CArbon-14 Source Term

CAST

Determination of ^{14}C Content and Release from Steel (D2.10)

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CAST – Project Overview

The CAST project (CArbon-14 Source Term) aims to develop understanding of the potential release mechanisms of carbon-14 from radioactive waste materials under conditions relevant to waste packaging and disposal to underground geological disposal facilities. The project focuses on the release of carbon-14 as dissolved and gaseous species from irradiated metals (steels, Zircaloys), irradiated graphite and from ion-exchange materials as dissolved and gaseous species.

The CAST consortium brings together 33 partners with a range of skills and competencies in the management of radioactive wastes containing carbon-14, geological disposal research, safety case development and experimental work on gas generation. The consortium consists of national waste management organisations, research institutes, universities and commercial organisations.

The objectives of the CAST project are to gain new scientific understanding of the rate of re-lease of carbon-14 from the corrosion of irradiated steels and Zircaloys and from the leaching of ion-exchange resins and irradiated graphites under geological disposal conditions, its speciation and how these relate to carbon-14 inventory and aqueous conditions. These results will be evaluated in the context of national safety assessments and disseminated to interested stakeholders. The new understanding should be of relevance to national safety assessment stakeholders and will also provide an opportunity for training for early career researchers.

For more information, please visit the CAST website at:
http://www.projectcast.eu

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Executive Summary

Within the CAST project, the JRC Karlsruhe (formerly JRC Institute for Transuranium Elements) developed a new device for the determination of total carbon using a gas extraction method. For the steel investigations in WP2, an accuracy of 10 to <1% can be expected for carbon contents in the 5 to 500 ppm range respectively. Further equipment to perform leaching tests and ^{14}C determination by liquid scintillation counting (LSC) has been prepared. Their deployment has been hindered due to technical difficulties and finally due to an upgrade of the cells to meet new legislation. This report summarises the achievements made so far.

This report has been compiled and edited by J. Somers following the retirement of the project leader David Bottomley in February 2017.

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CAST D2.10 Determination of ^{14}C Content and Release from Steel

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Title CAST D2.10 Determination of 14C Content and Release from irradiated steel cladding

Abstract

Within the CAST project, the JRC Karlsruhe (formerly JRC Institute for Transuranium Elements) developed a new device for the determination of total carbon using a gas extraction method. For the steel investigations in WP2, an accuracy of 10 to <1% can be expected for carbon contents in the 5 to 500 ppm range respectively. Further equipment to perform leeching tests and ^{14}C determination by liquid scintillation counting (LSC) has been prepared. Their deployment has been hindered due to technical difficulties and finally due to an upgrade of the cells to meet new legislation. This report summarises the achievements made so far.

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1 Introduction

The main objective of these investigations is the release and speciation of ^{14}C during corrosion of irradiated steels under disposal conditions pertinent to a cement-based repository. Even though one might expect very low release rates under such conditions, considerable uncertainties exist, and are acerbated as ^{14}C is a difficult to measure nuclide. The potential speciation of ^{14}C is particularly important, especially the relative proportions of organic ^{14}C vs. inorganic ^{14}C that might be released. Thus the degree of speciation can also affect the safety assessment of the waste repository.

The main objectives of WP2 of the CAST project are the identification and quantification of ^{14}C species formed during corrosion of irradiated steels under conditions expected in cement based repositories. Overall, the project aims at reducing uncertainties and setting “bounds” on the release rates. Improved measurements, or even bounded uncertainties lead to reduction in conservatism, and more realistic safety assessments.

A major effort in CAST WP2 is the development of methods to extract (or leach under cementitious conditions) ^{14}C from neutron activated steels (structural materials, cladding from fast reactors, etc.) as well as the improvement of methods to assay ^{14}C in differing chemical species. The determination of typical release rate from the steel and concomitant dose rates, under conditions expected in the repository is of essential importance in the overall safety assessment. Migration of ^{14}C is of substantial interest in such safety assessments. In the case of ^{14}C, this aspect is of unprecedented importance as its transport can occur in a number of ways, i.e. in gaseous form (CO, CO_2, CH_4,…) as well as in liquid form (hydrocarbons or carboxylic acids, etc.). The total ^{14}C released and its rate of release represent important parameters, but universalisation of the data implicitly requires a deep knowledge of the ^{14}C speciation, i.e. the fractional release rates of the chemical species involved.

The JRC Karlsruhe (formerly JRC ITU) contributes to the determination of the influence of pH on the corrosion rate (see review by Swanton 2014). Though highly alkaline conditions would be expected in cementitious conditions, lower pH values could also prevail (e.g. if decomposition of organic materials would neutralise the alkalinity). WP2 partners cover arrange of pH with JRC focussing on pH 5-6. Thus the contribution of the JRC is

- the determination of ^{14}C in AIM1 steel irradiated in a fast neutron flux,
- the determination of the release rate of ^{14}C therefrom at pH 12 and below

For this work JRC Karlsruhe selected steel cladding from experimental fuel pins irradiated in the Phenix fast reactor in France. As such, the results have a direct bearing on wastes produced in a closed fuel cycle, i.e. the samples and their preparation simulate accurately the conditions encountered for the disposal of fuel cladding hulls, packed for storage after the fuel has been dissolved using PUREX conditions. On account of the acid wash (essential for the determination method) the results might not exactly emulate the conditions encountered for disposal of structural components from various reactor types. This report summarises the progress and achievements made.

Waste management is a key component of the JRC direct actions in support of the Member States.

2 Materials

2.1 Material selection

The JRC Karlsruhe chose stainless steel cladding material for the tests. The samples were derived from AIM1 (15:15Ti) steel as used and licensed in the Phenix reactor. This material was chosen as it likely has been exposed to a higher neutron flux (albeit in a short time ~ 1 year) than in the case of steels in Light Water Reactors (LWR), where in reactor life can be far longer. The samples themselves stem from the irradiation programme known as NIMPHE 2, where carbide ($(U_{0.8}Pu_{0.2})C$ designated pin number NI-27) and nitride ($(U_{0.8},Pu_{0.2})N$ designated pin number NI 25) fuels were irradiated under relatively high linear power conditions in the Phénix reactor in the late 1980s and early 1990's. The mean burnups achieved were 53.8 GWd.t^{-1} and 51.6 Gwd.t^{-1} for the nitride and carbide fuels, respectively. In contrast to LWR pins, where a relatively flat burnup profile along the longer pins is found, considerably peaking occurs at the mid-plane in the Phenix reactor, where the fuel column is only about 1m. At the mid-plane position, one expects a higher ^{14}C generation.

Virgin samples of this type of cladding material were supplied to the JRC Karlsruhe from CEA and other organisations, explicitly for the preparation of test irradiation of innovative fuels. These samples have been stored securely in the meantime and recovered for a series of chemical analysis by Inductively Coupled Plasma Mass Spectrometry (ICPMS) on dissolved samples have been performed to determine the metallic impurities. There was no pre-treatment of the samples in advance of dissolution. The non-metallic impurities were determined also by a total combustion technique (also known as gas extraction method). The results are shown in Tables 2.1 and 2.2. The major metallic constituents were confirmed (14.97%Ni, 14.97% Cr, 0.22% Ti (and 0.4% Si)). All other elements were within usual specifications. The major non-metallic impurity is carbon, whose content (789 ppm) was less than specification as desired (<1000 ppm). During this set of measurements, the operators noted difficulties so that a second campaign was made under contract with the company Bruker in Karlsruhe. The results are given in Table 2.3. The repeat measurements were not entirely in agreement with the first set, but they are very close.

An important caveat in this material selection should be mentioned. Both carbide and nitride fuel swell considerably under irradiation. The NIMPHE 2 irradiation test was no exception. Gap closure occurred. Thus, there exists a distinct possibility that carbon from the carbide fuel, and as an ever present impurity in the nitride fuel (prepared by carbothermal reduction of the oxides), could have diffused into the surface regions of the steel cladding. Furthermore, ^{14}C produced in either of these cladding samples during irradiation could be enhanced compared to the fresh unirradiated materials by diffusion of ^{14}C produced in the fuel itself. Indeed, the nitride fuels were produced with natural nitrogen, from whence additional ^{14}C would have been generated in significant quantities during irradiation. It should be borne in mind that the total carbon content in the cladding will have increased during irradiation, and that a non-uniform distribution of the carbon in the cladding may have existed at the end of irradiation.

Table 2.1 Steel cladding metallic constituents determined from ICP MS measurements

Element	Concentration (wt%)	Uncertainty (absolute)
B	<0.0070	
Si	0.412	0.021
P	0.0482	0.0024
Ti	0.2261	0.0045
Cr	14.97	0.30
Mn	1.253	0.025
Fe	67.0	1.3
Ni	14.97	0.30

Table 2.2 Steel cladding non-metallic constituents determined by total combustion methods

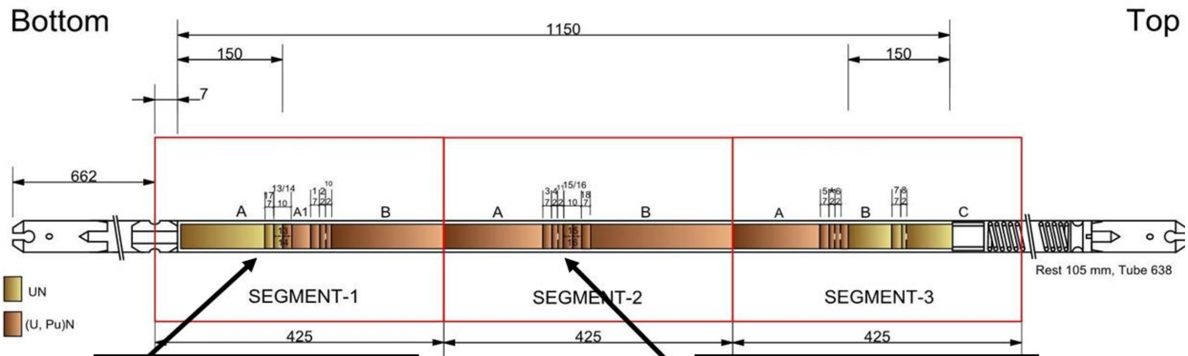
Element	Concentration (wt%)	Uncertainty (absolute)
C	0.0789	0.0055
O	0.00267	0.00093
N	0.0041	0.0023

Table 2.3 Repeat steel cladding non-metallic constituents determined by total combustion methods at Bruker

	JRC Karlsruhe		Bruker	
Element	Concentration (wt%)	Uncertainty (absolute)	Concentration (wt%)	Uncertainty (absolute)
C	0.0789	0.0055	0.0971	0.0007
N	0.0041	0.0023	0.0075	0.0006

2.2 Material preparation for the leach testing

Samples in the form of 3-8mm long rings (1-0.5g) were cut from the NIMPHE carbide & nitride fuel pins as shown for the nitride fuel pin (carbide is the same) in Figure 2.1. All rings were defueled mechanically before applying chemical removal, which simulates the condition of a fuel rod hull following aqueous recycling in the PUREX process. For chemical removal, it was planned to dip the sample twice in acid (8M HNO_3) for 15 minutes each dip. The samples were then to be washed in distilled water in an ultrasonic bath and finally allowed dry in the hot cell ambient atmosphere (dry N_2). There is of course some risk that the acid treatment could cause modification of the surface, potentially leading to a lack of “availability” of carbon for leaching, but this is an unavoidable step as the final ^{14}C determination would be rendered useless should fuel cross pollute the leachates. An example of the defueled (chemically uncleaned) sample from a NIMPHE 2 carbide fuel is shown in Figure 2.2.



rings and segments from top of fuel/ plenum samples 13-14, 17

5mm rings from mid-height (max. power) - samples 15,16 & 18

Figure 2.1 Cutting plans for steel sample rings from NIMPHE 2 pins


a) b)

Figure 2.2: NIMPHE carbide fuel cladding following defueling. A dark thin oxide layer is observed before cleaning.

3 Methods and experimental conditions

3.1 Collection of Carbon from the Samples using the gas extraction method

Total carbon measurement in carbide and nitride fuels has been made at JRC Karlsruhe for more than 40 years and was driven by the advanced nitride and carbide fuel for fast reactor programmes. In this manner, JRC Karlsruhe has built up a strong capability and knowledge base for Carbon determination using so called gas extraction method, which is often referred to as a total combustion method.

To our knowledge, no such gas extraction facility has been installed in a hot cell environment, for carbon determination though many laboratories have such devices installed for H determination in cladding materials. For the CAST project, JRC Karlsruhe has purchased a device from Bruker known as ICARUS (see Figure 3.1). The device is centred around a compact high frequency induction furnace in which the sample is incinerated in a flowing oxygen atmosphere to generate CO_2, which is then detected using a solid state infrared detector system (see Figure 3.2 – JRC Karlsruhe device is not equipped for Sulphur detection). About 1 gram of high purity copper is added to the crucible for each measurement as an accelerator (achieved by the relatively low melting temperature of Cu). This addition means that the sample melts quickly and that a large pulse is detected rather than a broad peak. Due to the integration over time, the former signal yields more accurate data. Supplier specifications cover operation in the 1-60000 ppm range for sample masses in the 0.5-1.0 gram range. Calibration is performed against certified standards. The device measures total carbon. The detector system is not capable of distinguishing between ^{14}C and ^{12}C.

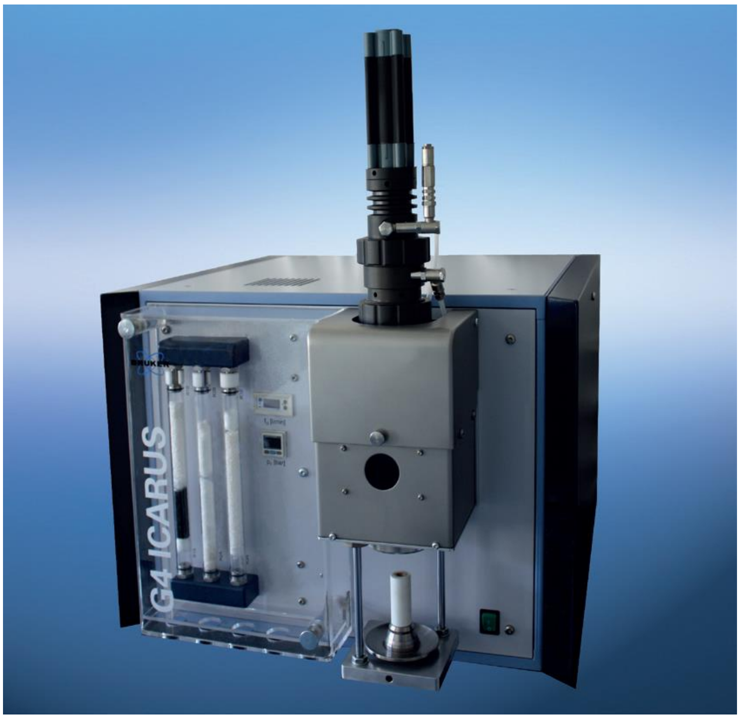


Figure 3.1 ICARUS device in its standard configuration

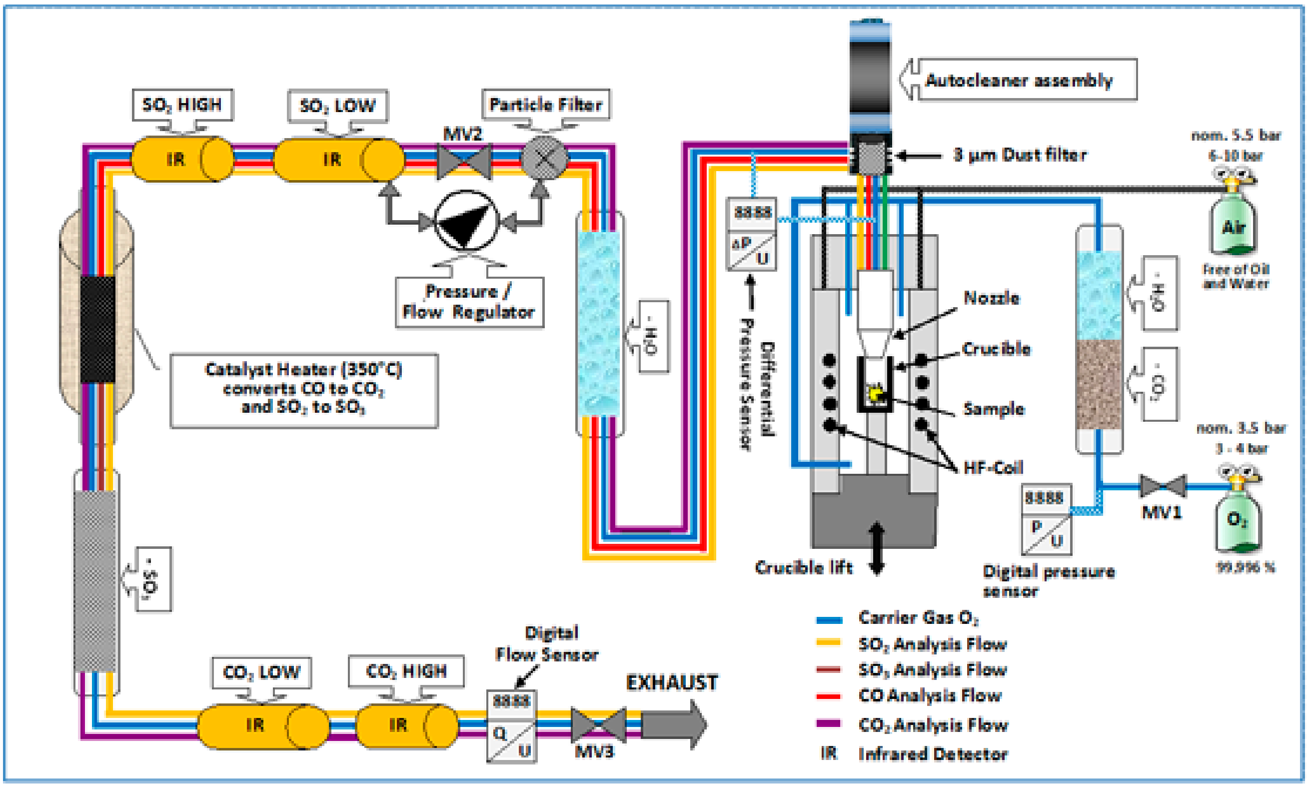


Figure 3.2 ICARUS device operating principle

The device needed major adaptation to render it suitable for hot cell application, so that the major electronic components had to be separated from the core component to be incorporated in the hot cell. This has been achieved and the separated device is shown in Figure 3.3. The overall layout and integration of the device for hot cell work is shown in Figure 3. 4. Briefly, only the section containing the furnace is inserted in the hot cell. To do so a lengthy procedure is needed and is achieved in the following steps.

- The hot cell must be thoroughly cleaned using its telemanipulators so that manual intervention is possible (i.e. dose rates must be reduced to a minimum)
- An intervention cell is docked onto the back of the hot cell
- Physical access to the cell is then achieved, whereby staff operating in fully ventilated suits, open the door and lift the equipment inside.
- Connecting all cables for power and sensors to the standard connector bank in the cell
- Connecting the gas lines from the ICARUS furnace via double filters to the outside, from whence the gas passes through the detector system, before being returned into the cell via a second set of double filters.

Within this configuration, total carbon is measured directly by the instrument. All carbon generated is collected on a molecular sieve, located in the cell. For ^{14}C determination, this molecular sieve must be transferred to the ^{14}C preparation glove box, which is described later.

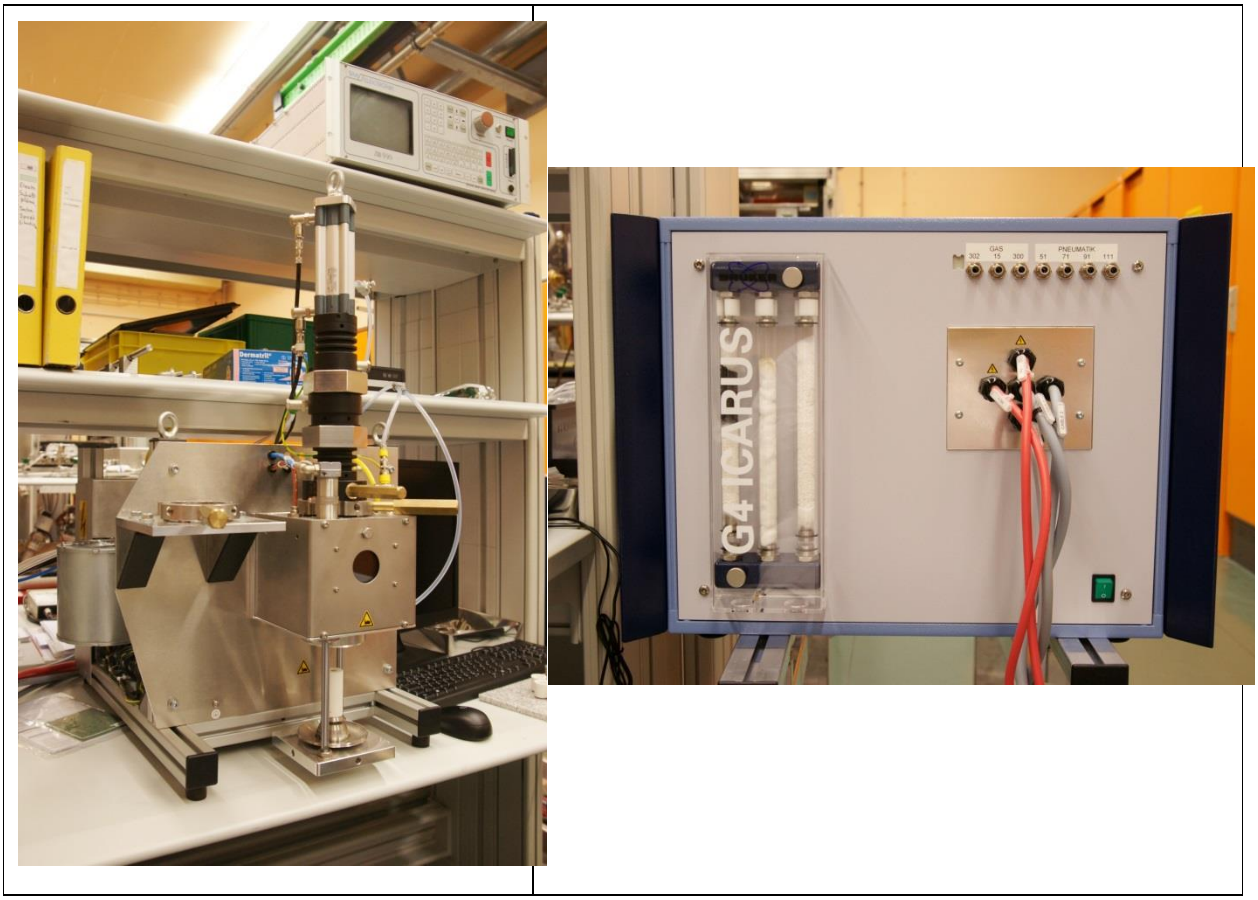


Figure 3.3 Icarus device separated into 2 components

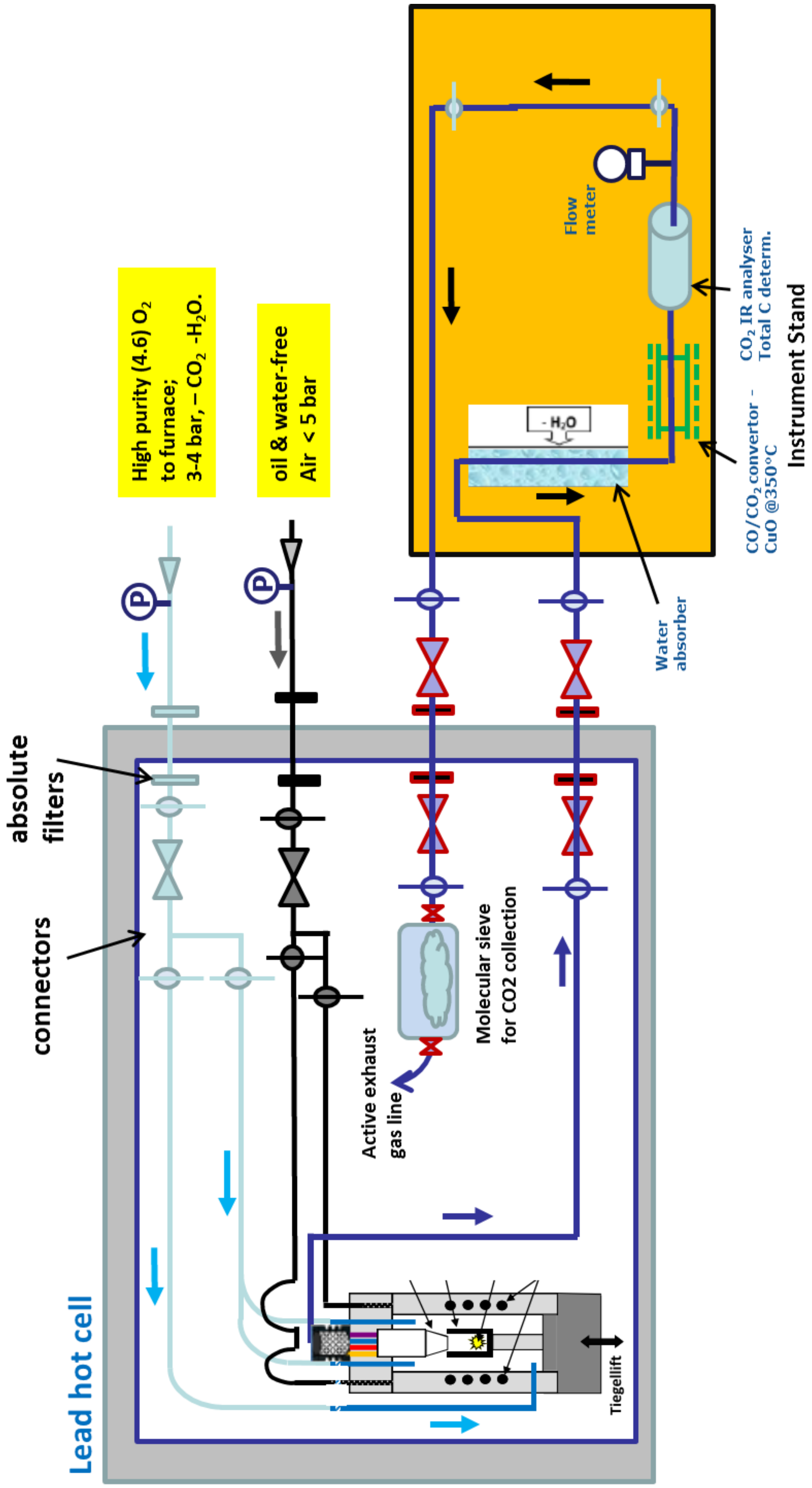


Figure 3.4: Concept for integration of ICARUS device in a Hot Cell

On delivery, the performance of the device has been determined against a set of materials containing known quantities of carbon. The results are summarised in Table 3.1 below. The calibration is made with a set of standards. In all cases preheated crucibles have to be used to ensure no residues of carbon are within the sample crucible itself. The set of follow up measurements made with the same standards show exceptionally good agreement.

Table 3.1: Measured Carbon content in a set of qualification materials

	Calibration			Repeat measurements			
Sample	Mass (g)	[C] ppm	SD*ppm	Mass (g)	[C] ppm	SD ppm	No of analyses
Blank (Cu)	1						
CRM 088-2	1	6	2	1	5.51	0.51	5
ZRM 191-2	1	43	2	0.5 1.0	43.98 43.45	1.90 0.38	5 5
ZRM 284-2	1	201	5	0 1.0	202.48 200.77	1.11 1.68	5 5
IRSID 114-1	0.5	440	20	0.5	442.91	1.63	5
Stainless steel				0.16	956.24	2.94	3

* Data provided by the standard supplier

3.2 Leaching of ^{14}C from samples in autoclaves

While the ICARUS device provides a convenient method to collect total carbon from a sample, the same can be achieved using an autoclave, when the sample is digested. For the CAST project, however, an autoclave is an essential tool to collect ^{14}C from a leach test, whereby the sample is exposed to an aqueous solution with a chemical composition similar to that expected in a geological repository.

The autoclave is essentially an in expensive device, and at the JRC-Karlsruhe hot cells lends itself to insertion in a cell via the connecting tunnel underneath the cells, whose dimensions are large enough to permit its transfer to the cell. Assembly has to be made by telemanipulators, with a restriction that components should weigh less than 5 kg.

The major operational problem encountered in a JRC Karlsruhe hot cell is that of potential cross contamination. JRC Karlsruhe hot cells are old (some approaching 50 years) and extremely dirty, so that careful handling and packaging of all of the experimental components is essential, especially as the final ^{14}C determination was to be made by Liquid Scintillation Counting (LSC). Successful application of this technique requires that the final ^{14}C sample is free of radioactive isotopes that could interfere with the measurement.

Two autoclaves were prepared at JRC Karlsruhe to permit two experiments to run in parallel, a contingency plan to mitigate the consequences of ever increasing delays within the project. The autoclave with its internal components is shown in Figure 3.4. The device can withstand severe conditions (150 bar and 200°C), but for the purpose of the CAST programme was to be operated at 1-2 bars excess pressure (Argon) and without heating (i.e. at hot cell ambient temperature of about 30°C). The internal liner is made of PEEK, a material highly resistant to strong irradiation fields. The samples themselves are placed on pre fired ceramic holders. The second autoclave is shown in Figure 3.6 and has similar characteristics.

Figure 3.5: Berghof autoclave (left) with its gas sampling vessel, and a view of its internal components (right)

Figure 3.6: Second Autoclave

A key aspect of the CAST project is the collection of the ^{14}C sample, which can be in both liquid and gaseous forms. Liquid samples can be removed by direct sampling, taking due care that the vessels do not pick up contamination from the surrounding hot cells.

The gas sample is more problematic and is achieved using a gas sampling vessel (a transport vessel to collect gas see Figure 3.5). At the end of the experiment, the valve connecting the gas sampling vessel to the autoclave is closed and the gas sampling vessel unit can be detached using the quick fit connector system and transferred into the hot cell support tunnel. From there, it can be removed from the hot cells using the main auxiliary support glovebox at the end of the service tunnel. Again, contamination is an issue (especially in view of 3 month leaching times), and must be avoided. To achieve this goal the gas sampling vessel is packed in 3 layers of polyethylene sacks. The last sack is installed on the gas sampling vessel, just when it is connected in the cell. This outer layer is removed in the hot cell, before its transfer to the tunnel. The next sack is removed in the support hot cell, and the gas sampling vessel is bagged out using a double bag system to be installed in the glove box for the actual ^{14}C determination.

The handling procedures are very similar to those used at KIT-INE, with the well-known caveat that JRC Karlsruhe hot cells are older and more highly contaminated than those at KIT INE, necessitating yet more care and vigilance in sample handling.

3.3 Glovebox for sample preparation and LSC determination

In all, three types of sample must be foreseen for treatment to effectively collect the ^{14}C in a manner that it can be used for LSC determination of the ^{14}C content.

- Liquid samples from the autoclaves: The ^{14}C in the liquid sample is released in several steps
    - By dropping the solution, taken from the autoclave, into a solution of 8M H_2SO_4, which is simultaneously purged by a N_2 flow (typically 250 ml per minute at a slight (0.2 mbar) under pressure), whereupon inorganic CO/CO_2 is released.

  - Further addition of a strong oxidising agent (potassium peroxodisulphate ($K_2S_2O_8$) with $AgNO_3$ as catalyst) combined with simultaneous heating (95°C) to oxidise all organic carbon into CO/CO_2.
- Gas samples from the autoclaves are taken “as is”. In contrast to KIT-INE where very clean sampling could be achieved and the gas gas sampling vessel was attached to the outside of the glovebox, the “dirtier” conditions of the JRC hot cells restrict handling options so that the gas gas sampling vessel would have had to be inserted in the glove box and linked to the wash stations therein
- Molecular sieve samples from the total C determination using the ICARUS device, which need to be purged of their CO_2 by an N_2 flow with or without gentle heating

The steps followed are drawn entirely by a scheme developed and well documented by KIT-INE [Herm et al 2015; Herm 2015]. A new glove box has been constructed at JRC Karlsruhe according to the technical design in Figure 3.7 with the finished product shown in Figure 3.8.

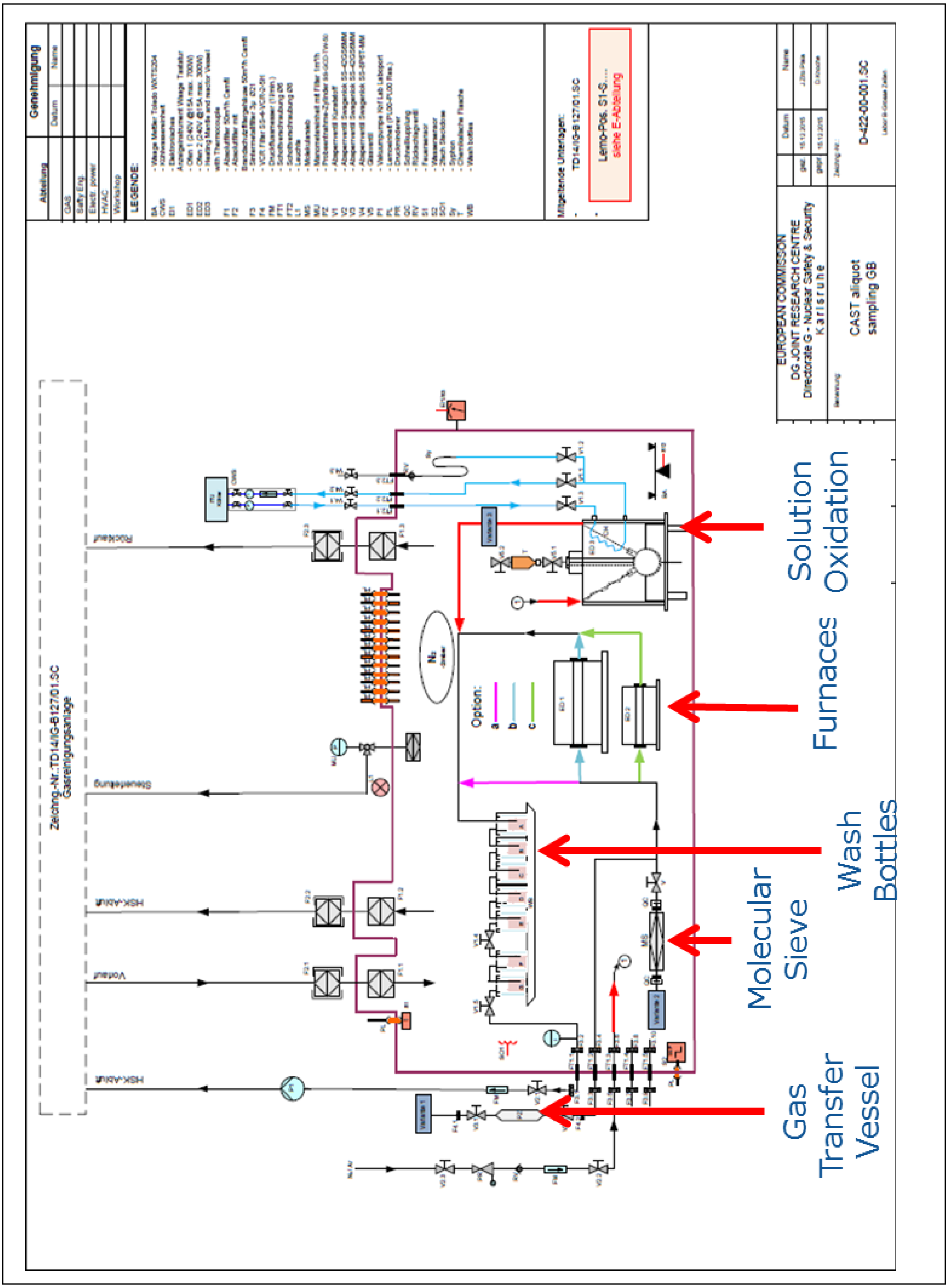


Figure 3.7: Technical design for the ^{14}C preparation glove box

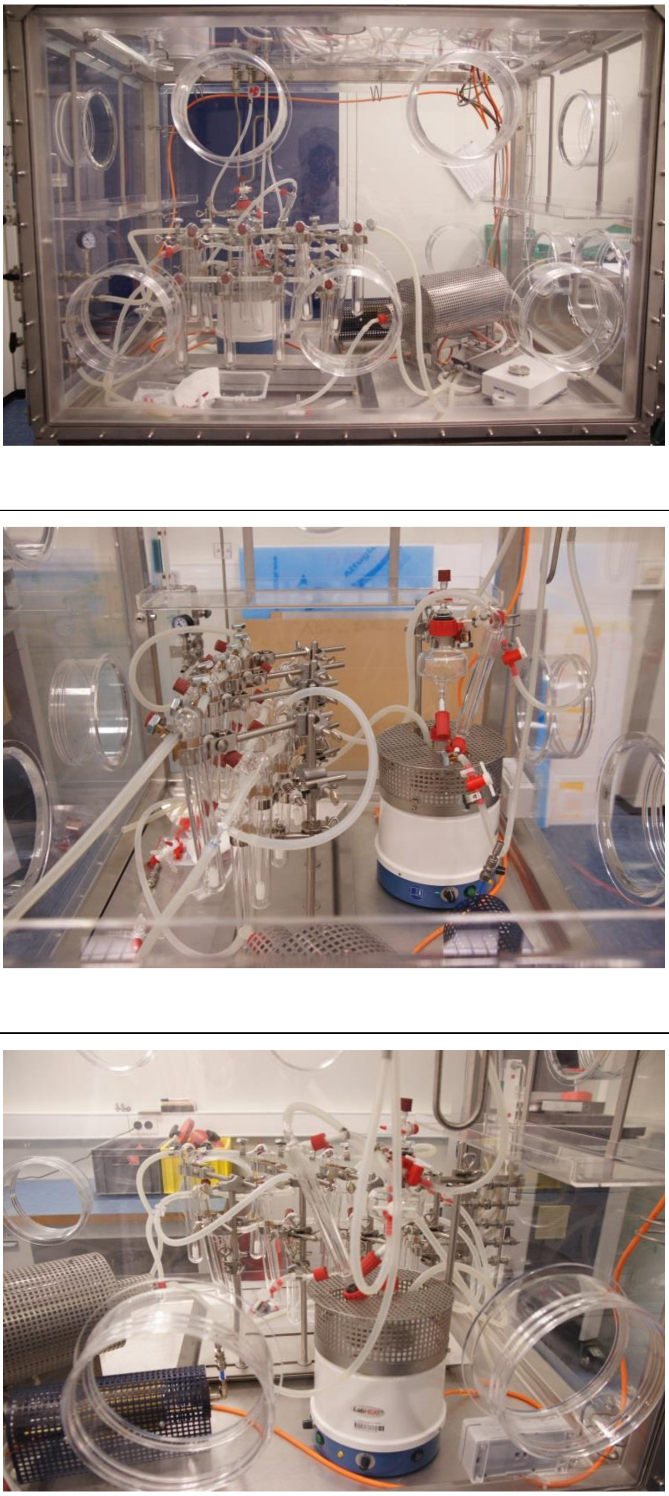


Figure 3.8: Completed Glove box for ^{14}C sample preparation.

The above construction might appear complicated, but at its heart has the following steps. The gas containing CO/ CO_2 mixtures is passed through a variety of wash bottles or furnaces:

- 1st set of Wash bottles
    - Wash Bottle 1: 0.1 M $AgNO_3$ to trap ^{129}I
    - Wash Bottle 2: 5% H_2SO_4 to trap tritium[1]
    - Wash bottle 3: 2M NaOH to trap CO_2
    - Wash bottle 4: 2M NaOH to trap CO_2
- Furnace 1: at 400°C with a CuO catalyst to convert CO to CO_2, which with a by-pass can be bled to second set of wash bottles. (This option was envisaged to quantify CO/ CO_2 ratios in the samples)
- Furnace 2: at 750°C fitted with a CuO / Pt/Al catalyst to convert CH_4 or CO to CO_2
- 2nd set of wash bottles
    - Wash Bottle 5: 5% H_2SO_4 to trap ^3H
    - Wash Bottle 6: 5% H_2SO_4 to trap ^3H
    - Wash bottle 3: 2M NaOH to trap CO_2
    - Wash bottle 4: 2M NaOH to trap CO_2

This rather intriguing separation design (copied in its entirely from KIT INE) opens the possibility to determine ^{14}C as

- Inorganic in the solution from the autoclave
- Inorganic in the gas phase from the autoclave (also with the potential to distinguish CO and CO_2 via two furnaces)
- Organic in the solution from the autoclave
- Organic in the gas phase from the autoclave.

Being ever cautious of the possibility of deleterious cross pollution and contamination of the samples with isotopes detrimental to liquid scintillation counting, an additional precaution was taken and an independent molecular sieve trap is available in the glove box to collect CO_2 without going over the NaOH traps. The molecular sieve chosen is from CARBOSPHERE® carbon molecular sieve (60/80 Mesh). In this way, the ampoule could be switched to an

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[1] ^3H has to be removed before the counting by LSC

independent high purity N_2 gas line to purge the CO_2 captured on the sieve and collect it directly in the liquid scintillation cocktail ready for counting by LSC.

At the end of the process, there are 4 solutions bearing NaOH, from which aliquots (2cc) can be taken and added to 18 cc of the organic fluoride solution used for liquid scintillation counting (LSC). Samples are bagged out and measured in an independent device ((Perkin Elmer Wallac Guardian liquid Scintillation Counter 1414 with Wimspectral software 2.0))

4 Results

Currently, the ICARUS device is ready to be placed in the hot cell. No measurements on irradiated steel samples have been performed yet. Several campaigns on non-irradiated steels have been performed and the results are given in Tables 2.2, 2.3 and 3.1.

In terms of repeatability, the gas extraction method performs well. Such devices lend themselves to optimisation. For example Table 3.1 shows that at the 1 gram sample size, the error is as high as 10% when the total carbon content is 6 ppm. The error decreases from 10% to less than 1% as the carbon content increases. Thus, the operator has the opportunity to increase the sample mass (if sufficient available) when the carbon content is low to improve the accuracy of the total ^{14}C determination.

The operation in a hot cell environment brings additional challenges, which can also influence the accuracy of such measurements. The cleanliness of the crucible is of utmost importance, as any “foreign” carbon can severely impair the results. The cells themselves at JRC Karlsruhe are operated under N_2 which is purged of particulate matter by the double filters of the cell itself. Further precaution includes treatment of the crucibles in oxygen beforehand and their storage inside the cell in sealed containers to ensure no contact with carbon sources therein.

Finally, applying an acid wash to remove fuel from the samples is fully legitimate if the focus is on steel spent fuel hulls from recycling of fast reactor fuels. Nevertheless, it could markedly affect the availability of ^{14}C at the surfaces of the sample. Thus, the reliability of the results with respect to deep geological disposal of structural components of reactors could be impaired. Here, there is a need for innovation to improve the procedure or develop new methods less sensitive to cross pollution than LSC. Cavity ring down spectroscopy (CRDS) [Giusfredi et al., 2010] is a tool being developed for environmental monitoring of ^{14}C (as CO_2) which might bring a step change in ^{14}C analysis, matching the accuracy of accelerator mass spectrometry (AMS) but at a fraction of the cost, and eventually as a mobile measurement station.

5 Conclusions

Considerable effort has been made and progress achieved at the JRC Karlsruhe during the course of the CAST project, as testified above. Nevertheless, the work was stricken by numerous delays and it has not been possible to complete all experiments. The glovebox is ready to be installed at the hot cell building. The autoclaves are ready too. An upgrade at the insistence of the JRC Karlsruhe's licensing authorities had to be made in 2017, rendering insufficient space to install the autoclaves. Thus, final experimental activity must be suspended until 2018. On completion, by the end of 2018, a complete report with the outstanding results will be provided to all partners, and added to the CAST publication website.

Acknowledgements

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Abstract

Within the CAST project, the JRC Karlsruhe (formerly JRC Institute for Transuranium Elements) developed a new device for the determination of total carbon using a gas extraction method. For the steel investigations in WP2, an accuracy of 10 to <1% can be expected for carbon contents in the 5 to 500 ppm range respectively. Further equipment to perform leeching tests and ^{14}C determination by liquid scintillation counting (LSC) has been prepared. Their deployment has been hindered due to technical difficulties and finally due to an upgrade of the cells to meet new legislation. This report summarises the achievements made so far.

This report has been compiled and edited by J. Somers following the retirement of the project leader David Bottomley in February 2017.

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