

## The Chemistry and Conditioning Programme for MYRRHA

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**Abstract.** In 2010 the Chemistry and Conditioning Programme (CCP) has been established to provide R&D support for engineering and licensing of MYRRHA. MYRRHA is an accelerator-driven nuclear system, using liquid lead-bismuth eutectic (LBE) as spallation target material and coolant. The CCP team studies various chemistry-related aspects of LBE which are important for safety, operation and decommissioning of MYRRHA.

A large portion of our research is devoted to the measurement and control of dissolved oxygen in LBE. Our achievements in this domain include the development of a family of new oxygen sensors that perform reliably down to 200 °C, and several systems for oxygen control based on controlled lead oxide (PbO) dissolution, gas-liquid interactions and electrochemistry. The chemistry of impurities such as corrosion products in LBE and methods for managing these impurities are also being studied. An integrated experimental study of oxygen control, sensing and impurity chemistry and removal is carried out in our large chemistry loop MEXICO. This experimental work is supported by a variety of theoretical calculations. Thermochemical calculations have been successful in predicting the influence of temperature and impurities on measured concentrations of oxygen. Detailed CFD calculations coupled with chemical reactions are developed to assess reactions and transport of oxygen and impurities in the complex geometry of the primary system of MYRRHA.

The second priority of the CCP team is the experimental study of evaporation of safety-critical radionuclides from LBE. These radionuclides are formed in the LBE by activation of the coolant (Po), by spallation (Hg, ...) or may be released into the LBE through leaking fuel pins (fission products such as I). For the experimental study of polonium release, considered to be one of the most important safety issues of LBE-cooled reactors, a dedicated Po lab has been set up. The evaporation of other elements is typically studied using stable isotopes in a newly built heavy metals chemistry lab. In international collaborations, several physicochemical mechanisms by which Po can be released from LBE have been discovered and studies on methods to capture especially volatile gaseous Po molecules have been performed.

**Key Words:** MYRRHA, LBE, coolant chemistry, oxygen control

### 1. Introduction

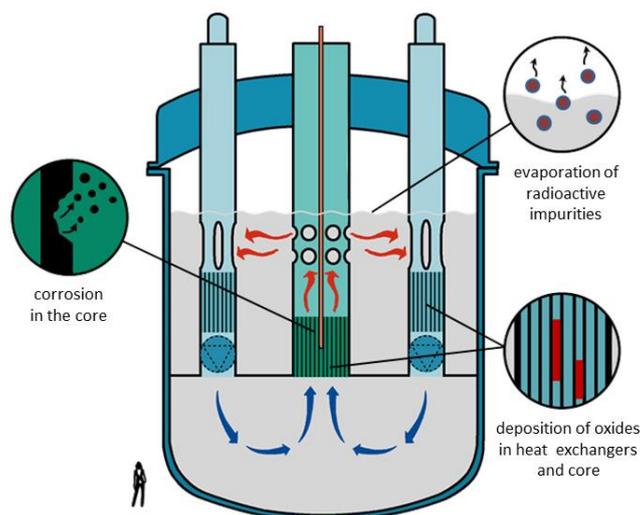
MYRRHA, a flexible fast spectrum research reactor (50-100 MWth) under design at the Belgian Nuclear Research Centre, is conceived as an accelerator driven system (ADS), able to operate in sub-critical and critical modes. It contains a proton accelerator of 600 MeV, a spallation target and a multiplying core with MOX fuel, cooled by liquid lead-bismuth eutectic (LBE).

The application portfolio of MYRRHA includes demonstration of the physics and technology of an Accelerator Driven System (ADS) for transmuting long-lived radioactive waste [1], the testing of materials and fuels for fast spectrum reactor and fusion technology development, the production of radioisotopes for nuclear medicine and fundamental research. As an LBE cooled nuclear system, MYRRHA is also listed as the pilot plant for lead fast reactor technology in the European demonstration programme for fast neutron reactors, ESNII.

In MYRRHA, LBE is besides the primary coolant also the spallation target. The use of LBE and the choice to implement several advanced safety features in the MYRRHA design presents several safety and technological challenges, which have been under investigation at SCK•CEN during the past years.

The chemistry of LBE coolant involves the reactions of the main coolant constituents lead and bismuth, of dissolved oxygen and of a large variety of impurities, present either already in the non-irradiated LBE, released in the LBE by steel corrosion (Fe, Ni, Cr, Mn) or formed by nuclear reactions with the coolant. In an LBE-cooled ADS such as MYRRHA, critical nuclear reaction products are spallation products with masses close to that of the coolant atoms (such as mercury) and coolant activation products, most importantly polonium. Chemical reactions of LBE and its impurities have a strong influence on several safety related processes (*FIG. 1*). Dissolved oxygen has been shown to affect corrosion of steel used for fuel cladding and structural components. Reactions of lead and corrosion products may cause the formation of solid particles and deposits, frequently as oxides, and nuclear reaction products may evaporate from the LBE into the cover gas space.

Many important chemical processes in the coolant are not well understood or lack sufficiently accurate data to allow predictive calculations and technology development [2]. With the conditioning and chemistry programme, SCK•CEN has addressed several technological challenges and scientific gaps, in particular in the fields of oxygen control, impurity chemistry and release of safety-critical radionuclides from LBE. In the following sections we present an overview of the state-of-the-art of SCK•CEN's activities in these fields.



*FIG. 1. Sketch of the MYRRHA reactor vessel illustrating safety issues caused by chemical reactions in the LBE coolant. The direction of the LBE flow in hot and cold plenum is indicated by the red and blue arrows, respectively.*

## 2. Oxygen measurement and control

Within the multitude of possible chemical interactions in the coolant of MYRRHA, oxygen is a central element because of its high chemical affinity with most other elements. At SCK•CEN, the project on oxygen control is a continuous effort, with the aim to develop systems that are able to accurately measure and control the oxygen concentration in the LBE of MYRRHA to avoid coolant oxidation and reduce corrosion.

The range of allowed oxygen concentrations in the LBE depends on temperature and is bounded at high oxygen concentrations by the lead oxide (PbO) solubility line (top blue line

in FIG. 2). On the low-oxygen concentration end, typically the lower magnetite stability line is taken to be the boundary (where magnetite is in equilibrium with solid iron). However, when considering a set of corrosion test results reported in the OECD handbook on heavy liquid metal technology (black markers in FIG. 2) [3], it is clear that undesired dissolution corrosion may also occur above this magnetite stability line. On the other hand, dissolution corrosion does not seem to take place with high probability above the stoichiometric line for the magnetite solubility product (where the red lines in FIG. 1 converge, see also Eq. 1). In LBE cooled systems, it seems therefore desirable to target oxygen concentrations in the excess oxygen region. In MYRRHA, which will normally operate between *ca.* 200 °C and 400 °C, an oxygen concentration of about  $10^{-7}$  wt.% is therefore envisaged.

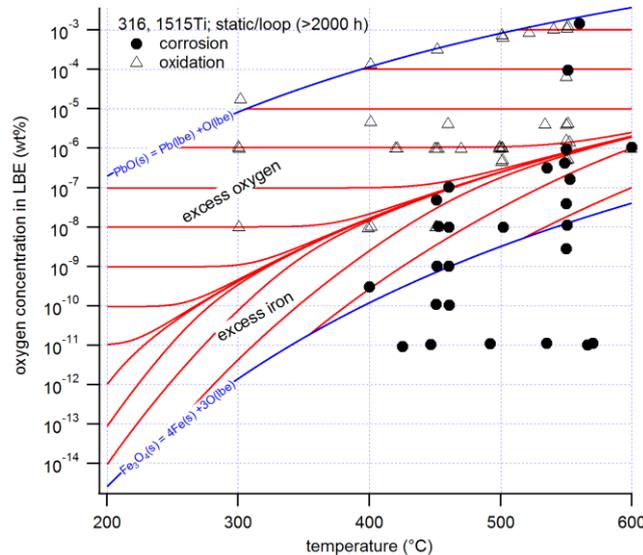


FIG. 2. Oxygen control boundary for LBE cooled systems.

Oxygen sensors capable of measuring down to 200 °C have been developed [3,4], to accurately measure the concentration of dissolved oxygen in the entire MYRRHA temperature range. Ceramic solid electrolyte, reference electrode and sensor assembly methods have been optimized for application in loops, where the ceramic-to-metal joint is not exposed to LBE, as well as for application in deep LBE pools, where long sensors with submerged joint are required.

To allow for diversity, multiple approaches for oxygen control have been evaluated and developed. Oxygen control systems based on gas-LBE interaction, either *via* a cover gas or through bubbling have been studied [5]. Oxygen addition by controlled dissolution of lead oxide in a so-called PbO mass exchanger (PbO MX) has been evaluated experimentally and numerically [6-8]. A new approach toward oxygen control in LBE called electrochemical oxygen pumping (EOP) has been developed [9] and tested in various experimental conditions. Currently, oxygen reduction by means of cold trapping is also investigated.

LBE chemistry and oxygen control in large systems has been studied at SCK•CEN in the MEXICO, HELIOS 3 and CRAFT installations. MEXICO is a pilot-scale non-isothermal loop with an LBE inventory of 7 tons, where cold and hot leg temperatures are representative for MYRRHA, i.e. ~200 °C and ~400 °C, respectively (FIG. 3). At present MEXICO accumulated more than 10000 h of operation, during which it has been used for oxygen control, lead oxide deposition, and impurity filtering experiments (FIG. 4). Accurate oxygen

control in MEXICO has been achieved using the PbO MX technology (FIG. 4, inset) [10] and electrochemical oxygen pumping (4-1 in FIG. 4).

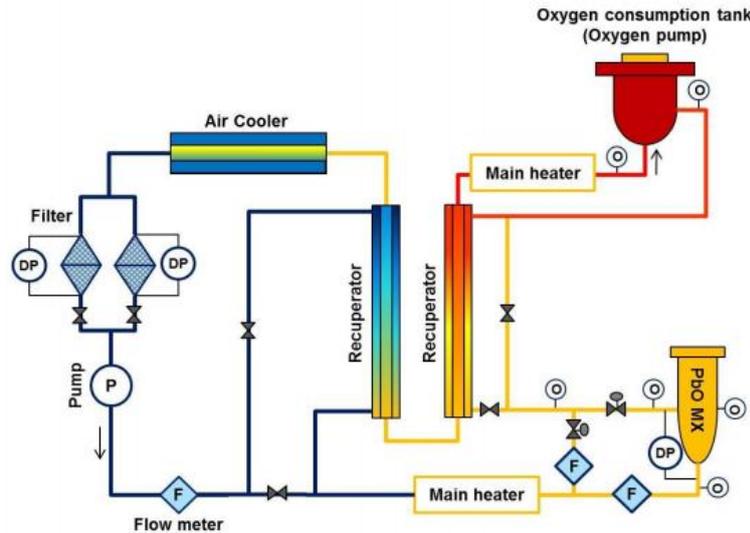


FIG. 3. Flow diagram of the MEXICO chemistry loop [10].

In parallel, several small LBE setups are used to test new oxygen sensors. An autoclave equipped with a stirrer called CHECKMATE is used to investigate mass transfer effects on oxygen control by the electrochemical pumping method. Small autoclaves are also being used for the measurement of key chemical properties of dissolved oxygen in LBE, such as the Sieverts constant which describes the equilibrium between dissolved oxygen and oxygen gas.

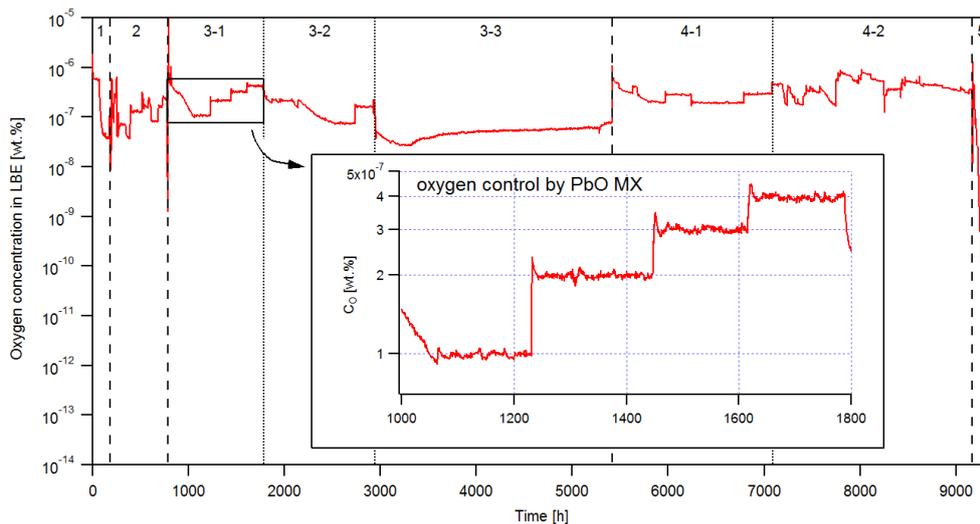


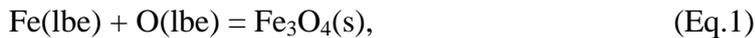
FIG. 4. Flow Oxygen concentration history of the MEXICO loop. Each section represents a different experiment. Inset: enlarged section 3-1, in which accurate oxygen control by the PbO MX technology was demonstrated. Cold and hot legs were at 210°C and 430 °C, respectively [10].

### 3. Impurity chemistry and management

Impurity chemistry and management deals with dissolved and solid impurities in the coolant which are released to that extent that they may accumulate to macroscopic amounts during the lifetime of MYRRHA. Corrosion products are expected to be released continuously in the LBE of MYRRHA in amounts that can potentially cause blockages of narrow flow paths

inside the primary system. Blockage formation may occur if dissolved corrosion-derived impurities deposit on surfaces, or first precipitate in the bulk of the LBE and then deposit. The driving forces for precipitation and deposition are governed by the thermodynamics of solids-forming reactions. In a first step toward predicting the location and amount of deposits in MYRRHA, it is therefore important to gain understanding in the precipitation-dissolution equilibria in LBE.

One of the abundant corrosion products expected to be released in the LBE of MYRRHA is iron. The reaction between dissolved iron, and dissolved oxygen in liquid LBE has been characterized [11]. It was found that many experimental observations, such as the change of dissolved oxygen concentration during temperature changes in a system with constant composition, can be described by the simple equilibrium:



where dissolved species are indicated by (lbe).

This reaction was also found to have a strong impact on the local oxygen concentration and precipitate formation in larger LBE installations such as the CRAFT corrosion loop [11]. Large differences of the oxygen concentration between the hot leg, where the oxygen control point was located, and the cold leg were observed under certain conditions. To investigate these effects, a dedicated test was conducted (FIG. 5) in which the oxygen concentration was gradually decreased. At a certain oxygen concentration in the hot leg, the oxygen concentration in the cold leg started to deviate strongly from that in the hot leg (FIG. 5, blue curve). The concentration at which the deviation started was close to the stoichiometric point of the magnetite equilibrium, suggesting the involvement of the iron-oxygen-magnetite equilibrium and that the deviation occurred upon transition of the LBE from an excess oxygen to an excess iron regime.

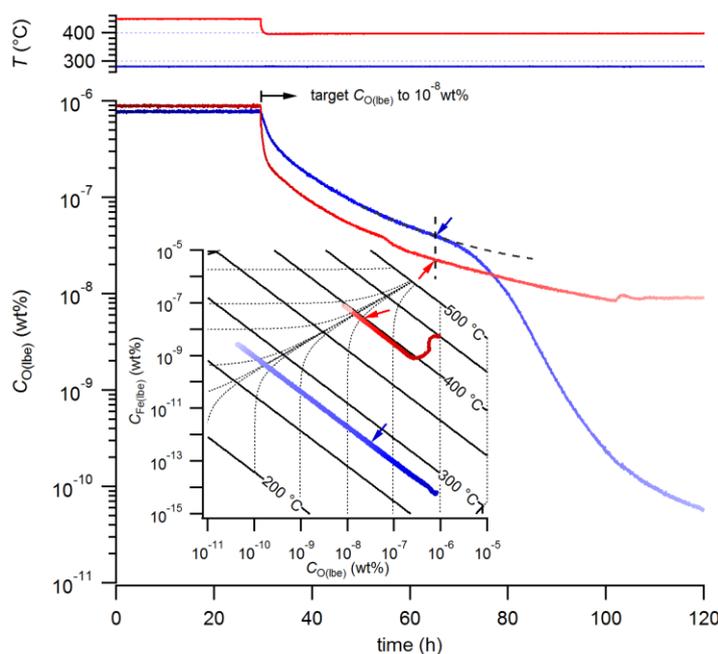


FIG 5. Oxygen concentration variation in the hot (red curves) and cold (blue) zones of the CRAFT LBE loop. The trend in the cold zone starts deviating from that in the hot zone after about 65 h due to magnetite precipitation, as indicated by the arrows. The inset shows the evolution of hot and cold zone oxygen concentrations in the magnetite solubility product diagram [11].

Another important step toward prediction of impurity particle and deposit formation in a nuclear system such as MYRRHA is the characterization of the formation kinetics of solids in the LBE coolant. FIG. 6 shows experimental results of the ratio of the measured dissolved oxygen in LBE to the concentration in equilibrium with lead oxide at each temperature, i.e. the degree of oversaturation. In an experiment in which the temperature of the LBE was cycled between 300 °C and 450 °C, the trend of the oversaturation can be attributed to nucleation and growth (upon temperature decrease) and dissolution (upon temperature increase) of lead oxide particles in the LBE. The observed trends were surprisingly well-described by a model based on classical nucleation and growth theory (FIG. 6) [12].

Phenomena related to lead oxide nucleation, growth and dissolution in LBE flowing in a non-isothermal system were clearly observed in the MEXICO chemistry loop (Fig. 6, right). Using the PbO MX, the oxygen concentration in the hot leg (350 °C) was increased to 1.53 times the saturation concentration at the cold leg (210 °C). Subsequently, oxygen addition was stopped. When flowing from hot to cold leg, the oxygen oversaturation in the LBE dropped to 1.12, indicating PbO formation either as suspended particles or as deposits. The observation of a slight remaining oversaturation indicated that the PbO kinetics were not sufficiently fast to completely equilibrate with the temperature of the flowing LBE (4 kg/s). Redissolution of PbO was observed by an increase of the oversaturation to 1.52 measured in the intermediate temperature region (280°C) which is located downstream of a filter section in the cold leg. This indicated that most of the PbO precipitated as small particles in the LBE bulk, rather than deposited on the walls of the cold leg.

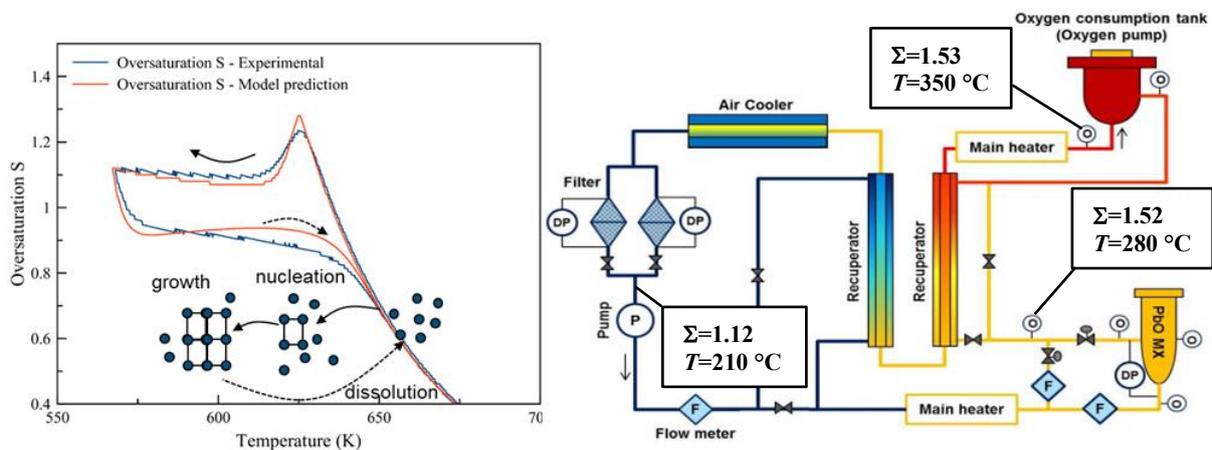


FIG. 6. Left: (blue curve) Measured degree of oversaturation of dissolved oxygen in LBE during an experiment in which the LBE temperature was cycled between ca. 450 °C and 300 °C. The arrows indicate the direction of time. (red curve) Model for the observed oversaturation based on classical nucleation and growth theory. Right: Experimental measurement of degree of oversaturation at different locations in the MEXICO LBE loop with respect to cold leg temperature ( $\Sigma$ ).

Thermodynamic and kinetic models of solids forming reactions due to corrosion products impurities and PbO are being implemented in CFD models to predict solids formation, transport and deposition in the the complex geometry, flow and temperature fields of the MYRRHA primary system. For example, a single-phase chemical equilibrium model was recently developed in order to identify the regions in the reactor with the highest probability for magnetite formation. FIG. 7 shows a quadrant of the MYRRHA core and upper plenum. The color scale indicates the concentration of dissolved iron in equilibrium with magnetite at the local temperature and oxygen concentration. The simulation shows that entrained magnetite particles dissolve when they pass the core, and then reprecipitate in the colder, lower regions of hot plenum.

The transport of already formed impurity particles in the primary system is studied with a multiphase particle tracking model. This allows to investigate the behavior of a dispersed phase having a drift velocity with the main flow due to buoyancy and the effect of particle size. This is of primary importance to identify whether the formed particles will accumulate and where accumulation might occur, for example at the cover gas – LBE interface, or whether the particles will remain suspended in the LBE and follow the flow. Such models are used for safety evaluations and to engineer LBE filter systems for MYRRHA.

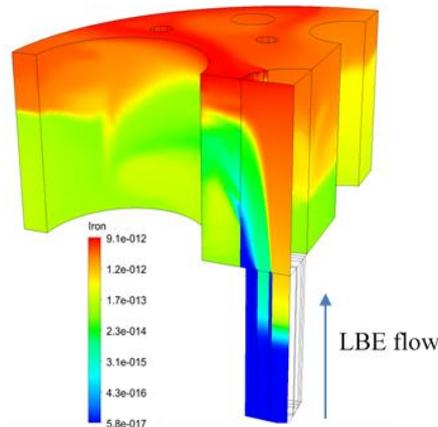


FIG. 7. Predicted concentration of dissolved iron in equilibrium with magnetite at the local temperature and oxygen concentration in LBE in a quadrant of the MYRRHA core and upper plenum.

Dedicated experiments which address deposition phenomena are currently being carried out in the MEXICO chemistry loop. In MEXICO, a test section is equipped with a scaled MYRRHA fuel bundle (FIG. 8) to study lead oxide deposition and associated increase of the pressure drop under both normal operating and accidental conditions. Such experiments are only feasible because of the very accurate control and measurement of dissolved oxygen in both hot and cold legs of the MEXICO loop.

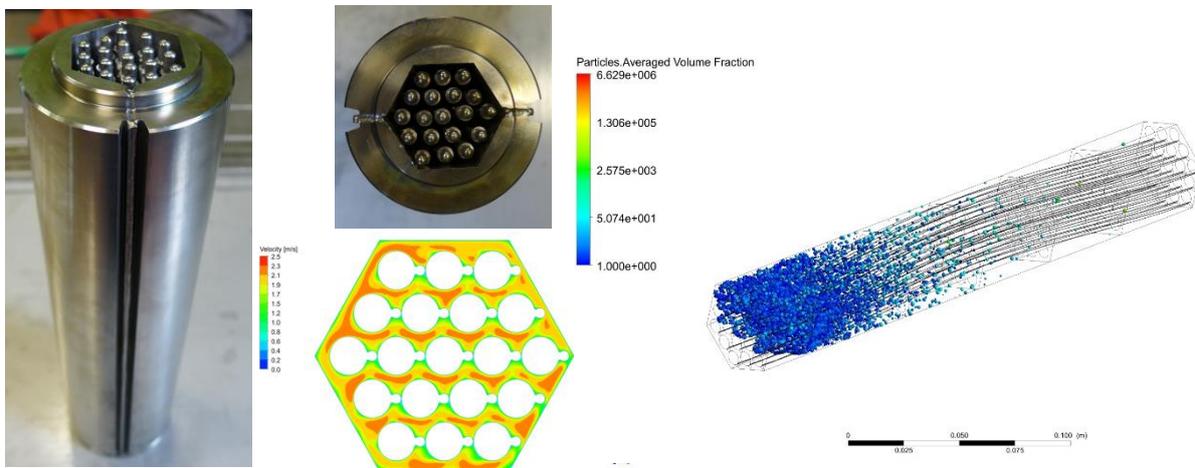


FIG. 8. (left) Pictures of the MYRRHA fuel assembly mock up used to study deposition phenomena in the MEXICO LBE chemistry loop and cross-sectional velocity field predicted by CFD. The figure on the right depicts a simulation of impurity particle transport in the fuel assembly.

#### 4. Fission and spallation product chemistry and evaporation

Volatile fission products (I, Cs, Te, ...) may be released into the LBE coolant after a breach in the fuel cladding. As input for safety calculations, the release of fission products from LBE needs to be known quantitatively under various MYRRHA relevant conditions and scenarios. To certain extent, the chemical properties of fission products are known or can be estimated from existing data. Therefore, we currently focus our experiments to cases where literature data are lacking.

Iodine is a clear example of a radiologically important element for which thermochemical data in literature were insufficient to evaluate its equilibrium evaporation from LBE. Iodine is commonly believed to be well retained in lead alloy coolants [2]. To verify this claim, we have characterized iodine evaporation from dilute solution in LBE by the so-called transpiration method and confirmed, consistent with experiments at much lower concentrations using a different source of iodine,[13] that iodine evaporation from LBE is indeed strongly suppressed compared to evaporation of pure iodine.

The experimental iodine evaporation data from LBE were then analyzed by thermochemical modeling (*FIG. 9, left*), which allowed estimating the distribution and chemical speciation of iodine among liquid, condensed and gas phases. Under the conditions of the experiment (*FIG. 9, right*), most of the iodine is dissolved in the LBE above 250 °C. Similar calculations were performed for the MYRRHA system. The partial pressure of all iodine species combined is then used to determine the contribution of iodine to the gaseous source term for the evaluation of the radiological consequences of accidents.

As opposed to fission products, spallation products are unique to ADS systems such as MYRRHA in which coolant and spallation target form a single entity. One important spallation product is mercury, which is both volatile and produced with high yield. In the frame of MYRRHA, mercury evaporation from LBE has been characterized using a dedicated setup [14].

Even when considering only the fission products, the number of possible interactions in the primary system is extremely large. It is therefore not feasible to perform experiments that fully cover the range of compositions and conditions to be expected in MYRRHA. For example, the currently performed iodine evaporation experiments do not take into account interactions with other fission products which may affect retention capacity of LBE for iodine, both positively and negatively. Thermochemical modeling is used to study such complex interactions and identify possible safety issues. Targeted experiments are then conducted to verify the model predictions. Results from the MEGAPIE experiment serve as ultimate cases to test model predictions against.

Besides the simple experimental setup for transpiration-type evaporation experiments, the thermochromatography technique is being used in the frame of MYRRHA, at the Swiss Paul Scherrer Institute (PSI), to characterize vapors of fission products that emanate from LBE [15]. At SCK•CEN, a more advanced experimental setup to characterize fission product evaporation from LBE using mass spectrometry of stable fission product isotopes has also been constructed recently.

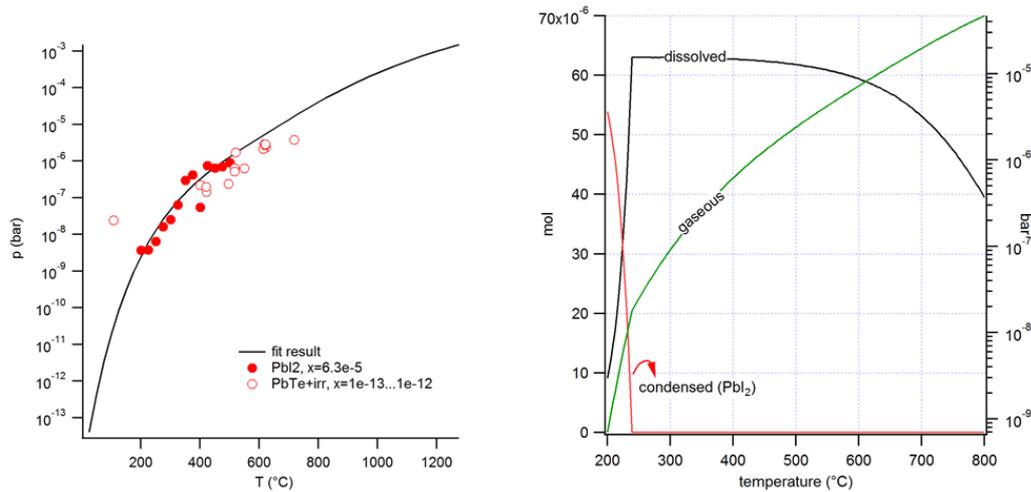


FIG. 9. (left) Data and simulation of the iodine partial pressure at equilibrium with dilute solution of iodine in LBE. (right) Calculated partitioning of iodine in solution, gas and condensed phases in the conditions of the experiment.

## 5. Polonium chemistry in LBE

In addition to spallation and fission products, radionuclides will also be formed due to neutron activation of the coolant. The presence of 55% bismuth in the LBE leads to the production of relatively large quantities of polonium-210 during operation of MYRRHA. The chemistry of polonium and especially its release has been one of the focus points of the conditioning and chemistry programme at SCK•CEN. Using the transpiration method, the release of polonium from dilute solution in LBE has been quantified under various experimental conditions [16-20], importantly the composition of the gas atmosphere in which polonium evaporates (inert, reducing, oxidizing, humid). To enable constructing models which describe the observed evaporation behavior, the thermochemical properties of the relevant polonium gas molecules need to be known. These properties cannot be measured using traditional methods because of the low concentration of gaseous polonium molecules in experiments. We rely instead on thermochromatography experiments [21-23] which provide qualitative information on the nature of the gas species under various conditions, and on modeling. Two approaches towards modeling are adopted in parallel, *viz.* methods that rely on extrapolation of trends in the chalcogenide group and quantum chemical methods [24]. Since polonium gas molecules involve heavy atoms, these quantum chemical methods need to take into account sufficient extent of relativistic effects in order to produce accurate results.

Besides providing insight into the nature of polonium gas molecules, thermochromatography experiments also allow the characterization of the interaction of polonium vapors with surfaces. Recent important results obtained in collaboration with PSI include the characterization of adsorption (sticking) of polonium-bearing vapor molecules on surfaces, and the derivation of the corresponding thermochemical interaction properties. Concurrently, *ab initio* studies of the interaction of polonium with metal surfaces have been performed [25,26]. The latter results are used in safety studies to assess transport of polonium vapors, after their release from LBE, in various components of MYRRHA such as the proton beam tube.

## 6. Conclusions

The main coolant chemistry issues encountered in LBE-cooled nuclear systems are addressed by the Chemistry and Conditioning programme at SCK•CEN in the frame of MYRRHA.

Oxygen sensor and control technology is now considered to have reached a high degree of maturity. In terms of technological readiness level (TRL), as defined by the European Commission, we estimate that a TRL of 6 is reached.

In the field of impurity chemistry and management, significant progress has been made but several open questions regarding precipitate and deposit formation still need to be addressed.

Using a combination of experiment and modeling, the understanding of fission, spallation and activation product chemistry in LBE has been greatly improved. Complex interactions are now thought to be important and will be addressed in future studies. To refine polonium release calculations from LBE, future research will include among others, the determination of the thermochemical properties of critical polonium gas molecules.

It is finally noted that many of the technologies and scientific results presented in this paper are not limited to application in MYRRHA or even LBE. They can be applied to lead coolant with only minor adjustments.

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