

## Advances on Containment Iodine Chemistry

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### Summary

The 5<sup>th</sup> FWP EURSAFE project highlighted iodine chemistry in the containment as one of the issues requiring further research in order to reduce source term uncertainties. Consequently, a series of studies was launched in the 6<sup>th</sup> FWP SARNET project aimed at improving the predictability of iodine behaviour during severe accidents via a better understanding of the complex chemical phenomena in the containment. In particular, SARNET has striven to foster common interpretation of integral and separate effect test data; production of new or improved models where necessary needed, and compilation of the existing knowledge of the subject. The work has been based on a substantial amount of experimental information made available from bench-scale projects (PARIS and EPICUR), via intermediate-scale tests (CAIMAN) to large scale facilities (SISYPHE, THAI and PHEBUS-FP).

In the experimental field, particular attention has been paid to two specific issues: the effects of radiation on both aqueous and gaseous iodine chemistry, and the mass transfer of iodine between aqueous and gaseous phases. Comparisons between calculations and results of the EPICUR and CAIMAN experiments suggest that the aqueous phase chemistry is reasonably well understood, although there are still some areas of uncertainty. Interpretation of integral experiments, like PHEBUS-FPT2, indicated that radiation-induced conversion of molecular iodine into particulate species ( $I_xO_y$ ) could be responsible for the gaseous iodine depletion observed in the long-term. However, the results of much simpler, small-scale experiments have shown that further improvements in understanding and modelling are still needed. These data will be further supplemented by those obtained in the forthcoming NROI project. Mass transfer modelling has been extended to cover evaporating sump conditions based on SISYPHE data; however, application of this model to the larger scale THAI experiments seems not to be straightforward. In addition to these two major issues, some specific studies have been carried out concerning the potential effect of passive autocatalytic hydrogen recombiners on iodine volatility. The RECI analytical experiments have shown that metal iodides (namely CsI and CdI<sub>2</sub>) are not stable and yield gaseous iodine when heated, in a humid atmosphere, at temperatures representative of recombiner operation.

Another important undertaking successfully carried out has been the compilation of an Iodine Data Book, which provides a critical review of the experimental data and modelling approaches that have been used in the development of iodine source term methodologies. This should assist in a proper use of such models, and inform their future development.

Finally, it should be emphasized that an important outcome of this iodine group in SARNET has been the establishment of links with other related projects, such as Phebus FP, ISTP, ISTC-EVAN, OECD-BIP and THAI. Collaborative studies based around data from

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these projects will facilitate the progressive resolution of the remaining issues in a potential follow-up programme.

### A INTRODUCTION

The 5<sup>th</sup> FWP EURSAFE project highlighted iodine chemistry in the containment as one of the issues requiring further research in order to reduce source term. A series of studies was therefore undertaken within SARNET aimed at gaining a better understanding of the complex phenomena and hence improving the predictability of iodine behaviour within the reactor containment. The work has largely been centred upon a number of small- and intermediate-scale experimental programmes relating to different phenomena: radiolytic oxidation of iodine in solution and gaseous phases, mass transfer of volatile iodine between solution and gas, and decomposition of iodide aerosols in passive autocatalytic recombiners. Interpretation of the large-scale Phebus tests has also formed part of this work, but this was discussed in the previous ERMSAR meeting [1] so will not be considered further in this paper.

The iodine chemistry work in SARNET has been organised into a number of specialised circles, each focussed on a particular issue or set of experiments. The achievements of each of these circles are described in the following sections.

### B RADIOLYTIC OXIDATION OF IODINE (ROX)

Radiolytic oxidation of iodine is fundamental to the behaviour of radioiodine in the containment following a reactor accident. The rate, extent and products of radiolytic oxidation determine the volatility of iodine within the containment and hence its potential release to the environment via containment leakage or breach. The changes in iodine speciation that can occur as a result of radiolytic reactions, both in the aqueous and gaseous phase, also influence the extent to which iodine can react with surfaces, which in turn can result in both iodine retention and the formation of volatile organic iodide. Furthermore, radiolytic oxidation in the gas phase results in the transformation of gaseous species to solid aerosol particles, with implications for settling and release characteristics.

Radiolytic oxidation of iodine in solution had been extensively studied before the start of SARNET, and the main mechanisms and sensitivities were generally well understood. There were, however, some differences in the ability of codes to model experimental results, and the available data were rather sparse for some relevant conditions (high temperature, high pH, and high dose rate). Moreover, the significance of other uncertainties (e.g. impurity effects) was not known. Gas-phase reactions had been studied to a lesser extent, and although various models had been developed, some fundamental aspects remained poorly understood.

#### B.1 Radiolytic oxidation in solution

New experimental data on iodine radiolysis in aqueous solution have mainly been provided by the EPICUR tests carried out by IRSN at CE Cadarache []. In these tests, a radiolabelled iodide solution contained within a stainless steel vessel is simultaneously heated and irradiated with a Co-60 source. Volatile species produced in the solution are removed by a sparging gas which transfers the volatile iodine to a series of species-specific traps. The amount of iodine transferred to the traps is monitored constantly by gamma counting of the radioactive tracer, giving a continuous measurement of the iodine concentration in the gas phase. A schematic diagram and photograph of the facility are shown in Figure 1.

Sixteen iodine irradiation tests have been performed in the EPICUR facility over the duration of the SARNET programme. Most of these have used a weakly acidic iodine solution ( $\text{pH}_{25^\circ\text{C}} = 5$ ) although two tests have been performed at  $\text{pH}_{25^\circ\text{C}} = 7$  to test models of the effect of pH on volatility. All the tests have been carried out at high temperature (80 or 120°C) with a  $\gamma$  dose rate of 2 – 3 kGy hr<sup>-1</sup>. In some tests a painted coupon was immersed in the liquid or suspended in the gas phase to act as a source of volatile organic iodide. The results of a typical

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test, showing the increasing activity of iodine on the inorganic and organic iodide traps, are shown in Figure 2.

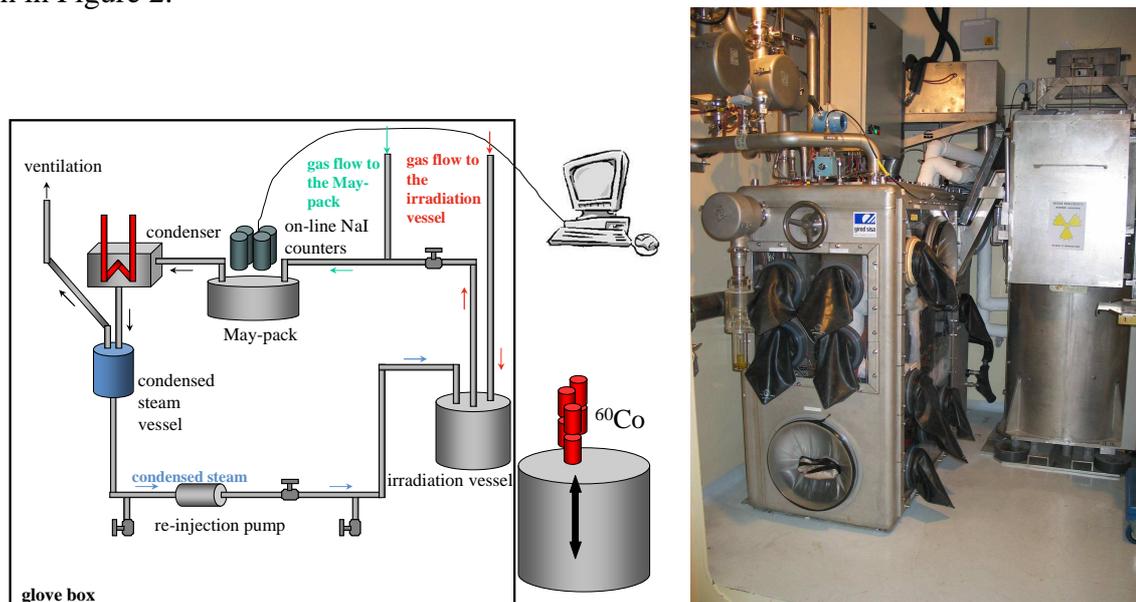


Figure 1. Schematic diagram and photograph of the EPICUR facility

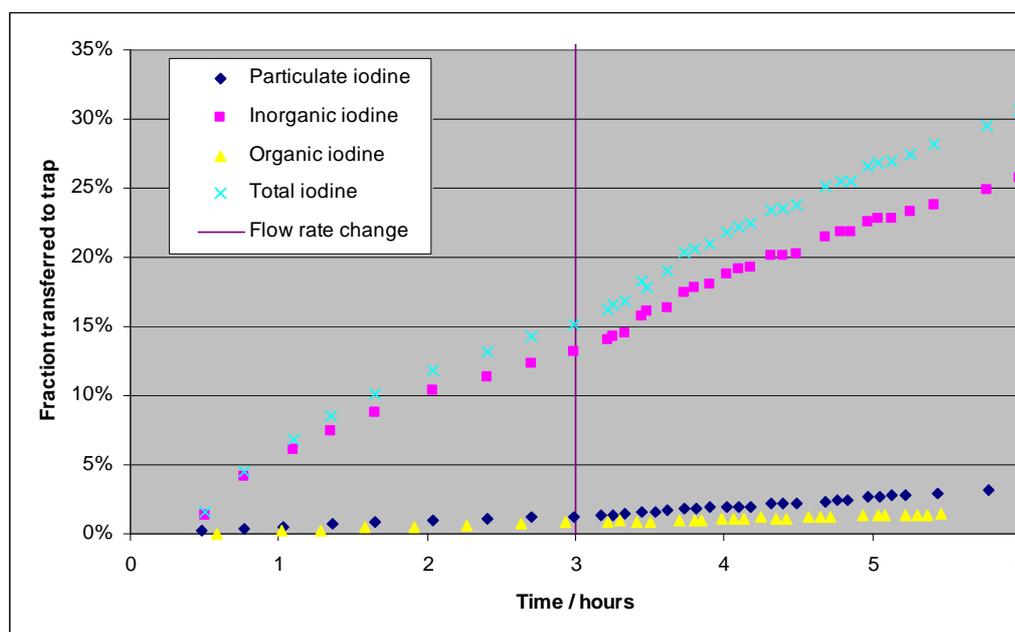


Figure 2. Typical EPICUR test results

Model simulations of the EPICUR tests have been made using the COCOSYS-AIM [2], ASTEC-IODE [3], INSPECT [4] and LIRIC [5] codes. The empirical models (AIM and ASTEC-IODE) were able to reproduce most of the experimental observations acceptably well for the tests at pH 5. These models were not able to simulate the effect of changing the sparge gas from argon to air, which is to be expected since this is thought to result from the detailed radiation chemistry of dissolved oxygen. However, they did confirm that the higher iodine volatility observed in argon was unlikely to be caused by physical effects. The results of the tests at pH 7 revealed significant uncertainties in the radiolysis models at higher pH, and

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additional work will be required to define the most appropriate set of rate constants to capture the high pH data.

The mechanistic models (INSPECT and LIRIC) were also able to simulate the results of the tests reasonably well. These calculations showed that uncertainties in the rate of iodine adsorption – desorption on the vessel surfaces can have a significant effect on the predicted iodine release, and it is difficult to separate such effects from other potential uncertainties. For example, boric acid has been shown to have a catalytic effect on certain key radiolytic reactions, but this has not been quantified at high temperatures.

More limited interpretation studies have been performed around the CAIMAN 2005 SA test, which was performed under similar chemical conditions to the EPICUR tests at low pH but with a different experimental geometry. Again the model simulations give reasonable agreement with the experimental results, taking account of the effect of surface reactions.

#### **B.2 Radiolytic oxidation in the gas phase**

The PARIS programme has provided new experimental data on the radiolytic oxidation of iodine in the gas phase [6]. In these tests, various gas mixtures (air/steam, air/steam/H<sub>2</sub> and O<sub>2</sub>/steam) containing very low concentrations of I<sub>2</sub> were irradiated and the conversion of I<sub>2</sub> to oxidised products was measured. A major objective of the test programme was to extend the measurement of radiolytic destruction rates to the low concentrations of gaseous iodine expected in containment, since earlier tests with higher I<sub>2</sub> concentrations indicated that the destruction reaction was zero-order [7]. If the same kinetic behaviour was found at lower concentrations, as indicated by mechanistic modelling, this would imply a very short lifetime of I<sub>2</sub> in the gas phase. The PARIS tests were performed with initial I<sub>2</sub> concentrations in the range 10<sup>-9</sup> – 10<sup>-6</sup> mol dm<sup>-3</sup>. Different surfaces have the potential to influence the radiolytic decomposition of I<sub>2</sub>, either by directly influencing the radiation chemistry in the gas phase or providing a competitive reaction route in surface deposition. This was studied in tests with steel, silver or painted coupons in the irradiation vessel. All of the tests were performed at 80 or 130°C and dose rates of about 1 kGy hr<sup>-1</sup>.

The I<sub>2</sub> oxidation rates measured in the PARIS tests were an order of magnitude lower than in the earlier tests. Although there was still a tendency towards higher decomposition rates at lower concentrations, taken as a whole the results are not consistent with simple zero-order kinetics as previously thought. The measured oxidation rates are much slower than predicted from mechanistic modelling, and the reasons for this discrepancy are not at all understood at the present time. Further experiments are planned within the SARNET-2 programme; these will use a new facility (EXSI) and measure the production of solid oxide aerosol when I<sub>2</sub> is exposed to UV irradiation. However, these tests will not provide new kinetic information or elucidation of the reaction mechanisms.

### **C SUMP – ATMOSPHERE MASS TRANSFER (MAT AND THAI)**

Mass transfer between the sump and the atmosphere is an important aspect of iodine behaviour in the containment since it affects both exchange kinetics and equilibrium concentrations. SARNET work in this area has had two objectives: validation of mass transfer models currently used in iodine chemistry codes against large scale tests, and extension of sump-atmosphere mass transfer models to evaporating conditions. The former has been addressed through a code benchmark based on simulation of the THAI test Iod-9. The latter has been met by developing an extended model and validating it, as far as possible, against the SISYPHE database.

#### **C.1 Benchmark on THAI test Iod-9**

Test Iod-9 was carried out 2003 at the large-scale THAI test facility (THAI = Thermal hydraulics, Hydrogen, Aerosols, Iodine) of Becker Technologies in Eschborn [8]. The main

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processes addressed in Iod-9 are (1) mass transfer of molecular iodine ( $I_2$ ) from the gas into the sump, (2) iodine transport in the stratified and mixed sump, and (3)  $I_2$  adsorption onto and desorption from the vessel walls with and without wall condensation.

The 60 m<sup>3</sup> THAI vessel is 9.2 m high, 3.2 m in diameter and made of stainless steel. In the configuration for Iod-9 it has two sumps, the main sump at the bottom and the flat so-called elevated sump at 4 m (Figure 3). Iod-9 has four test phases. In the conditioning phase the steel vessel was heated up to approximately 90 °C. The real test started with the injection of 0.53 g  $I_2$  labelled with radioactive I-123. The released  $I_2$  was distributed within the vessel atmosphere by convective flows, deposited onto the vessel walls and some  $I_2$  was transferred into the sumps. During phase A1 ( $t = 0$  to 4 h) the main sump was thermally stratified, with a temperature between 59 °C at the bottom and 66 °C near the water table. In phase A2 ( $t = 4$  to 24.45 h) the main sump was mechanically mixed. In phase B ( $t = 24.45$  to 32.57 h) steam was injected. Iodine adsorbed during wall condensation was washed down into four drain channels together with the dry adsorbed iodine which was deposited earlier. From the channels the condensate was conducted outside into tanks for measurement.

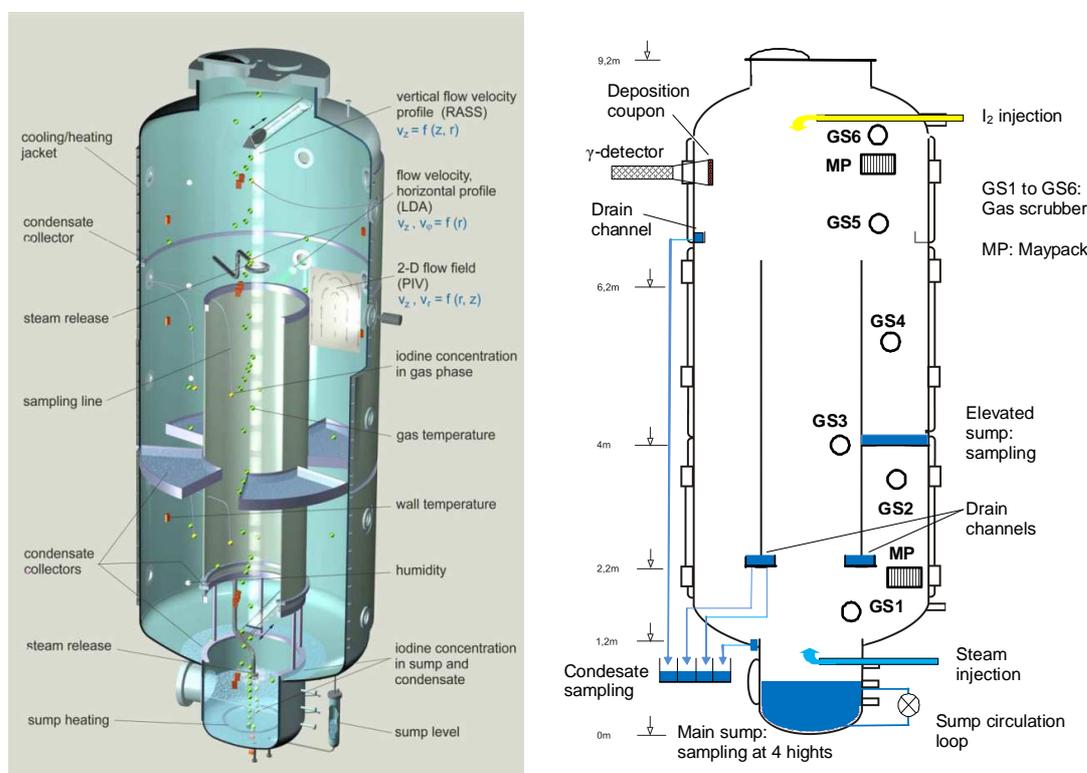
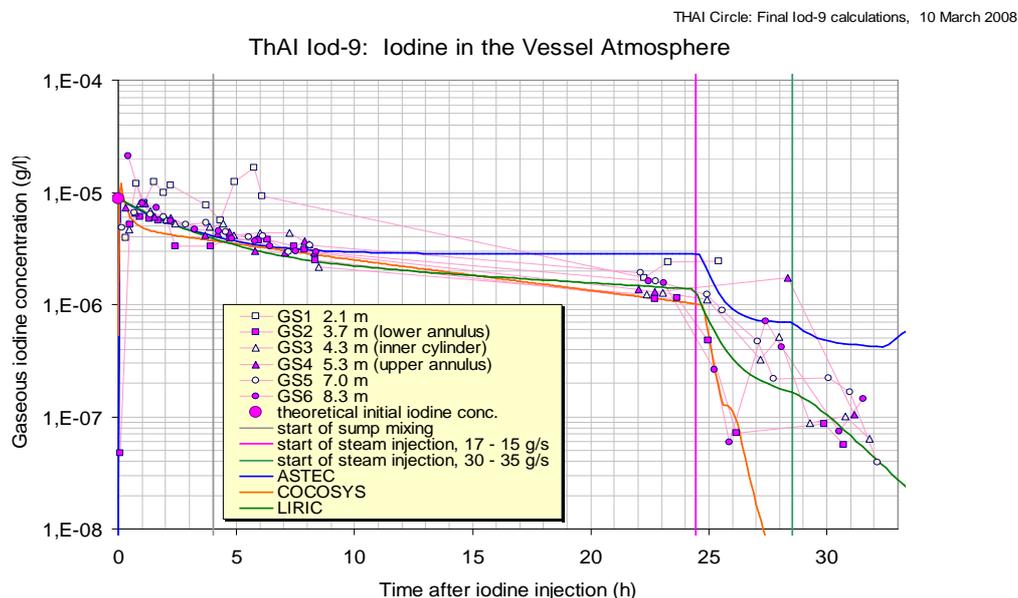


Figure 3. THAI vessel in the Iod-9 configuration

The codes applied by the partners were ASTEC-IODE (IRSN) [3], COCOSYS-AIM (GRS) [2] and LIRIC (AECL) [5]. IODE and AIM are semi-empirical iodine models which are integrated in the lumped-parameter codes ASTEC and COCOSYS. With both codes multi-compartment iodine calculations can be performed. The more detailed mechanistic iodine model LIRIC is for single compartment stand-alone calculations. Each THAI Circle partner made two calculations, the first without knowledge of the other partners' results, and the second after intensive discussion of the first results during a dedicated workshop. Only the final results are described here. Four special issues which came up during this work were treated in more detail:  $I_2$  diffusion in the sump,  $I_2$ /steel reactions, gas/water mass transfer, and CFX-calculation on the  $I_2$  transport in the stagnant sump.

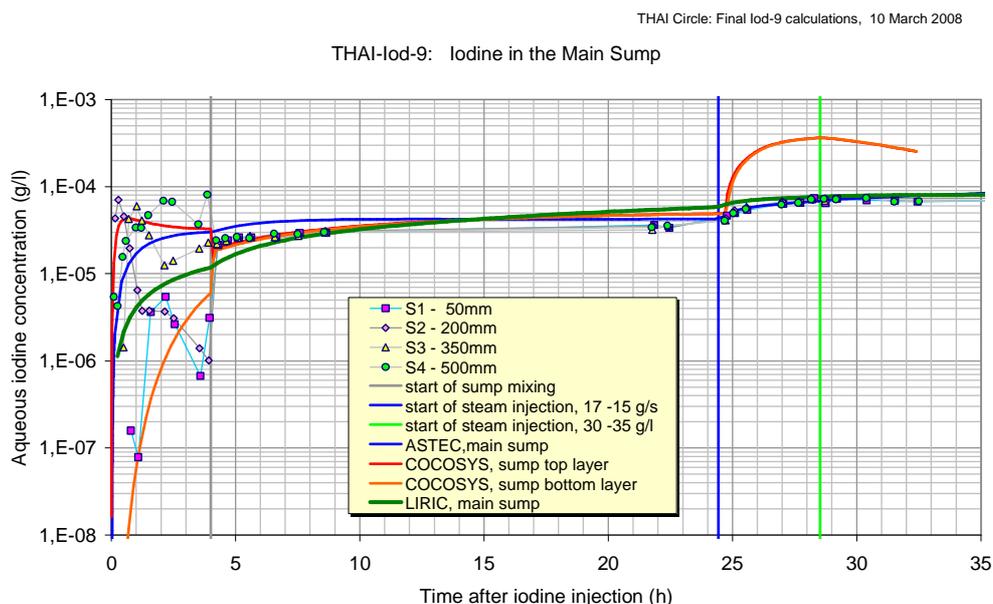
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Figure 4 shows the measured and calculated gaseous  $I_2$  concentrations. In phases A1 and A2 (0 – 24 h) the concentration was reduced due to dry  $I_2$  adsorption on the steel walls and mass transfer into the sumps. In the washing phase B the remaining gaseous  $I_2$  was mainly washed onto the walls.



**Figure 4: Gaseous iodine in the vessel**

Figure 5 shows the evolution of the measured and calculated iodine concentration in the main sump. In the stagnant phase A1 the iodine concentration is strongly inhomogeneous. With the activation of the re-circulation loop in phase A2 the sump was quickly homogenized. The aqueous iodine concentration rose slightly because equilibrium with the gaseous iodine was not yet reached. Most iodine washed down in phase B was drained into the external tanks, but some was washed into the main sump increasing the concentration slightly.



**Figure 5: Iodine in the main sump**

The iodine behaviour in THAI test Iod-9 is fairly well simulated by the three codes. The measured iodine behaviour is well understood and all measured data are found to be consistent. The very slow iodine transport within the stratified main sump was simulated with COCOSYS only.

This work highlighted the need to improve modelling of (1) iodine washdown from the walls, (2) I<sub>2</sub> mass transfer between gas and sump, and (3) the I<sub>2</sub>/steel reaction in the gas and water phases. New washdown data are expected from planned OECD THAI tests. A further validation of the mass transfer correlations would require new data from large-scale tests. For the I<sub>2</sub>/steel reaction simplified models which are already validated exist. Detailed models suggested need further experimental studies.

The analyses of the large-scale iodine test Iod-9 are an important validation step for the codes applied. Two more THAI tests, Iod-11 and Iod-12, will be analysed in SARNET-2. In these tests the distribution of I<sub>2</sub> in a 5-room geometry under severe accident typical conditions were measured.

## C.2 Evaporative liquid-gas exchange model

The existing data base on sump-to-atmosphere iodine mass transfer in containment under evaporating conditions is scarce and, generally, not representative. As a result, the models have not been fully validated under these conditions. Most of existing models in codes are based on the two-film theory [9], which does not consider phase changes at the gas-liquid interface. As a consequence, their accuracy is not satisfactory under conditions other than purely convective ones. An extension of the two-film model was developed and compared to available data at the beginning of SARNET [10]; however, the parametric approach of this adaptation left room for developing a more mechanistically based model.

A semi-mechanistic model has been developed based on: the two-film model, the heat-mass transfer analogy and the surface renewal surface theory. Two mechanisms have been accounted for as sump-atmosphere carriers: diffusion and “steaming” (i.e., entrainment by steam evaporating at the liquid-gas interface). The effect of evaporation on the liquid side of boundary layer has been neglected, so that the iodine mass flow rate once the gas and liquid mass flow rates at the interface have been balanced results in:

$$\varphi = A_{\text{int}} \cdot \left( \frac{k_g \cdot k_\ell}{k_g + H \cdot k_\ell + \frac{\dot{m}_{\text{ev}}}{A_{\text{int}} \cdot \rho_{\text{sat}}(T_\ell)}} \right) \cdot \left( H \cdot C_g - \left( 1 + \frac{\dot{m}_{\text{ev}}}{A_{\text{int}} \cdot \rho_{\text{sat}}(T_\ell) \cdot k_g} \right) \cdot C_\ell \right)$$

The iodine concentrations ratio defining the equilibrium condition ( $\varphi=0$ ) then becomes:

$$\frac{C_\ell}{C_g} = \frac{H}{\left( 1 + \frac{\dot{m}_{\text{ev}}}{A_{\text{int}} \cdot \rho_{\text{sat}}(T_\ell) \cdot k_g} \right)}$$

It may be noted that in the absence of evaporation equilibrium is characterized by the iodine partition coefficient, whereas under evaporative conditions the concentration ratio decreases.

The mass transfer coefficients are important unknowns in the equations above. To estimate the gas mass transfer coefficient the heat-mass transfer analogy has been applied under natural convection conditions [11]:

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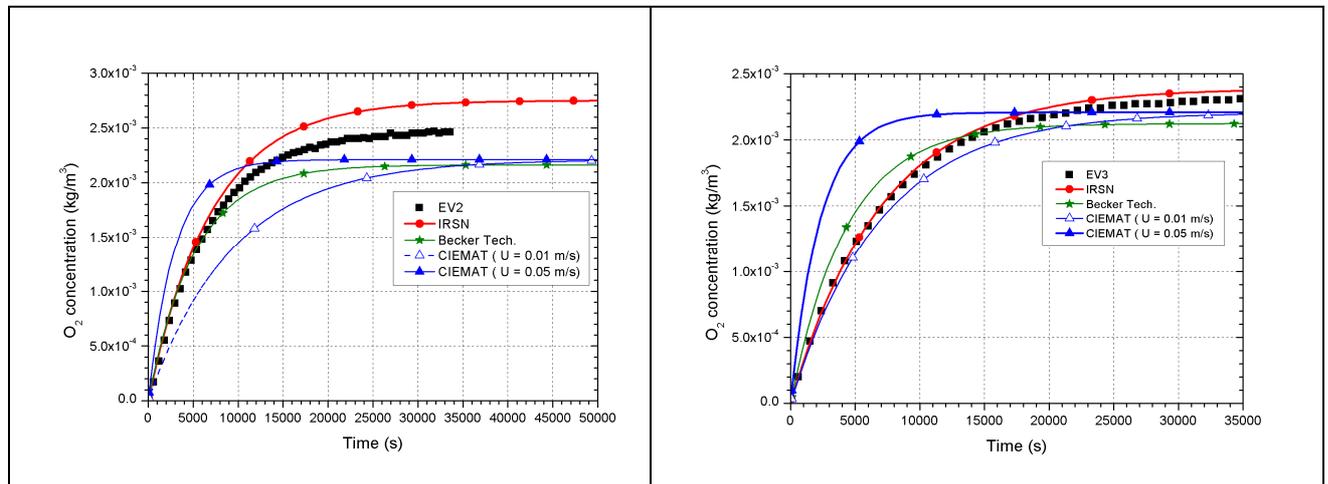
$$k_{g,12} = 0.15 \cdot D_{g,12}^{2/3} \cdot \left( \frac{g \cdot \Delta \rho}{\mu} \right)_{H_2O_v}^{1/3}$$

For the liquid side mass transfer, the surface renewal theory has been used to derive a correlation adapted to the anticipated conditions in containment [12]:

$$k_{\ell,12} = Sc_{\ell,12}^{-1/2} \left[ 1.48 \cdot 10^{-4} + 4.8 \cdot 10^{-4} \left( \frac{U}{0.029} \right)^{1.015} \right]$$

This equation assumes that the liquid volume is well mixed. Under non-well-mixed conditions the first term in the brackets should be  $2.9 \cdot 10^{-5} \text{ m} \cdot \text{s}^{-1}$ .

The model has been compared to a set of data from the SISYPHE program [10] in which boundary conditions were representative of post-accident conditions in containment (Figure 6). The model behaved rather accurately yielding errors between 9 and 25% in the long-term aqueous concentration of oxygen (used as an iodine substitute) and slopes with a noticeable qualitative consistency with data. Such accuracy is considered very satisfactory for a model which does not rely on any empirical parameters.



**Figure 6. Aqueous oxygen concentration in the EV2 and EV3 SISYPHE experiments**

Compared with the model used in the ASTEC code at present, the so-called CIEMAT model presents three major advantages: it is not fitted to any specific data set; although precise, estimates are always conservative; and, no less important, its bases, as well as hypotheses, are thoroughly documented. Nonetheless, the model is far from having been submitted to an extensive validation. Available data are scarce, but as soon as further representative data become available model validation should be continued, preferably against data from large scale experiments, as highlighted above.

## D IODINE IN PASSIVE AUTOCATALYTIC RECOMBINERS (IPAR)

### D.1 RECI program outcomes and prospects

Investigations into the potential impact of Passive Autocatalytic Recombiners (PARs) on iodine chemistry in the containment were prompted by the well known thermal instability of metal iodides in a mixture of air and water vapour. Due to their high surface/volume ratio, micron-sized iodide particles circulating between the catalytic plates of a recombiner in

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operation are likely to be melted and partially vaporized, facilitating the thermochemical equilibration of the iodide vapour with the carrier gas. Both THERMODATA calculations and RECI (RECombiners and Iodine) experiments showed that production of gaseous iodine  $I_2$  is significant, even for the most stable of iodides (CsI), at the operating temperature of recombiners ( $> 500^\circ\text{C}$ ). Thermal decomposition of iodide aerosol by recombiners thus has a potential impact on the iodine source term, but the findings of RECI experiments need to be confirmed by integral tests, with more representative conditions.

Some preliminary integral tests have been carried out outside the scope of the 6<sup>th</sup> FWP SARNET Project in the ThAI facility (AER-2 and AER-5) [13], and it was concluded that further investigations are required beyond these technical scale experiments. A more comprehensive integral test is planned in 2009 within the ThAI OECD Project.

The need for new analytical experiments (RECI+) at the bench scale, with variation of more parameters than the already completed RECI program, is also being considered.

#### **D.2 Technical advances.**

The results of the RECI program were analysed at IRSN using the ASTEC/Sophaeros code, showing in particular that the rapid cooling of the gaseous mixture in the RECI experiments quenches the chemical composition of the effluent. This leads to a higher concentration of volatile iodine compared to what would be expected in the more gradual temperature gradient of a recombiner "chimney". Similar results were obtained at Demokritos from a multidimensional analysis performed with an in-house developed computational fluid-particle dynamics code (see Figure 7). Both studies point towards the main departure of RECI experiments from the representative thermohydraulic regime of an actual recombiner. The results will be presented at the 2008 European Aerosol Conference in Thessaloniki [14].

Another study, carried out at IRSN, has evaluated the impact of an additional volatile iodine source term one day after a severe accident transient, with ASTEC simulations on PWR 900 reactor. The trends obtained confirm that the recombiner/iodine issue deserves further investigation since, in some cases, it could significantly impact on the iodine source term. Knowledge gained at this stage on iodine volatility in recombiners can be extrapolated to the potential interference of recombiners with the ruthenium source term.

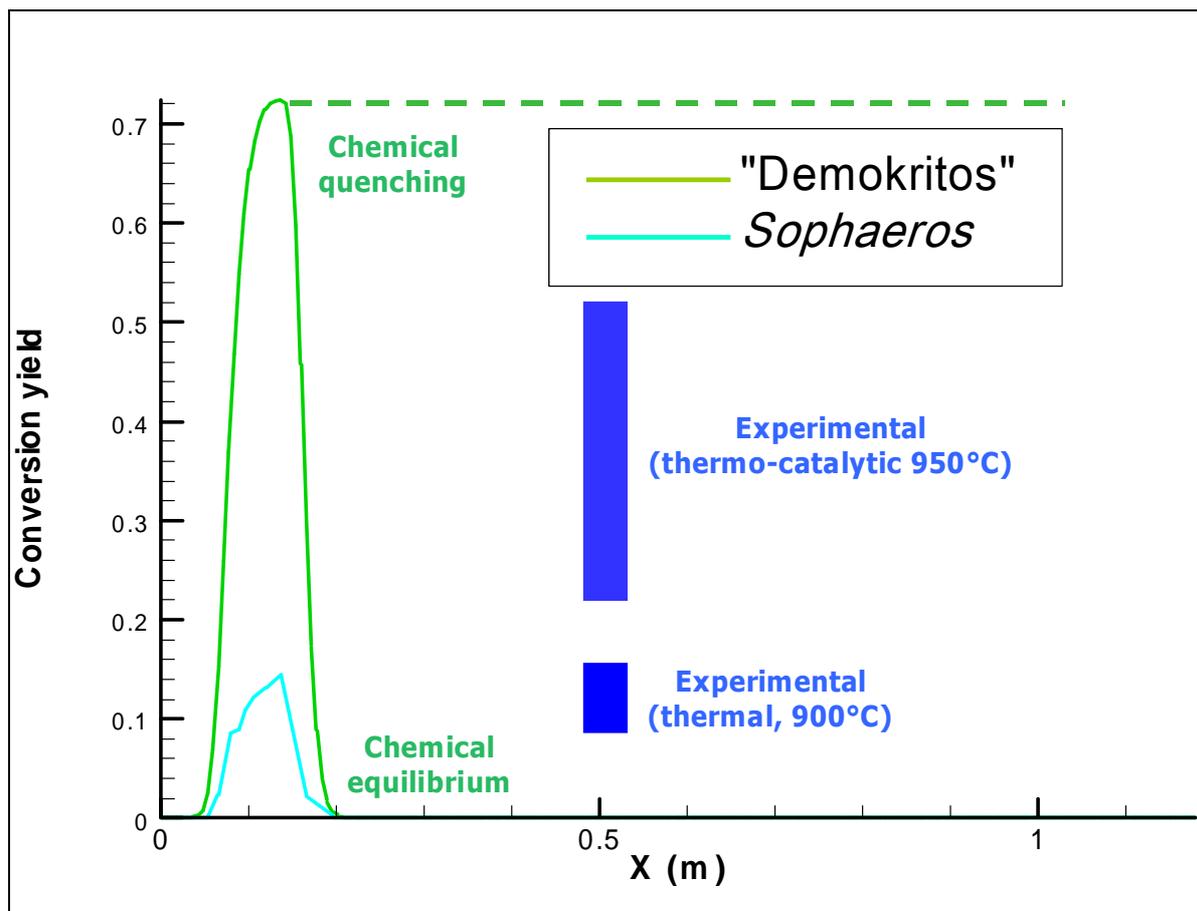


Figure 7. Numerical simulations (950°C) by "Demokritos" and ASTEC/*Sophaeros*, and comparison with RECI (900°C) experimental results, the later ranging between perfect quenching and thermochemical equilibrium. At the heated tube entrance, flow velocity (STP) is  $0.3 \text{ m.s}^{-1}$  and aerosol diameter  $0.32 \text{ }\mu\text{m}$ .

## E IODINE DATA BOOK (IDB)

The chemistry of iodine in the context of reactor safety assessments has been extensively studied over the past two decades. This has resulted in a large body of data, which has been used in the development of different models and methodologies for the prediction of the iodine source term. Although research in this area is continuing, notably in France and Canada, work on iodine chemistry is generally diminishing; for example the experimental programme within the UK nuclear industry came to an end in 2003. It was therefore considered timely to undertake a collation of the experimental and theoretical information that provides the foundation for the iodine chemistry safety case for Sizewell B. The Iodine Data Book aims to provide a critical review of the data that have been used in the development and validation of iodine chemistry models. The data book is in six parts, covering aqueous phase inorganic radiation chemistry, organic iodine chemistry, surface reactions, mass transfer, gaseous iodine chemistry, and large scale experiments and models. It is intended that the Iodine Data Book be a living suite of documents that is updated to take account of new experimental data from existing and future programmes.

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