
THE EC CAST PROJECT

(CARBON-14 SOURCE TERM)

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ABSTRACT

Carbon-14 is a key radionuclide in the assessment of the safety of underground geological disposal facilities for radioactive wastes. It is possible for carbon-14 to be released from waste packages in a variety of chemical forms, both organic and inorganic, and as dissolved or gaseous species. The EC CAST (CARBON-14 SOURCE TERM) project aims to develop understanding of the generation and release of carbon-14 from radioactive waste materials under conditions relevant to packaging and disposal. It focuses on the release of carbon-14 from irradiated metals (steels and zirconium alloys), from irradiated graphite and from spent ion-exchange resins. The CAST consortium brings together 33 partners. CAST commenced in October 2013 and this paper describes progress to March 2015. The main activities during this period were reviews of the current status of knowledge, the identification and acquisition of suitable samples and the design of experiments and analytical procedures.

Key words: carbon-14, steel, Zircaloy, ion-exchange resins, graphite

Introduction

Carbon-14 is a key radionuclide in the assessment of the safety of underground geological disposal facilities (GDFs) for radioactive wastes. Neutron activation of materials containing ^{13}C , ^{14}N or ^{17}O is a major source. ^{14}C has a half-life of 5,730 years, so if it can be retained within the multi-barrier system of a disposal facility for sufficient time it will decay. However, it is possible for carbon-14 to be released from waste packages as a result of dissolution and/or waste degradation processes in a variety of chemical forms. These may be both organic and inorganic, depending on the nature of the waste materials and the chemical conditions. It may also be transported as volatile species in a bulk gas phase (e.g. hydrogen from the corrosion of metal wastes and containers) if the rates of gas generation, combined with the hydrogeology of a disposal facility, are such that a bulk gas phase is able to form and migrate through the near field and geosphere to the biosphere. Carbon-14 is one of the most important contributors to the dose in assessments of the post-closure performance of disposal systems envisaged by many waste management organisations.

The EC CAST (CARBON-14 SOURCE TERM) project aims to develop understanding of the generation and release of carbon-14 from radioactive waste materials under conditions relevant to waste packaging and disposal to GDFs (repositories). The increased understanding provided through CAST should decrease uncertainties in long-term safety assessment and increase confidence in the safety case. The project focuses on the release of carbon-14 as dissolved and gaseous species from irradiated metals (steels and

zirconium alloys), from irradiated graphite and from spent ion-exchange resins. 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: ANDRA, AREVA, ARMINES/SUBATECH, CEA, CNRS/IN2P3, EDF and FNAG (France); Ciemat and ENRESA (Spain); Nagra, PSI and MCM-International (Switzerland); LEI (Lithuania); JRC-ITU (EC); KIT, GRS and FZJ (Germany); SKB (Sweden); COVRA and NRG (Holland); RWMC (Japan), ENEA (Italy), INR and IFIN-HH (Romania); Ondraf/Niras and SCK-CEN (Belgium); IEG NASU (Ukraine); VTT and Fortum Power and Heat (Finland); ÚJV Řež and SÚRAO (Czech Republic) and AMEC and NDA(RWM) (UK).

The objectives of CAST are met through a seven Work Packages (WP). WPs 2, 3, 4 and 5 undertake experimental work on: steels; Zircalloys; spent ion-exchange resins; and graphite, respectively, and will develop conceptual models for the release of carbon-14 from these. WP 6 relates the results to national safety cases, while WP7 disseminates the project's results and their implications to partners and interested stakeholders (CAST is coordinated under WP1 by NDA(RWM) and MCM-International).

CAST commenced in October 2013 and will conclude in March 2018. This paper describes progress to March 2015. The main activities during this period in WPs 2 to 5 have been the preparation of reviews on the current state of knowledge, the identification and acquisition of suitable samples for study and work on the design of experiments and analytical procedures. A review of the treatment of carbon-14 in safety assessments is being prepared under WP6.

Carbon-14 release from irradiated steels (WP2) [1]

WP2 addresses the release and speciation of ^{14}C from activated steel during corrosion under conditions of a cement based deep geologic repository. In many programmes, activated steel represents the major source of ^{14}C . There are twelve partners participating in WP2: AMEC, Armines, Ciemat, ENRESA, JRC, KIT, Nagra, PSI, NRG, RWMC, SCK-CEN and VTT.

The relevant literature on steel corrosion mechanisms and rates, as well as the speciation of carbon (stable isotopes and ^{14}C) in iron and steel after release due to corrosion, has been reviewed [2]. The review gives a comprehensive compilation of corrosion rates under conditions relevant for a cement-based deep repository. In general, the review does not change the view of the likely corrosion rates for carbon steel. In the case of stainless steel, however, a recent paper has provided data for the very low corrosion rates experienced by stainless steels under anoxic alkaline conditions of relevance to long-term post-closure release of ^{14}C from stainless steels. These new data give a mean anaerobic corrosion rate of $0.0008 \mu\text{m yr}^{-1}$ for 18/8 stainless steel at 30°C after 2 years exposure. It was noted that all of the corrosion data compiled for the review were obtained from unirradiated steels and this could have an effect.

The review found much less information on the speciation of ^{14}C released from activated steels. Although the review sought to include all available information on the releases of stable carbon to supplement this, it found that information on the chemical forms of carbon released from steels by corrosion is also limited and the processes involved not well understood. The results of experiments reported in the literature are not conclusive taken together. Some studies on inactive iron-water systems have shown evidence for the release of carbon as hydrocarbons (C1 to C5) in the gas phase. Other experiments focussing on releases to solution have found soluble organic and inorganic species. One study with irradiated stainless steel has reported a small release of ^{14}C (approximately 5 Bq) on leaching in alkaline solution for 42 months, with 25% of ^{14}C in the gas phase and a ratio of about 1:1 organic- ^{14}C to inorganic- ^{14}C in solution. Although the total amount of information found is sparse it can be concluded from the review that a range of aqueous and gaseous ^{14}C -compounds can be expected to be released from the corrosion of irradiated steels.

The development of analytical methods and protocols to measure the release of ^{14}C from activated steel samples and the speciation in the aqueous and gaseous phase started early in the project as this is an extensive task and the methods to be developed are essential for the further experimental work. This task is done in close cooperation with WP3. Experiments with non-irradiated iron samples have been

performed to develop analytical strategies for sampling, separation and quantification of possible ^{14}C species. They are complemented by stability tests of organic molecules in highly alkaline solutions. The focus was put on low molecular weight organic substances. For their separation and identification, High-Performance Ion Exchange Chromatography and Gas Chromatography, both coupled to Mass Spectrometry, are proposed for aqueous and gaseous samples, respectively. It is estimated that in most cases a highly sensitive detection technique such as Accelerator Mass Spectrometry may be needed to detect ^{14}C at the expected extremely low concentrations.

The participants involved in the corrosion experiments to measure the release and speciation of ^{14}C have started to plan and coordinate the experimental design and the conditions. It is foreseen to use irradiated carbon steel and different grade stainless steel samples, but also unirradiated iron and carbide materials. The leaching solution should simulate the expected conditions in a cement based deep repository but will need to avoid the potential precipitation of any ^{14}C released as carbonate. In some experiments, pH and oxygen availability will be varied to cover a range of conditions and to facilitate process understanding.

Carbon-14 release from irradiated Zircalloys (WP3) [3]

WP3 aims to better understand ^{14}C behaviour in waste Zr fuel claddings under disposal conditions with regard to C-14 inventory (and origins), release from waste packages and speciation of released C-14. Ten partners participate in WP3: ANDRA, CEA, Armines, AREVA, EDF, INR, SCK-CEN, KIT, RWMC and JRC.

The start of the art of current understanding of Zircaloy corrosion and carbon-14 release has been reviewed [4]. Inventories of ^{14}C in fuel hulls have usually been determined by calculation, more rarely by direct measurement. Analysis of collected data shows that the power-related ^{14}C production is 1.9 ± 0.4 GBq/GWyr.ppm N although experience suggests that this is likely to be an overestimate. The distribution of ^{14}C between the metal cladding and its external zirconium oxide layer depends on the thickness of the oxide.

The mechanisms and the rate of ^{14}C release from hulls under repository conditions are expected to be controlled largely by the uniform corrosion rate of Zircaloy, the diffusion rate of ^{14}C from zirconium oxide layers and/or the dissolution rate of these layers. Zirconium alloys are highly resistant to uniform corrosion at low or moderate temperatures and unlikely to be susceptible to localized corrosion and stress corrosion cracking in anaerobic groundwaters. However, the physical condition of the hulls, i.e., their state of division and fragmentation may influence this. The bulk Zircaloy of hulls becomes hydrided during reactor operation, which makes the metal brittle and probably fragmented if hulls are compacted during waste processing. Nevertheless, it can be considered that ^{14}C in corroding bulk metal is released congruently. However, the ^{14}C may not be released immediately to solution but can be incorporated into the oxide film and released subsequently by diffusion or during zirconia dissolution. This is supported by the observation that measured ^{14}C specific concentrations in zirconia oxide layers are about twice of that of Zircaloy metal after irradiation in a reactor.

Various studies show that the uniform corrosion rates of zirconium alloys are very low in anaerobic neutral or alkaline waters at low temperature. The most recent results lead to corrosion rates of 1 to 2 nm.y⁻¹ after corrosion for a few years. Such low corrosion rates would lead to decay of much of the inventory of ^{14}C before it could be released from the metal. However, the knowledge on the corrosion resistance of zirconium alloys at low temperature is based on the very beginning of the corrosion regime. Study of the corrosion behaviour of Zircaloy in high temperature water has shown that, when the zirconia oxide layer reaches a critical thickness, there is a change in corrosion kinetics. It is not possible, at low temperature, to corrode samples sufficiently to explore the corrosion regime beyond this point and corrosion tests on pre-oxidized samples are required.

The non-corroded metal of hulls will be gradually transformed into brittle zirconium hydride as it corrodes. This will generate on the external surface of hulls a high-density region of hydrides, acting as a brittle layer, and presumably having a corrosion behaviour different from that of zirconium metal. The

hydrogen pick-up ratio for Zircaloy reaches values of about 90 % in alkaline and in pure water between 30 and 50°C.

The zirconium oxide layer formed on spent fuel rod cladding is very insoluble in pure water (a solubility of 10^{-9} M can be considered as a conservative and realistic estimate). However, the solubility increases with increasing alkalinity, and reaches values of the order of 10^{-6} M at pH 12.5 at ambient temperature. Chloride ions at low to moderate concentrations do not seem to have any significant effect on the zirconia solubility, except in CaCl_2 solutions of concentration higher than 0.05 M at pH > 10 due to the formation of a highly soluble complex with calcium. The dissolution rates of zirconia are less well known and there is a lack of knowledge of whether the release of radionuclides can be considered as congruent with the dissolution of zirconia. Recent Japanese leaching experiments in alkaline solutions at pH 12.5 at room temperature suggest that ^{14}C release would be congruent with the oxide layer dissolution and metal corrosion.

Both organic and inorganic carbon have been identified in leaching experiments with irradiated hulls or non-activated Zr-based materials (Zr and ZrC powders). A higher proportion of the carbon is released as small organic molecules than inorganic molecules but the origin of these compounds and their reaction mechanisms are not fully understood. Significant uncertainties remain with respect to inorganic vs. organic distribution of ^{14}C as well as the identities of the organic ^{14}C species.

Analytical development is proceeding in conjunction with WP2. The main analytical technique to measure ^{14}C inventory is currently envisaged to be Liquid Scintillation Counting (LSC). If this is not sufficiently sensitive, AMS will be used as it has a lower detection limit. Spectroscopic methods such as infra-red spectroscopy will be used to identify the main chemical functions (e.g. carboxylic acids, aromatic compounds). Chromatographic techniques will be used to detect and quantify families of molecules.

The experimental work to be performed on Zircaloy during the CAST Project will involve both non-irradiated and irradiated Zr alloys. The use of NaOH solution as the reference leaching solution is under discussion because the use of a portlandite solution ($\text{Ca}(\text{OH})_2$, pH 12.5) may lead to calcite precipitation preventing optimal ^{14}C measurements in solution. The reference time duration has been set at one year, although a few teams will perform intermediate samplings. One team will perform digestion experiments in acidic media ($\text{H}_2\text{SO}_4/\text{HF}$) to measure total ^{14}C inventory on irradiated Zr4, while the other teams will use the reference solution. Corrosion experiments will be performed on both non-irradiated and irradiated Zr, in the reference solution.

Carbon-14 release from spent ion-exchange resins (WP4) [5]

Seven partners (CEA, EDF, ENEA, FZJ, INR, SKB, ÚJV) are involved in WP4, which is dedicated to the study of speciation, source term, leaching, and gaseous release of ^{14}C from spent ion exchange resins that are widely used in nuclear facilities for the purification of liquid process or waste streams.

A state of the art review on sample choice, analytical techniques and current knowledge of release of ^{14}C from spent ion-exchange resins has been prepared [6]. Most of the ^{14}C determinations deal with the maximum activity determination, percentage of recovery and differentiation between organic and inorganic fractions. There is only limited information about the form of ^{14}C in spent ion-exchange resins and most of the information is related to the partition between inorganic (carbonate) and organic fractions. However, available results on the distribution of ^{14}C between inorganic and organic molecules are contrasting. For spent ion-exchange resins from PWRs around 20% of the ^{14}C was in the form of organic molecules compared to 5% to 20% of ^{14}C for resins from CANDU reactors. In contrast the ^{14}C in two cemented spent ion-exchange resins from a RBMK reactor was found to be more evenly partitioned with about 40 to 50% as organic ^{14}C .

At present no identification of organic molecules in leachates from spent ion-exchange resins has been published. It has been hypothesized that acetate and formate are the possible organic forms of ^{14}C since they are expected in the reactor coolant of PWRs and BWRs. No published investigations on the release of gaseous ^{14}C have been found. In general, inorganic releases in the gas phase are expected to be in the

form of $^{14}\text{CO}_2$. Factors such as changes in solution pH or composition, service/storage temperatures, radiolysis and microbial activity could affect the release of ^{14}C .

Radiolysis could also affect the release and possibly the speciation of other radionuclides. Effects on the speciation of radionuclides due to the degradation of mixed bed ion-exchange resins have been reported with releases of trimethylamine (TMA), $\text{H}_2(\text{g})$ and $\text{CO}_2(\text{g})$ from anion exchange resins due to γ -irradiation. A distinction between anaerobic and aerobic conditions was made with $\text{CO}_2(\text{g})$ detected only in the presence of oxygen. Studies on radiolytic and chemical degradation of strong acidic ion exchange resins found mainly sulphate and dissolved organic carbon in solution. Among these, only oxalate was identified, comprising 10-20% of the organic fraction. Other degradation products were CO_2 and H_2 .

In the experimental studies most of the partners will study spent ion exchange resins from pressurised water reactors, including CANDU reactors, and two partners (FZJ, SKB) will study resins taken from boiling water reactors. Analytical strategies for speciation determination by the partners are also described in the review.

Carbon-14 release from irradiated graphite (WP5) [7]

WP5 considers irradiated graphite and related ^{14}C behaviour with the objective of understanding the factors determining release of ^{14}C from irradiated graphite under disposal conditions. This will be achieved through:

- Determining the ^{14}C inventory and concentration distribution in irradiated graphites, and factors that may control these;
- Measuring the rate and speciation of ^{14}C release to solution and gas from irradiated graphites in contact with aqueous solutions; and
- Determining the impact of selected waste treatment options on ^{14}C releases and relating this to the nature of ^{14}C in irradiated graphite.

Thirteen organisations participate in WP5: NDA, ANDRA, INR, ENEA, FZJ, ENRESA, LEI, IEG-NASU, FNAG, IFIN-HH, CNRS, Ciemat and EDF.

The current understanding of irradiated graphite and carbon-14 release has been reviewed on an organisation basis [8]. An important part of this review was to capture relevant knowledge from the EC CARBOWASTE project [9]. Experimental measurements of the ^{14}C inventory in RBMK-1500 reactor graphite were performed within CARBOWASTE and a short leaching test for RBMK graphite was also performed at the end of CARBOWASTE. An independent programme of graphite sampling from the graphite stack of Unit 1 was developed by Ignalina Nuclear Power Plant staff, and sampling was accomplished at the end of 2013 year. This work also included some leaching experiments for several radionuclides.

Characterisation data on the speciation of ^{14}C in MTR and HTR i graphites and relevant information from CarboDISP, decommissioning projects in Germany and the ASSE test repository were summarised in the review.

Graphite samples from MTR reactors (JEN-1 (Spain) and TRIGA (Romania)), UNGG reactors (Vandellos-1 (Spain) and SLA-2 (France)) and a UK Magnox reactor have been studied in Spain using a range of characterization techniques.

Thermal annealing of graphite in an inert atmosphere does not induce any migration of artificially implanted ^{13}C up to 1600°C , even if the structure of the graphite is initially disordered; at 1600°C slight diffusion occurs. Irradiation with Ar^{3+} induces a strong re-ordering of the graphite even at room temperature, compared to He^{2+} irradiation or thermal annealing alone. Extrapolation of these results to ^{14}C and the ^{14}N precursor suggested that:

- ^{14}N tends to migrate to the free surfaces where it is partially released under the effect of temperature (500°C). Therefore, most of the ^{14}C formed by activation of the remaining ^{14}N might be located close to free surfaces (open pores).

- The sole influence of heat at UNGG reactor temperatures (200 – 500°C) did not promote ^{14}C release but both ^{14}N and ^{14}C would be released through radiolytic corrosion when located close to free surfaces.

- The results strengthen conclusions arguing that the remaining ^{14}C inventory in French irradiated graphites has been mainly produced through the activation of ^{13}C .

Data on ^{14}C release and speciation from French irradiated graphites has been reviewed. The main conclusions are that the ^{14}C leaching rate is very slow for the stack graphite, and in most cases a quasi-steady state leach rate appears to be achieved after around 100 to 200 days. Over that period, the calculated mean radiocarbon leach rate lies between 10^{-11} and 10^{-8} m.day $^{-1}$. A faster carbon-14 leach rate seems to be observed for the sleeve graphite (operational waste), which represents a small part of the total inventory of ^{14}C . The pH of the leaching liquid (deionised or alkaline water) does not seem to have any clear impact on the ^{14}C leaching behaviour.

UK review and experimental studies have found that, in general, only a small fraction of the total carbon-14 inventory (up to ~1%) is released on leaching in solution over timescales of up to 3 to 4 years. The majority of release occurs to the solution phase; small amounts of gaseous phase releases have been measured. Leaching studies show an initial fast release followed by an approach to a steady state with a very low incremental release rate. Crushing may increase the accessibility of carbon-14 to water but volatile carbon-14 may be lost during crushing. Even when harsh acidic conditions are applied, <30% of the carbon-14 inventory is released over experimental timescales. This points to the likelihood that there are two main forms of carbon-14 in irradiated graphite: leachable (with the leachability depending on accessibility to leachant) and non-leachable (probably part of graphite matrix). There is evidence that ^{14}C is released to the solution phase in organic as well as inorganic ($^{14}\text{CO}_2$ /carbonate) forms under alkaline conditions and that carbon-14 is released to the gas phase. Gas phase releases include both volatile ^{14}C hydrocarbons (probably $^{14}\text{CH}_4$) and ^{14}CO (or possibly $^{14}\text{C}_x\text{H}_y\text{O}_z$). $^{14}\text{CO}_2$ is also released from solution at near-neutral pH. The form of gaseous ^{14}C release is affected by redox conditions with a lower redox potential seeming to favour ^{14}C -hydrocarbons.

There is evidence that most carbon-14 in irradiated graphite arises from the activation of carbon-13 (1.07% natural abundance) and is bound covalently in the graphite homogeneously throughout the matrix. Thus the removal of this carbon-14 could only occur by oxidation with conversion to either ^{14}CO or $^{14}\text{CO}_2$. However, the graphite matrix is extremely resistant to oxidation at temperatures expected in a geological disposal facility and is also unlikely to oxidise because of the anaerobic conditions that will develop after closure. Kinetic and mechanistic studies undertaken to understand the corrosion behaviour of graphite under disposal conditions, as well as the natural analogues of graphite deposits in nature, show that the graphite has an extremely long life time. Significant graphite corrosion requires high dose rates, which would not be expected from i-graphite, not even spent HTR fuel. However, there is also a heterogeneously distributed ^{14}C portion that is enriched in hotspots and on surfaces and may arise from the activation of ^{14}N . This may be the source of the ^{14}C detected in leaching experiments under simulated disposal conditions.

The experimental work in WP5 consists of several leaching studies of irradiated graphites. These include a comparison of samples that have undergone decontamination treatments to untreated samples to confirm a labile ^{14}C fraction, rather than slow diffusion of ^{14}C from within the graphite. Other leaching experiments will study release from the TRIGA 14MW reactor thermal column, the thermal column of VVR-S Reactor, the Vandellós I reactor and from a graphite glass coated waste form (IGM, Impermeable Graphite Matrix). Irradiated graphite from the Latina power plant has also been exfoliated to extract ^{14}C intercalated between the graphene layers. Three different organic solvents with good solvency properties and water-miscibility are being tested, initially on non-irradiated virgin graphite and then on samples from the Latina moderator.

Carbon-14 in performance assessments (WP6)

The results from WPs 2 to 5 will be evaluated in the context of national safety assessments in WP6. There are fourteen partners in WP6: NDA, Nagra, ANDRA, ONDRAF/NIRAS, INR, GRS, ENEA, RWMC, ENRESA, Fortum, LEI, NRG, SURAO and SKB. A review of the treatment of ^{14}C in safety assessments has been prepared by the participants in WP6 and is being reviewed and finalised prior to publication (expected in June 2015). An important point is to identify whether ^{14}C migration as gas or in solution is most important for individual concepts and safety cases. For some concepts gaseous transport is not an important priority.

Dissemination (WP7)

The results from the CAST project will be directly applicable to organisations that either evaluate or make safety cases for the geological disposal of radioactive wastes containing ^{14}C and will be disseminated to a wide audience. The main outputs from CAST will include final reports from each Work Package, along with an overall overview report summarising the results and achievements of the whole project. COVRA, KIT, JRC and LEI participate in WP7. The CAST website (<http://www.projectcast.eu/>) has been established to enable reports from CAST to be readily accessed and downloaded. Events, such as workshops and courses, will also be publicised on this website. A CAST newsletter [10] is published on the website on a periodic basis.

Two workshops will be held as part of the project. The first aims to outline the initial findings from the research and allow interested parties to familiarise themselves with the proposed work and ask questions of the Work Package teams. This will provide interested parties with an opportunity for early communication and engagement. The second workshop will present the overall results and findings of CAST and discuss these with target groups. Two training courses will also be provided: the first organised by KIT to provide experience in radioanalytical techniques and sample handling. The second will be run by COVRA to provide understanding of carbon-14 generation and wastes, and experience in transport modelling. There is also opportunity for early-career researchers to exchange with other partners within the project.

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