SLA-2 Samples characteristics and distribution

MASS (g)						
Adress	**Height**	**µSv/h**	**Subsample 1**	**Initial**	**Subsample 1**	**Total sub1**
F5M7C17	1680	239	CIEMAT	24	24	111,5
F5M7C17	2460	78	CIEMAT	18	18	
F5M7C17	5510	131	CIEMAT	23	23	
F5M7C17	7880	145	CIEMAT	25	24,5	
F5M7C17	9260	47	CIEMAT	22	22	
F10M16C20	1680	241	CEA+ENS	22	22	137
F10M16C20	6500	107	CEA+ENS	20	20	
F10M16C20	9260	45	CEA+ENS	25	25	
F7M15C19	1080	16	CEA+ENS	25	25	
F7M15C19	4480	15	CEA+ENS	22	22	
F7M15C19	9260	96	CEA+ENS	23	23	
F4M10C19	1680	185	FZJ	18	18	104,5
F4M10C19	2460	193	FZJ	22	22	
F4M10C19	5120	462	FZJ	21	21	
F4M10C19	7880	107	FZJ	21	21	
F4M10C19	9260	9	FZJ	23	22,5	
F10M16C20	2070	210	CEA+ENS	23	22,5	132
F10M16C20	7280	60	CEA+ENS	19	18,5	
F10M16C20	8660	35	CEA+ENS	23	22,5	
F7M15C19	2070	16	CEA+ENS	23	22,5	
F7M15C19	7280	286	CEA+ENS	24	23,5	
F7M15C19	8660	81	CEA+ENS	23	22,5	
F10M10C20	3840	25	SUBATECH			
F4M10C19	1080	17	SUBATECH			
F5M19C20	1080	8	SUBATECH			
F5M19C20	4480	30	SUBATECH			

All relevant operational data and the coordinates of the distributed sample were also supplied by the owner in order to correlate the experiments with the conditions during operation.

4.3. **Magnox Oldbury-2 Samples**

The graphite samples were cut from a cylindrical spacer piece on a sample pot assembly (carrier) (see figure,4.3 component to right of section H-H) . The assembly sits in an interstitial channel. The carrier is held together by a titanium rod (which will enhance gamma but not affect fast neutron dose).

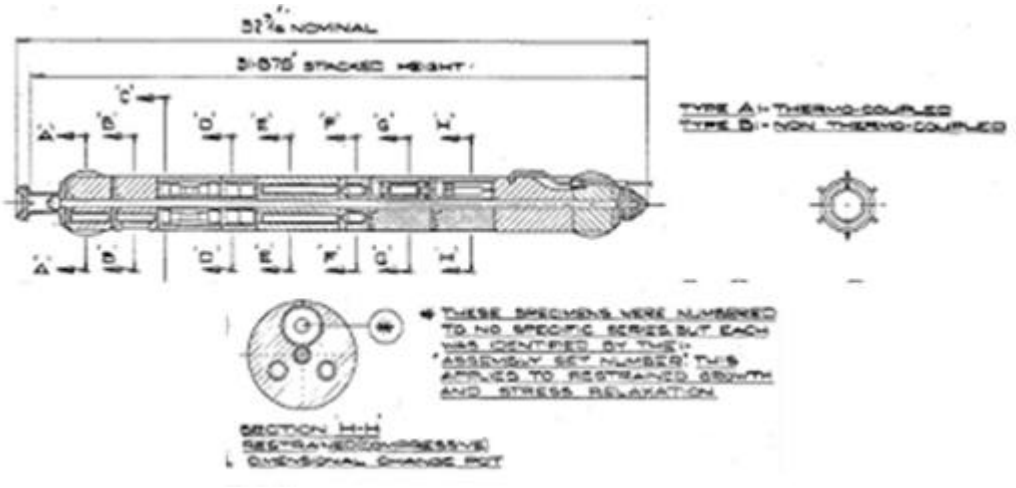

Figure 4.3 Sampler assembly

Pot 634 was located at position 4 in Channel S77 (BNL Channel 3) of Oldbury Reactor 2. This is the flattened region of the core and position 4 is just below mid-height. The Type A carrier had been in place continuously from start of life up to June 2005, Mean Core Irradiation 30658 MWd/t.

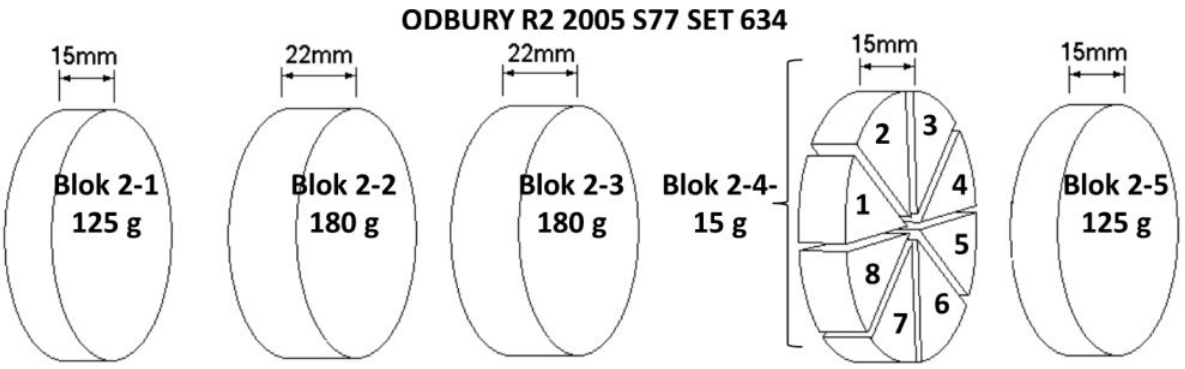

Figure 4.4 Block 634 specimen QA documentation

The rest of the sample distributed documentation are collected in figure 4.5

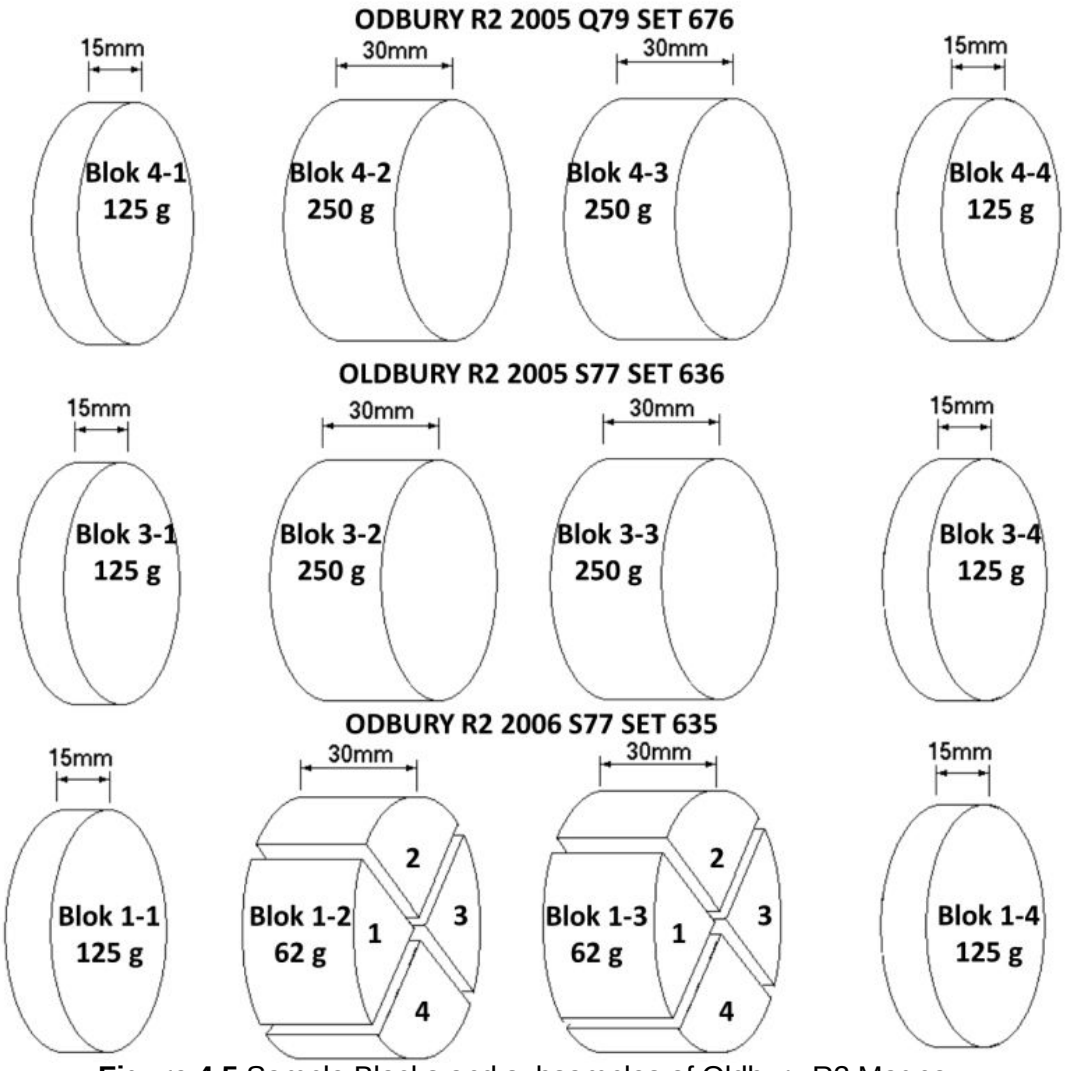

Figure 4.5 Sample Blocks and subsamples of Oldbury R2 Magnox

NNL was never contractually instructed by Magnox to carry out dosimetry calculations specifically for carrier removals at this outage. Instead, dosimetry was based upon and scaled from Oldbury Reactor 1 2004 data. On this basis:

- Irradiation temp: 543 K
- Adjacent fuel dose: 52827 MWd/t

In January 2009 University of Manchester request Magnox samples to study in Carbowaste project and MAGNOX authorise the availability of the requested irradiated graphite material.

In March 2009 samples were prepare in cave and prepare for export but in April Windscale lab suffered a major equipment failure that prevented samples for being exported.

In March 2010 was restarted the export from Windscale lab and the samples were moved to a low activity storage ready for shipment. In May of this year in order to share the management costs of samples, CIEMAT in agreement with the consortium and on charge of the transport costs of RRT samples ask to University of Manchester the delivery of samples to CIEMAT

facility in order to redistribute among the other partners involved. UoM ordered to supply the samples to CIEMAT.

At the same time problems on fingerprint of Magnox graphite showed-up which prevented the transport of the samples due to was impossible to complete the required documentation.

In October 2010 the problems with fingerprint were solved and the set of samples to UoM, that do not need export authorisation, was delivered. Concerns on export documentation and control rose.

In January 2011 the export control issues was and the shipment was schedule for February. In February 2011, new Sellafield Site process results in chosen shipment route being blocked and alternative route is identify. In March 2011 the new alternative route was not viable due to lack of "designated consignor" status for air shipment.

After exploring other routes and challenging basis for closure of original route, original route to CIEMAT is reopened by diverse interventions of British partners in the project. In May 2011 samples dispatched and arrive at CIEMAT.

The data of the samples sent to CIEMAT and UoM and the redistribution of the samples to FzJ, CEA and ENEA Labs are in the table 4.2.

Table 4.2 Samples distributed from Oldbury-2 Magnox Reactor

	MANAGEMENT AT UK			MANAGEMENT AT SP		
Item Identifier	**Description**	**mSv/hr ?**	**Dispatch to**	**(µSv/h) ?**	**mass (g)**	**Dispatch to**
634-BLOCK-2-1	125g disk, 17mm thick	1	**UOM**			
634-BLOCK-2-2	180g disk, 27mm thick	1	**UOM**			
634-BLOCK-2-3	180g disk, 27mm thick	0,7	**UOM**			
634-BLOCK-2-4-1	15g 1/8 wedge, 15mm thick	0,7	**UOM**			
634-BLOCK-2-4-2	15g 1/8 wedge, 15mm thick	0,7	**CIEMAT**			
634-BLOCK-2-4-3	15g 1/8 wedge, 15mm thick	0,6	**CIEMAT**			
676-BLOCK-4-1	60,1g disk? 17mm thick?	1	**CIEMAT**			
634-BLOCK-2-4-4	15g 1/8 wedge, 15mm thick	0,6	**CIEMAT**	18	8	**CEA**
634-BLOCK-2-4-5	15g 1/8 wedge, 15mm thick	0,5	**CIEMAT**	30	10	**CEA**
676-BLOCK-4-4	68g disk?, 17mm thick?	1,2	**CIEMAT**	915	72	**CEA**
634-BLOCK-2-4-8	15g 1/8 wedge, 15mm thick	0,5	**CIEMAT**	28	28	**ENEA**
634-BLOCK-2-4-6	15g 1/8 wedge, 15mm thick	0,6	**CIEMAT**	30	10	**FzJ**
634-BLOCK-2-4-7	15g 1/8 wedge, 15mm thick	0,7	**CIEMAT**	25	8	**FzJ**
634-BLOCK-2-5	125g disk, 14mm thick	0,9	**CIEMAT**	200	25	**FzJ**

Samples received at IR-15 radioactive facility CIEMAT/DE/DFN/URBMA according to the safety QA requirements. The samples were unpackaged from a special steel transport container which was immediately checking in terms of surface contamination (inner and outer) and resending to origin with the absent of contamination certificate. This special transport was performed by ETSA.

At CIEMAT samples were weighted and the contact dose rate were measured which results are in table 1. Samples for each partner were packaging in Biotainer type A, it is a triple packaging designed for transporting type A radioactive material. (Developed in cooperation with Areva).

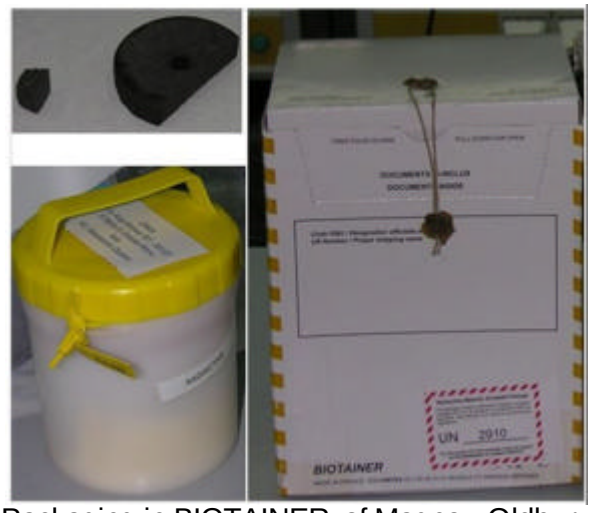

Figure 4.6 Packaging in BIOTAINER of Magnox Oldbury-2 samples

In order to reduce the dose rate and the budget of the shipping an extra led shielding was introduced inside de containers and the external boxes were sealed with adhesive tape to reinforce the structure.

A "Preliminary Agreement Application" was send to any receptor (Format in appendix 2) in order to authorise the reception of the material in the destination. With the specific activity sending with the samples from UK a individual packing list with full data of each shipments is fulfilled (Appendix 3).

The extra packaging conditions applied to the samples promoted non-conformity in the QA system at CEA Cadarache due to the un-fulfilment of transport and delivery requirements (Appendix 4).

4.4. UNGG Vandellos-1 Samples (CW-RRT)

Samples for CW-RRT were selected from a set of irradiated sleeves coming from Vandellos-1 reactor, taking from conditioning (milling) operation of this waste.

The samples were supply by ENRESA and at CIEMAT performed the homogenization of powder to build up a quasi-homogeneous sample that can be used for proficiency test.

Therefore powder samples from 5 different containers in origin with approximately 62 g were selected, mixed and divided into 5 g for 11 partners in the exercise.

Table 4.3. Mass of samples delivery per Lab

LAB#	GO m(g)	G1 m(g)	G2 m(g)	TOTAL m(g)	GV m(g)
L1	1.3009	2.3698	2.2539	5.9246	1.3093
L2	1.2812	2.2327	2.2944	5.8083	1.0821
L3	1.2977	2.2505	2.2156	5.7638	1.0152
L4	1.0797	2.2594	2.1780	5.5171	1.0265
L5	1.1829	2.2114	2.0986	5.4929	1.0556
L6	0.9660	2.2706	2.1661	5.4027	1.0938
L7	1.0621	2.2685	2.0813	5.4119	1.0357
L8	1.1392	2.4432	2.2435	5.8259	1.0919
L9	1.1394	2.4946	2.1382	5.7722	1.0471
L10	1.0308	2.1974	2.1046	5.3328	1.1601
L11	0.9713	2.1978	2.1869	5.3560	1.1671
TOTAL (g)	12.4512	25.1959	23.9611	61.6082	12.0844

After preparation of the vials that content the material, gamma spectrometry measurement of each vial (G-1, G-2 and G-0) of each lab were performed and checking the homogeneity using ANOVA test on the specific activity of 2 lines of Co-60, one line of Eu-154 and 1 line of Cs-137. The results are in the table and further explanation can be find in the results of the exercise report (D-3.1.5)[2]

Table 4.4. Data of the ANOVA test

ANOVA 1		^{60}Co 1173 keV
F	Prob	Crit.Value
17178.201	1.678E-79	3.9909237
ANOVA 2		^{60}Co 1332 keV
F	Prob	Crit.Value
14192.679	7.367E-77	3.9909237
ANOVA 3		^{152}Eu 334 keV
F	Prob	Crit.Value
235.72913	3.89E-23	3.9909237
ANOVA 4		^{137}Cs 662 keV
F	Prob	Crit.Value
311.9123	2.7E-26	3.9909237

In the results on the table we can see that in every case (even at micro-component level) the value of statistic F > Critical value, so that the sample can be distributed as homogeneous for the inter-comparison proposed and non-correction of the values will be applied in function of the heterogeneity of activity content.

For transport conditioning the following steps were performed:

- Selection of container.
- Packaging
- RP control

Boundary conditions for transportation obliges to reduce the dose rate to less than 5 µSv/h so that the samples must

The proposed selection of the container is to package the samples prepared according to the sample preparation [1] in order to have Exception packages. Due to the dose rate of the samples prepared (TABLE I) the vials must to be stored in a shielding container, individually or everyone, these stored into two over packages and finally the set stood in a box as final package.

Samples of 2g and 1g were placed into an individual already available shielding containers (from radiopharmaceutical items) (figure 4.7) being insufficient shielding for Dose rate reduction (especially in the bottom part of the shielding).

Special steel and lead containers were constructed in order to store the vials together and the reduction of the dose rate was not sufficient even with the over-package. (Figure 4.4)

Figure 4.7 Checking the dose rate for containers selection

Finally special shielding containers were design in cylindrical shape with 2 cm thickness wall and 5 cm thickness at the bottom.

The process of packaging consisted on:

- Label each Vial with his sample name and the mass of irradiated or virgin graphite and labelled each vial top with its sample name
- Insert each vial with irradiated graphite in the lead shielding container labelled with the sample reference and with the label “RADIOACTIVE”
- introduce the samples into the over-package and to put the top on
- Place the second over-package in the centre of the box introduce the first over-package with shielding containers and samples and to put the top on
- Introduce the packing list (example-format in appendix 3), to cover the set and to put the lid on.
- Label with the address of remittent and destination and with the perceptive ADR label.

External package has a dimension of 240 x 200 x 170 mm and a weight of approximately 3.5 kg (except for the one to UoM that had a weight approximately 3.9 kg).

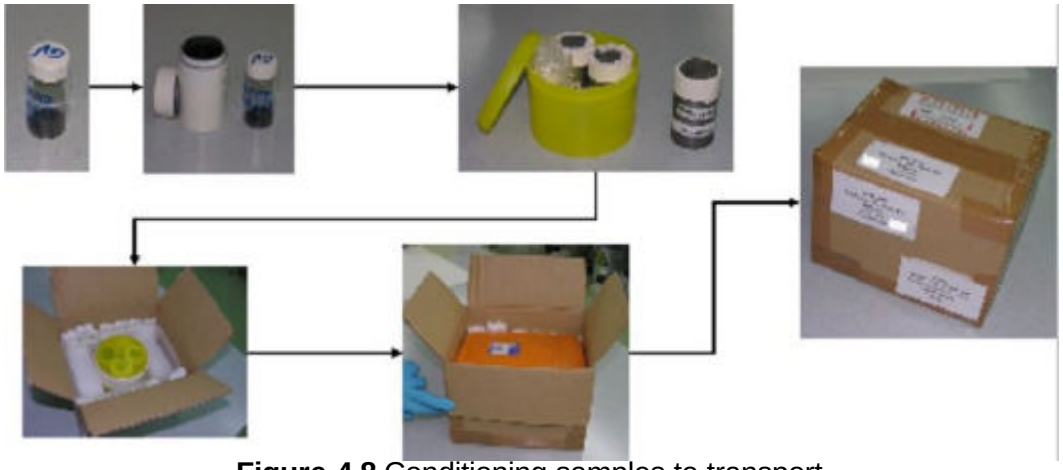

Figure 4.8 Conditioning samples to transport

Finally the Radiation Protection Control is performed measuring the surface contamination and the dose rate in contact at 1 m for transport requirements.

A strategy meeting among three parts: Transport Company (ETSA), Material Supplier (ENRESA) and Transport responsible (CIEMAT) was held in order to evaluate what was the best scheme for organise the transportation of samples, according to the location of lab's, Labs' requirements (dose rate, mass, dates, special conditions to receiving samples, etc.) and transport requirements in order to reach the objective of samples delivery and the most inexpensive transport cost due to budget limitations.

The general scheme was:

- Transport from original storage facility to CIEMAT (Madrid, Spain)
- Previous Acceptance sample format. (Appendix 1)
- Sample preparation and packaging at CIEMAT Lab
- Transport by road to
- France (3 labs)
- Italy (1 lab)
- Belgium (1 lab)
- The Netherlands (1 Lab)
- Germany (1 lab)
- Multimode Transport combined Air and Road mode to:
    - UK (1 Lab)
    - Romania (1Lab)
    - Lithuania (1 Lab)
    - Germany (1 Lab)

The Company in charge of transportation has agreement with the different national Radioactive Transport companies in the different countries to perform the final delivery. Companies involved were:

Figure 4.9 CWRRT Distribution map

- ① ETSA in Spain and Romania
- ② ISOLIFE in France
- ③ ISI in Benelux
- ④ MANFRED in Germany
- ⑤ MIT in Italy
- ⑥ BACUP in UK
- ⑦ AD REM UAB in Lithuania

The main requirement for transportation is the dose limitation (< 5 µSv/h) in order to fill-in the ADR641 for exception materials.

4.5. OTHER PARTICULAR PROCEDURES

4.5.1. Special Control at reception

Besides the normal RP control of the received samples and packages the extra control of the samples before preparing them for experimental work gives additionally information that could be essential in the interpretation of the results.

Checking the activity distribution of the sample by non-destructive method (i.e. gamma scanning) it is not an easy challenge due to normal sample size and the information getting from the study is only valuable for high energy gamma content. Another method applied in FzJ is the digital autoradiography that allows the determination of the emitters distribution in different ranges of energy and consequently the different nuclides distribution according to the macro-components of the sample.

In the figure we can see the test performed with one of the Magnox Oldbury samples delivered at Fz Jülich.

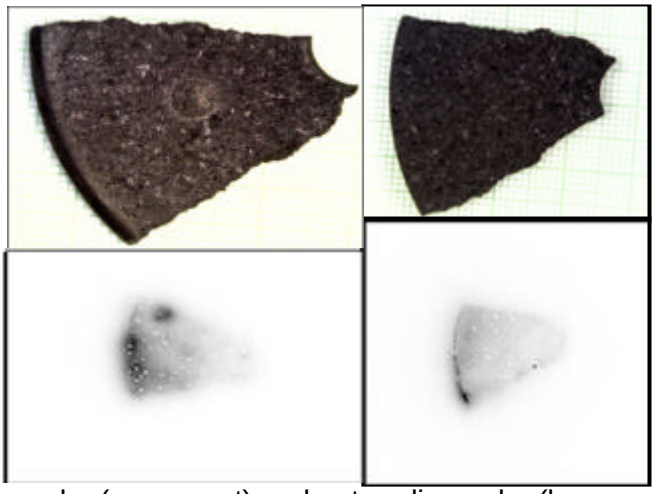

Figure 4.10 Photographs (upper part) and autoradiography (lower part) of OLDBURY 2 sample 634, 2-5 of the front side (on the left) and of the back side (on the right)

The analyses demonstrate a concentration of the activity in the external side of the circular sector that sample describes. By wipe sampling is demonstrated that non superficial contamination is associated with the distribution founded.

4.5.2. Sample Tests Preparation

In order to prepare samples for different analytical techniques and treatment experiments as well different techniques are described in the CW deliverables and technical reports [nnnn]. Here is described the method applied at CIEMAT for getting powder graphite from massive samples.

The powder graphite has been obtained filing on the graphite block surface with a grater coupled with a plastic box to collect the powder graphite (figure 4.11). This process has been performed treating each sample within a plastic bag inside a portable glove box placed into a fume hood chamber of the laboratory. After processing each sample the work area and the reusable stuff will be cleaned up applying decontamination methods.

Figure 4.11: Grater with plastic box

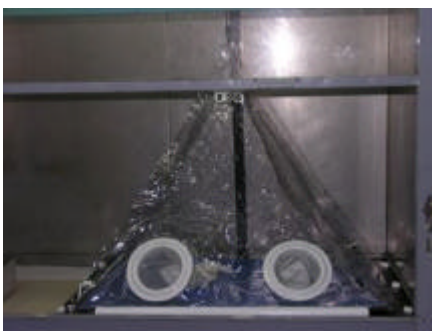

Figure 4.12 Portable glove box

5. USE OF SAMPLES WITHIN CARBOWASTE WP3&4

In this section is listed the use of the graphite samples within WP 3 and WP4 of Carbowaste.

FZJ

- UNGG VD 1:
  - Task 3.1 RRT
- UNGG SLA-2:
  - Task 3.2 Structural analyses by XRD
  - Task 3.3. Analytical procedures and analyses of
    - Gamma emitters
    - Pure beta nuclides: H-3, C-14, Cl-36,Sr-90 and Ni-63
    - X-ray: Ni-59 and Fe-55
    - Autoradiography
  - Task 3.3 Location of impurities: SIMS
  - Task 4.3.Thermal treatment
  - Task 4.3.Steam reforming
- MAGNOX Oldbury-2
  - Task 3.2 Structural analyses by XRD
  - Task 3.3. Analytical procedures and analyses of
    - Gamma emitters
    - Pure beta nuclides: H-3, C-14, Cl-36,Sr-90 and Ni-63
    - X-ray: Ni-59 and Fe-55
    - Autoradiography
- MTR-DIDO
  - Task 3.1 Sample Management
  - Task 3.2 Structural analyses by XRD
  - Task 3.3 Analytical procedures and analyses of
    - Gamma emitters
    - Pure beta nuclides: H-3, C-14, Cl-36,Sr-90 and Ni-63
    - X-ray: Ni-59 and Fe-55
    - Autoradiography
  - Task 4.3.Steam reforming
- MTR-MERLIN
  - Task 3.2 Structural analyses by XRD
  - Task 3.3 Analytical procedures and analyses of
    - Gamma emitters
    - Pure beta nuclides: H-3, C-14, Cl-36,Sr-90 and Ni-63
    - X-ray: Ni-59 and Fe-55
    - Autoradiography
  - Task 4.3. Sohlex extraction
  - Task 4.3. Thermal treatment: MS
  - Task 4.3.Steam reforming
- RMBK-Ignalina
  - Task 3.3. Analytical procedures and analyses of
    - Gamma emitters
    - Pure beta nuclides: H-3, C-14
    - Autoradiography
- HTR-AVR
  - Task 3.2 Structural analyses by XRD

- Task 3.3. Analytical procedures and analyses of
  - Gamma emitters
  - Pure beta nuclides: H-3, C-14, Cl-36,Sr-90 and Ni-63
  - X-ray: Ni-59 and Fe-55
  - Autoradiography
- Task 3.3 Location of impurities: SIMS, XPS; SEM
- Task 4.3. Sohlex extraction

CIEMAT

- UNGG VD 1:
  - Task 3.1 Sample Management and RRT
  - Task 3.2 Structural analyses by XRD BET and particle size
  - Task 3.3. Analytical procedures and analyses of
    - Alpha emitters: Am-241, Cm isotopes,Pu isotopes and U isotopes,
    - Gamma emitters: with different calibrations and geometries
    - Pure beta nuclides and volatile material: H-3, C-14, Sr-90. Cl-36, Tc-99 and I-129
  - Task 4.3. Chemical treatment decontamination. Intercalation process and SEM analyses
- UNGG SLA-2:
  - Task 3.1 Sample Management
  - Task 3.2 Structural analyses by XRD BET and particle size
  - Task 3.3. Analytical procedures and analyses of
    - Alpha emitters: Am-241, Cm isotopes,Pu isotopes and U isotopes,
    - Gamma emitters: with different calibrations and geometries
    - Pure beta nuclides and volatile material: H-3, C-14, Sr-90. Cl-36, Tc-99 and I-129
- MAGNOX Oldbury-2
  - Task 3.1 Sample Management
  - Task 3.2 Structural analyses by XRD BET and particle size
  - Task 3.3. Analytical procedures and analyses of
    - Alpha emitters: Am-241, Cm isotopes,Pu isotopes and U isotopes,
    - Gamma emitters: with different calibrations and geometries
    - Pure beta nuclides and volatile material: H-3, C-14, Sr-90. Cl-36, Tc-99 and I-129
- MTR-TRIGA-INR
  - Task 3.1 Sample Management
  - Task 3.2 Structural analyses by XRD BET and particle size
  - Task 3.3. Analytical procedures and analyses of
    - Alpha emitters: Am-241, Cm isotopes,Pu isotopes and U isotopes,
    - Gamma emitters: with different calibrations and geometries
    - Pure beta nuclides and volatile material: H-3, C-14, Sr-90. Cl-36, Tc-99 and I-129
- MTR-JEN-1
  - Task 3.2 Structural analyses by XRD BET and particle size
  - Task 3.3. Analytical procedures and analyses of
    - Alpha emitters: Am-241, Cm isotopes,Pu isotopes and U isotopes,
    - Gamma emitters: with different calibrations and geometries
    - Pure beta nuclides and volatile material: H-3, C-14, Sr-90. Cl-36, Tc-99 and I-129

CEA

- UNGG VD 1:
  - Task 3.1 RRT
- UNGG SLA-2:
  - Task 3.1 Sample Management
  - Task 3.2 Structural characterisation: Density, pycnometry, He porosity, Hydrostatic density, XRD
  - Task 3.3. Analytical procedures and analyses of
    - Gamma emitters:
    - Pure beta nuclides and volatile material: H-3, C-14, Cl-36 and Ni-63
  - Task 3.3 Location of Impurities:
- UNGG G-2:
  - Task 3.2 Structural characterisation: Density, pycnometry, He porosity, Hydrostatic density, XRD and Raman spectrometry, Microstructural characterisation
  - Task 3.3. Analytical procedures and analyses of
    - Gamma emitters:
    - Pure beta nuclides: H-3, C-14, Cl-36 and Ni-63
  - Task 3.3. chemical composition for Cl-36 characterisation

ENS

- UNGG SLA-2
  - Task 3.2. Characterisation with HRTEM, Raman and XRD
- UNGG G-2
  - Task 3.2. Characterisation with HRTEM, Raman and XRD

ENEA

- UNGG VD 1:
  - Task 3.1 RRT
- Magnox Oldbury-2
  - Task 3.3. Analytical procedures and analyses of
    - Gamma emitters:
    - Pure beta nuclides: H-3, C-14, Cl-36 and Ni-63
- Magnox Latina
  - Task 3.3. Analytical procedures and analyses of
    - Gamma emitters:
    - Pure beta nuclides: H-3, C-14, Cl-36 and Ni-63
    - Determination of impurities MS
  - Task 4.3. Chemical Treatment

ENRESA

- UNGG VD 1:
  - Task 3.3
    - Correlation factors calculations
    - Determination of impurities

INR

- UNGG VD 1:
  - Task 3.1 RRT
- MTR- Triga
  - Task 3.2.: Porosity
  - Task 3.3. Analytical procedures and analyses of
    - Gamma emitters
    - Pure beta: C-14

- Task 3.3. Analysis of impurities: EDS and XRF
- Task 4.3: Chemical treatment

FTMC

- UNGG VD 1:
  - Task 3.1 RRT
- RMBK-Ignalina
  - Task 3.3. Analytical procedures and analyses of
    - Alpha emitters: Am-241, Cm isotopes, Pu isotopes and U isotopes
    - Gamma emitters
    - Pure beta: H-3, C-14 and Cl-36

JRC

- MTR-MERLIN
  - Task 4.2.Pyrolysis treatment
- HTR-AVR
  - Task 4.2.Pyrolysis treatment
- Magnox Latina
  - Task 4.2.Pyrolysis treatment

NRG

- UNGG VD 1:
  - Task 3.1 RRT

SCK·CEN

- UNGG VD 1:
  - Task 3.1 RRT
- MTR BR-1
  - Task 3.3. Analytical procedures and analyses of
    - Gamma emitters
    - Pure beta: H-3, C-14 and Cl-36
- MTR Triga
  - Task 3.3. Analytical procedures and analyses of
    - Gamma emitters
    - Pure beta: H-3, C-14 and Cl-36

SUBATEC

- UNGG VD 1:
  - Task 3.1 RRT

UOM

- UNGG VD 1:
  - Task 3.1 RRT
- MAGNOX Oldbury-2
  - Task 3.1 Sample management
  - Task 3.2 Structural analyses by Pore structure analyses, Tomography, XRD BET, SEM, confocal microscopy, pycnometry and Raman
  - Task 3.3. Analytical procedures and analyses of
    - Gamma emitters:
    - Pure beta nuclides: H-3 and C-14
    - Distribution by autoradiograph

  - Task 4.3.: Thermal treatment

- MAGNOX Wilfa
  - Task 3.2 Structural analyses by Pore structure analyses, Tomography, XRD BET, SEM, confocal microscopy, pycnometry and Raman
  - Task 3.3. Analytical procedures and analyses of
    - Gamma emitters: with different calibrations and geometries
    - Pure beta nuclides: H-3, and C-14
    - Distribution by autoradiography
  - Task 4.2 Thermal Treatment
  - Task 4.3 Chemical leaching tests
- MTR-BEPO
  - Task 3.2 Structural analyses by Pore structure analyses, Tomography, XRD BET, SEM, confocal microscopy, pycnometry and Raman
  - Task 3.3. Analytical procedures and analyses of
    - Gamma emitters: with different calibrations and geometries
    - Pure beta nuclides: H-3, and C-14
    - Distribution by autoradiography
  - Task 4.2 Thermal Treatment
  - Task 4.3 Chemical leaching tests

6. ACHIVEMENTS AND REMARKS

The first achievement of this task is the wide distribution of irradiated graphite samples among the partners lab's to develop and applied competitive techniques over the main sources of irradiated graphite waste.

The effort and the compromise of the suppliers, EDF, Magnox, ENRESA, SOGIN, etc. together with the management agencies, regulators, member labs and project coordination make possible and effective the dissemination of samples in an effective an inexpensive manner.

Samples from the main sources of irradiated graphite within EU (UNGG, MAGNOX, RBMK HTR, MTR reactors) have been studied completing a not only a deep knowledge of this waste stream but a demonstrated extensive range of application.

Lessons learned on the management procedures imply an improvement on the standards applied on routine and will increase the reliability of the experimental results.

Main uses of the shearing and domestic samples were devoted to increase the Knowledge of the graphite characteristics after irradiation and to qualify the treatment and conditioning processes study in the project.

7. REFERENCES

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[6] Carbowaste Deliverable D-3.3.1.First Radionuclide Inventory Data on Untreated Graphite / Evaluation for Direct Disposal; D. Vulpius et al. 2010

[7] Carbowaste Deliverable D-3.3.2. Report on analysis of impurity and isotope sites before and after treatment; K.Baginski et al. 2010.

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[9] Carbowaste Deliverable (D.4.1.3) Final report on the relationships between structure and isotope distribution in graphite; Abbie Jones et al;2013

[10] Carbowaste Deliverable (D-4.3.6) Decontamination factors by treatment with oxygen; L.Mcdermott et al. 2013

[11] Carbowaste Deliverable D-4.3.4 Decontamination Factors by Steam Reforming; B. Kraus; 2013

[12] Carbowaste Deliverable (D-3.2.1) First structural data of virgin and irradiated graphite before treatment; A. Jones et al. 2010

[13] Carbowaste Technical Report T-4.2.2 Radionuclide release from contaminated graphite by high temperature pyrolysis

[14] **ADR**: The European Agreement concerning the International Carriage of Dangerous Goods by Road, (ADR from the French abbreviation Accord européen relatif au transport international des marchandises Dangereuses par Route), governs transnational transport of hazardous materials.

APPENDIX 1. Questionnaire for Sample Distribution

QUESTIONNAIRE.

The reasons to distribute and fill in this questionnaire are the following:

- To know the virgin and irradiated graphite available for the project,
- To fix the graphite samples to be characterised for each lab,
- To define the abilities of each partner in Characterisation processes and
- To define the characterisation processes to be development, implemented or needed for each partner.

1. **PARTNER:**

2. **What types of graphite are you interested in characterised into the project?**

☐ VIRGIN GRAPHITE

☐ MTR	☐ I need _____g	☐ I have enough
☐ UNGG	☐ I need _____g	☐ I have enough
☐ Magnox	☐ I need _____g	☐ I have enough
☐ _____(HTR/RBMK/other)	☐ I need _____g	☐ I have enough

☐ IRRADIATED GRAPHITE

☐ MTR	☐ I need _____g	☐ I have enough
☐ UNGG	☐ I need _____g	☐ I have enough
☐ Magnox	☐ I need _____g	☐ I have enough
☐ _____(HTR/RBMK/other)	☐ I need _____g	☐ I have enough

3. **Do you have enough graphite for delivering to other partners?**

☐ VIRGIN GRAPHITE

☐ MTR	Amount (g) _____
☐ UNGG	Amount (g) _____
☐ Magnox	Amount (g) _____

☐ _____(HTR/RBMK/other) Amount(g) _____

☐ IRRADIATED GRAPHITE

☐ MTR Amount (g) _____

☐ UNGG Amount (g) _____

☐ Magnox Amount (g) _____

☐ _____(HTR/RBMK/other) Amount(g) _____

4. Which are the presently developed structural analysis methods at your lab to be used in the project?

☐ EELS ☐ EDX ☐ HR Tomography ☐ O. Microscopy

☐ HRTEM ☐ XRD ☐ Raman Microscopy ☐ BET

☐ _____ ☐ _____ ☐ _____ ☐ _____

☐ _____ ☐ _____ ☐ _____ ☐ _____

5. Have you developed methodologies for diffusion coefficient determination in graphite samples? Are you interested in its application in this project?

6. Which are the presently developed radiochemical analysis methods at your lab to be used in the project?

☐ Gamma Emitting Nuclides

☐ Co-58 ☐ Fe-59 ☐ Co-60 ☐ Ru-106 ☐ Ag-108m ☐ Ag-110m ☐ Sb-125

☐ Cs-134 ☐ Cs-137 ☐ Ce-144 ☐ Eu-152 ☐ Eu-154 ☐ Eu-155 ☐ Am-241

☐_____ ☐_____ ☐_____ ☐_____ ☐_____ ☐_____ ☐_____

☐ Beta Emitting Nuclides

☐ H-3 ☐ C-14 ☐ Cl-36 ☐ Fe-55 ☐ Ni-59 ☐ Ni-63 ☐ I-129 ☐ Pu-241

☐____ ☐_____ ☐_____ ☐_____ ☐____ ☐_____ ☐_____

☐ Alpha Emitting Nuclides

☐ Pu-238 ☐ Pu-239/40 ☐ Am-241 ☐ Cm-242 ☐ Cm-244 ☐ U-234 ☐ U-235

☐ U-236 ☐ U-238 ☐_____ ☐_____ ☐_____ ☐_____ ☐_____

7. **Taking into account your participation in the project. Have you characterisation abilities available to complete the characterisation of other partners?**

8. **Taking into account your participation in other WP´s, Do you need the characterisation abilities from other partners in the project?**

9. **Write a brief description of your work at CW in each task/deliverable (one paragraph per deliverable is enough)**

APPENDIX 2. Preliminary Agreement Aplication

/D SDUWGHIP DJHO «

PRELIMINARY AGREEMENT REQUEST

/D SDUW GHIP D1

Record N° : 1 **Date :** 02/07/2013

Deliver Unit: CIEMAT/IR-15 **Building** : 18 **Department** :DE/DFN/URBMA

Centre CIEMAT

Shipper	**Transport Responsible:**
Family Name: PIÑA Phone : +34 91.346.64.	Family Name: RUIZ Phone l : +34 91.346.61.
Name: GABRIEL Fax : +34 91.346.65.	Name: EDUARDO Fax : +34 91.346.65.
Expedition Place : Madrid Address : Ciemat DE/DFN/URBMA Av. Complutense 22 28040-SPAIN	Expedition Place : Madrid Address : Ciemat DE/DFN/URBMA Av. Complutense 22 28040-SPAIN

Recipient Unit:

Recipient	**Transport Responsable (at destination)**
Fam. Name: VULPIUS Phone : +49 2461 61-5071	Fam. Name: PLUM Phone : +49 2461 61-3396
Name: DIRK Fax : +49 2461 61-2992	Name: MANFRED Fax : +49 2461 61-2488
Destination Place : Forschungszentrum Jülich GmbH Address : Leo-Brandt-Str., 52428 Jülich (Germany)	Destination Place : Forschungszentrum Jülich GmbH Address : Leo-Brandt-Str., 52428 Jülich (Germany)

Type of sample: 3 Graphite samples ..... **Physic-chemical state** : solid
Dimensions: 210 x 200 x 275 **mm** **Weight**: MAX. 2 kg **Sample Mass**: Packing list.....
Tot. Mass : 43 g **Activity & DR** : Packing list..... **DR max** : 17 µSv/h
Radionuclides: ^{3}H, ^{14}C, ^{55}Fe, ^{63}Ni, ^{60}Co, ... **Total Activity** : $5.05 \cdot 10^{8}$ Bq

NUCLEAR MATERIALS : NON

TRANSPORT :
Type : Type A Biotainer
Date prévue : October
Remarques du Correspondant transport :

Applicant : Family Name : Piña Name: Gabriel **Signature**
Unit : URBMA/DFN/DE Company: CIEMAT phone : +34913466476

AUTHORIZATION OF THE RECEIVING INSTALLATION:
AGREE **YES ☐** **NO ☐**
Recipient
Date : Name : **Recipient Signature:**
Transport Responsible (at destination)
Date : **Name :** **Transport Responsable Signature :**

APPENDIX 3. Packing List

LAB:	**FZJ**
CONTACT PERSON:	**Dr. Dirk Vulpius**
Address:	**Forschungszentrum Jülich GmbH 52425 Jülich (Germany)** **Leo-Brandt-Str., 52428 Jülich (Germany)**
e-mail:	**d.vulpius@fz-juelich.de**
FAX:	**+49 2461 61-2992**
Phone:	**+49 2461 61-5071**
SAMPLES TRANSPORT DATA	**FZJ**
CONTACT PERSON:	**Manfred Plum**
Address:	**Forschungszentrum Jülich GmbH** **Leo-Brandt-Str., 52428 Jülich (Germany)**
e-mail:	**m.plum@fz-juelich.de**
FAX:	**+49 2461 61-2488**
Phone:	**+49 2461 61-3396**
Dimensions:	210 x 200 x 275mm
Weight:	< 2.000 kg

Sample ID	(g)	(mSv/h) contact	mSv/h contact shielding	mSv/h contact package	uSv/h 1 m package	Nuclides	Total Activity (Bq)
634-BLOCK-2-4-6	**10**	**30**	**70**	**17**		**^{3}H, ^{14}C, ^{55}Fe, ^{63}Ni, ^{60}Co**	1.17E+08
634-BLOCK-2-4-7	**8**	**25**					9.39E+07
634-BLOCK-2-5	**25**	**200**					2.93E+08
MASS TOTAL (g)	**4.3E+01**				**TOTAL ACTIVITY (Bq)**		**5.05E+08**

APPENDIX 4: Receip with Non-Conformities

Direction de l'énergie nucléaire
Département de services nucléaires
Service d'exploitation d'expertise et de caractérisation

énergie atomique • énergies alternatives

CEADENCADDENSEECOLEC
DO 116 07/12/11

11FPDE0000116

diffusé le : 07/12/11

M. Gabriel Piña

CIEMAT/DE/DFN/URBMA
AVDA. COMPLUTENSE, 22 – ED. 18
28040 MADRID
ESPAGNE

Cadarache, December 7th, 2011

Object : Receipt with non-conformities of a biotainer contaning magnox graphite samples

Dear Sir,

We have received on November 17th, 2011, on the Chicade hot laboratory facility of the Cadarache CEA center, a Biotainer containing magnox graphite samples.

The driver arrived on the Cadarache center and **went first to the building 152,** as indicated on this consignment note and finally went to building 326 (Chicade nuclear installation) where the transport was accepted, in spite of the **absence of a previous transport notification.**

The documents accompanying the transport on arrival were the following :

- One consignment note (Cf. enclosure 1) indicating a radioactive materiel assigned to the category "II-yellow", a Transport Index (TI) of 0.2 and a mass of 11 kg, signed by the shipper and the conveyor,
- One "consignment note" (Cf. enclosure 2) indicating a radioactive materiel assigned to the category "III-yellow", a Transport Index (TI) of 1,2 and a mass of 11 kg, **not signed by the shipper and the conveyor,**
- A copy of the preliminary agreement for the reception of magnox sample in the Chicade nuclear installation (Cf. enclosure 3).

The Biotainer, on arrival, showed the following characteristics :

- The internal container was sealed, with a label indicating the consignee address (Cf. picture number 1),
- The external container was sealed, closed with the self-adhesive Scotch of the 4th flap of the packaging and **by using 2 red Scotch tape bands** on the top of packaging (Cf. picture number 2),
- Indication on the packaging of :
  - the UNO number preceded by the letter UN letters (UN 2915),
  - the the proper shipping name (radioactive material, type A packages),
  - the consignor and the consignee address,
  - the durably marked on the outside of the packaging with "TYPE A"; the standard mention (F)
- Labels "II-yellow" , TI = 0,2, activity 1.06 GBq, radioactive contents : H-3, C-14, Fe-55, Co-60, Ni-63,
- **Measured mass : 7.8 kg.**

Commissariat à l'énergie atomique et aux énergies alternatives
Centre de Cadarache – Bâtiment 707 – 13108 Saint-Paul-lez-Durance Cedex
Tél. : 33 – 4 42 25 41 67 – Fax : 33 – 4 42 25 65 34

Etablissement public à caractère industriel et commercial
R.C.S. PARIS B 775 685 019

cea

énergie atomique - énergies alternatives

We have accepted the package but noticed several non-conformities with the ADR rules :

- Absence of a consignment note who must be transmitted at least 48h before the departure of the transport. It allows to accept beforehand the transport, with the date of arrival, the name of the driver, the registration number of the vehicle and the list of the contents. This consignment note allows us to accept the packaging for the day of delivery envisaged, to prepare the documents permitting the access on the Nuclear Center for the driver and to warn the safety and radioprotection services of the center that a radioactive transport will arrive. The preliminary agreement for the reception does not replace the consignment note,
- Absence of the copy of the nuclear civil responsibility of the loading,
- Presence of 2 red Scotch tape bands instead of 4 transparent Scotch tape bands on the top of the packaging as requested in the compliance certificate of the packaging,
- Total mass higher than the maximum permissible weight in this type of packaging (7,8 kg measured instead of 2.65 kg authorized),
- Error in the documents and on the packaging for the consignee address (building 152 instead of 326) and for the name of the consignee (Mr. Pierry instead of Mr. Comte).

For your memory, the consignor is responsible for sending a conform packaging. So, to improve the practices, we ask you by advance to transmit us the measures you will take so that such an event will not be reproduced.

Jean Philippe DANCAUSSE
Head of Chicade hot laboratory facility

enclosure 1 : signed consignment note
enclosure 2 : Non signed consignment note
enclosure 3 : preliminary agreement for the reception