# Investigation strategy for chemical compatibility study on stainless steel with liquid sodium after in service solicitations: feedback from French Sodium Fast Reactor after 12 years of in power operation

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**Abstract**. Phénix, the French fast breeder Nuclear Power Plant (NPP) was shut down in 2009. The dismantling operations for some of its components are underway. Through modeling in the context of R&D projects the investigations of its components will help to predict the compatibility with Na for the components of the future Gen IV ASTRID prototype reactor. Since 2008, CEA, EDF and AREVA have identified more than twenty of these components as critical for obtaining relevant feedback relating to their operations. Substantial amounts of valuable information regarding Phénix NPP component materials such as austenitic stainless steels can be gained, since the base metal or welds subjected to normal and abnormal service conditions are difficult to reproduce in the laboratory. In 2012, the first sampling was performed on the Phénix CPML0373 rod made of 304L and 316L austenitic stainless steel which had been exposed to sodium (Na) at high temperature for about twelve years effective full power. This paper highlights (i) the investigation strategy, (ii) the dismantling steps for the rod, and finally, (iii) the sampling to obtain as much feedback as possible on such rods as regards the chemical compatibility of their materials with liquid sodium. Examinations (SEM, EDX, XRD) on the Phénix rod are underway and will be the opportunity to improve overall understanding of Na coolant chemistry and its interactions with materials. Finally, data will be obtained from the examinations as to long term corrosion behavior.

**Key words**: Sodium fast breeder reactor, investigation management, Phénix NPP, Astrid prototype

## 1. Introduction

The future ASTRID reactor (Advanced Sodium Technological Reactor for Industrial Demonstration), designed by the CEA together with its industrial partners, has been subjected to a very high level of requirements. Innovations have been included in the design with, for example, advances on core and sodium-related issues and the taking into account of lessons learned from the Fukushima accident. These enhance safety while improving reliability and operability, making the Generation IV Sodium Fast Reactor (SFR) attractive [ 1 ]. Consequently, these technological advances combined with the new safety features have led to new needs in terms of qualification and of demonstration of the appropriateness of the proposed safety options, as well as the efficiency and robustness of the concepts for a sixty year NPP lifetimes. In this context, the choice and qualification of in-core and out-of-core materials is a challenge. ASTRID has to integrate operational feedback from past reactors like Phénix and Superphénix NPP [2-3]. Phénix is part of the Marcoule site in France, on the banks of the Rhone River. Phénix is a prototype nuclear power plant of the sodium-cooled fast breeder reactor type. It is an integrated type reactor, meaning the core, primary pump and intermediate heat exchangers are located in the same reactor vessel.

The shutdown of Phénix NPP in 2009 and its current dismantlement have provided an opportunity to lever knowledge and know-how in support of the qualification of the ASTRID prototype and its associated R&D needs. A work package called “Phénix Treasures” has been set up to select relevant irradiated objects for R&D needs, and to propose future experimental examinations of standard or non-standard Phénix irradiated objects [4]. The examinations have been prioritised depending on their relevance with respect to current options for the ASTRID reactor [5].

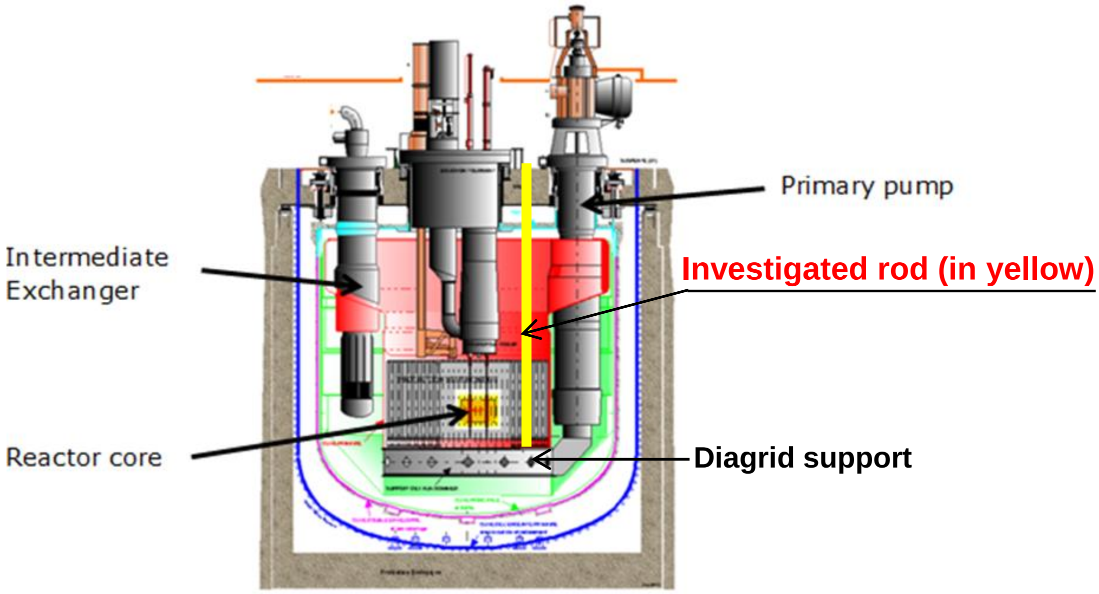

*FIG. 1. Phénix NPP, location of the rod (yellow) in the reactor vessel.*

In 2008, a special working group was set up, called GTEMP, or working group for Phénix NPP out-of-core materials feedback. This paper focuses on out-of-core materials and particularly on the preparation of samples through a specific organization. Preparation for the dismantling of some Phénix components has been underway for some time, and the CPML0373 rod is the first component starting to be investigated with the methodology developed by the GTEMP working group presented in this paper.

## 2. Context

The ASTRID design benefits from a vast experimental data base relying on the feedback from numerous tests, particularly in the French Phénix and SuperPhénix reactors. As indicated above, industrial maturity in terms of fabrication and irradiation-related experience gained through the French SFR program has enabled the development of robust design and material solutions for the first ASTRID core. These solutions have to be validated, either by post irradiation examinations on irradiated pins or structures from Phénix and SuperPhénix reactors, or by irradiation experiments under representative conditions.

The first task for GTEMP was to identify the useful parts of components in order to prepare an inventory and to prioritize the components with respect to their appropriateness for SFR materials and particularly for the ASTRID prototype qualification [6]. Possible investigations were assessed regarding different types of material damage, for example excessive and progressive deformation and compatibility issues in sodium, or the resistance of coatings. More than one hundred such investigations have been identified, with around fifty of great interest for fast reactor studies, especially the ASTRID project.

Prior to the implementation of these examinations, a considerable amount of work is needed to ensure relevant, correct sampling. Furthermore, material sampling has to be coordinated with the on-going dismantling process. As a consequence, investigation guide sheets have been written for the different components where investigations are needed. These documents are key technical reports required to successfully meet the objectives through targeted investigations.

## 3. Investigation methodology

After having identified the most useful component parts for the projects, the writing, the instruction, and further work on the investigation guide sheets is the present goal of the working group. To successfully implement them strong links are needed with different teams: project teams and operational teams from R&D and Phénix operation. The know-how and the recollections of former and present Phénix employees are recorded for future reference. This approach applies to all the components whose expert examination can contribute information of interest for ASTRID, but also for the SFR industry as a whole. It involves a huge volume of work which must continue over many years, taking into account the Phénix dismantling schedule and the dates foreseen for certain components to be made available (lasting through to 2040). The investigation guide sheets drawn up for each of the components by the GTEMP working group combine: (a) Capitalization on historical data, (b) Analysis of the data by the GTEMP working group, (c) Proposition of an investigation program, (d) Implementation of the investigation program in close collaboration with the dismantling project, (e) Carrying out expertise assessments, (f) Interpretations by the GTEMP, and (g) Transfer of the expertise results/interpretations as input data for Astrid and for R&D working groups.

As concerns capitalizing on historical data and the analysis of such data, since it was first set up the GTEMP has been writing data files for about fifteen components, for example the intermediate heat exchanger, primary and secondary pumps, secondary loop and the CPML0373 rod. The investigation guide sheets propose an investigation program in line with the needs of the ASTRID project, taking into account (i) the different types of material damage of interest include the effects of irradiation, thermal aging, corrosion under stress, compatibility issues in sodium, coating resistance, excessive and progressive deformation, thermal fatigue, and creep-fatigue/reheat cracking [7], (ii) Phénix dismantling constraints, and (iii) the special features of the experimental resources of the expertise laboratories.

Certain non-destructive analysis steps are essential to best locate the component sampling sites, see example of sample #9 location as identified from sample #7, hereafter (in section *proposition of an investigation program*). On the Phénix NPP site, the first step consists in an expert assessment at complete component scale: metrology to detect any deformation while in service, penetrant testing to reveal any cracks. Then samples are taken in the zones identified by the previous step (the most deformed or most corroded zones), with the sampling of sections of interest respecting the special instructions noted in the investigation guide sheet. The samples are labeled with information about the location (fluid circulation direction, angular sector), cutting precautions, and possible decontamination. This work is undertaken by the Phénix NPP teams. For sample transport, operational support is given by the "Trésor Phénix" project team, taking into account the sample radiological state and the expertise laboratory operating rules. Finally, an expert assessment is carried out on the samples at coupon test laboratory scale. The detailed investigation program is established in collaboration with the host laboratory which will precisely define the examinations to implement in order to meet the issues investigated.

The investigation guide sheets, which represent the basis for the program set up in order to acquire material data of interest, are updated appropriately when the steps initially planned for are carried out at Phénix or in the laboratories. The results and their interpretations by GTEMP are made available for future use in R&D, modeling and design decision validations. The case of the CPML0373 Phénix rod described in the next section is the first implementation of this investigation methodology.

## 4. Expertise of the rod

### 4.1. Capitalization and analysis on the rod historical by the GTEMP

The rod CPML0373 was located between the rotating cap and one of the primary pumps (*FIG. 2*). It was chosen because of its operational history (longitudinal thermal gradient differences) and because it was available for the application of the GTEMP investigation methodology. The rod is an interesting component because it had been in the Phénix NPP Na environment from the beginning (in 1974) and had had temperature gradients from 400°C to 550°C. Furthermore due to this gradient, there had been numerous variations of Na operating level meaning that investigation of the floating zone was of interest to study the mechanical properties.

The rod had been submerged in liquid sodium for about 234 000 hours (less than 27 years), including 100 000 hours of power operation (~12 years). During this time, the reactor had a total of 398 normal or abnormal start/stop transients (fast and emergency stops), including 145 normal start/stop transients. The rod environment is summarized in the table presented (*TABLE I).*

The investigated rod (*FIG. 2*) served several purposes in the Phénix reactor, for example measuring the position of the diagrid support and measuring temperatures in the hot collector (*FIG. 1)*. The CPML0373 rod consisted of 4 main parts and the investigation presented in this paper deals with the protection tube. It is about 11 m long and consists of a double diameter tube (Ø 219 mm and Ø 159 mm), perforated at 3 different levels to let the liquid sodium flow inside the tube. The lower part has a conical section which was plugged into the core support structure of the reactor vessel.

*TABLE I.* ENVIRONMENT OF THE ROD DURING ITS OPERATION.

| Period | Location | Environment |
|---|---|---|
| **1974-2001** | Reactor vessel | In Na for 27.5 years, including ~12 years at effective full power |
| **2001-2012** | Storage pit | Inert gas |
| **2012** | Dismantling | Air |

The upper part of the rod was made of 304L grade steel and has a 219 mm diameter, 3.76 mm thick. The lower part was made of 316L grade steel, 159 mm in diameter and 4.5 mm thick (*FIG. 2.*).

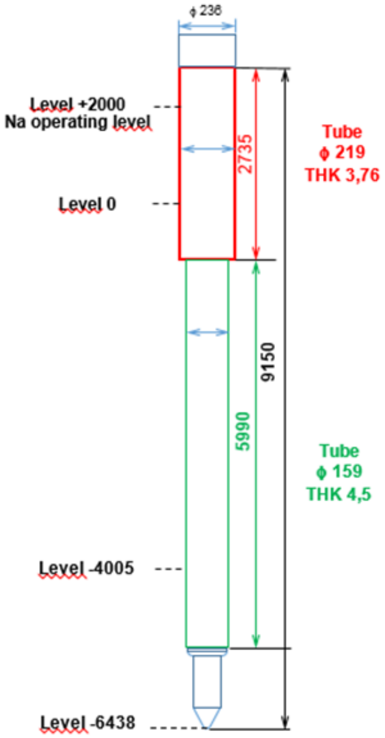

*FIG. 2. Phénix CPML03763 rod description.*

To confirm the rod fabrication report, ICP-AES and carbon analysis were carried out on 2 samples from each rod part (*TABLE II*). Carbon analysis results are the average of 3 different measurements.

*TABLE II: CHEMICAL ANALYSIS TO CHECK CPML0373 ROD UPPER AND LOWER PART MATERIAL.*

| **Element (Wt%)** | **Upper part analysis** | **Theoretical 304L** |
|---|---|---|
| **C** | *0.04 (+/-10%)* | *<0.08* |
| **Cr** | *18.1* | *17 to 19* |
| **Ni** | *9.2* | *8 to 10* |
| **Mn** | *1.4* | *<2* |
| **Si** | *0.8* | *<1* |
| **Mo** | *0.2* | *0* |
| **Fe** | *Bal.* | *Bal.* |

| **Element (Wt%)** | **Lower part analysis** | **Theoretical 316L** |
|---|---|---|
| **C** | *0.028 (+/- 10%)* | *<0.03* |
| **Cr** | *17.3* | *16-18* |
| **Ni** | *13.0* | *10-14* |
| **Mn** | *1.7* | *<2* |
| **Si** | *0.8* | *<1* |
| **Mo** | *2.7* | *2-3* |
| **Fe** | *Bal.* | *Bal.* |

### 4.2. Proposition of an investigation program

The GTEMP data analysis and interpretation has demonstrated that in Phénix, materials showed good chemical compatibility in a sodium environment where a low oxygen concentration was maintained. For austenitic steel components, corrosion in liquid sodium is considered to be negligible. Examinations of the rod from Phénix were the opportunity to assess this hypothesis and to obtain relevant data for corrosion modeling. To study the damage from a sodium environment, samples of 304L and 316L were cut before the decontamination process from zones differing in terms of environment, temperature, flux and sodium flow rate. To study mechanical properties, samples were cut after decontamination only on the 304L part. At this step, the GTEMP working-group had identified the rod sampling points and the Phénix operation and handling teams received detailed sampling specifications (*FIG. 3*). Two groups of samples were identified before and after decontamination.

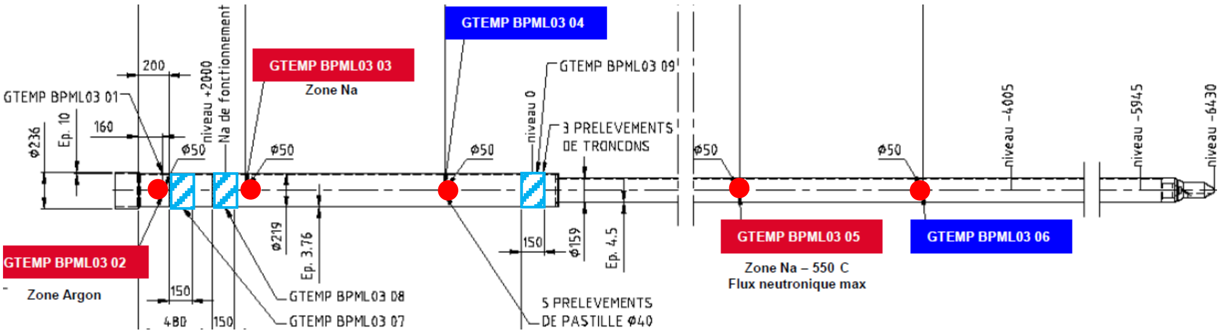

*FIG. 3. Identification of the CPML0373 rod sampling points for the ASTRID project (red dots = chemical compatibility studies, blue hatched areas = mechanical property studies).*

Before decontamination, five samples were identified (red dots, *FIG. 3, TABLE III*) as relevant for the chemical compatibility studies. After macrographic observations in order to have a general overview, GTEMP recommends all samples be observed with a FEG-SEM device in order to identify the morphology of the corrosion concerning the outside and the inside of the rod. The composition of each phase should next be consolidated by semi-quantitative analysis with EDX and by XRD measurements if the expert laboratory considers this useful.

After decontamination, the samples identified for the mechanical studies are shown on the figure above with the blue hatched areas (*FIG. 3, TABLE IV*). Sample #7 was cut first in order to identify the best investigation area for sample #9. Samples # 8, 9 and # 10 were cut afterwards in the thinnest area, respectively concerning the sample for cold area reference, the sample to be investigated and the hot area reference.

*TABLE III. BEFORE DECONTAMINATION, ROD SAMPLING FOR CHEMICAL COMPATIBILITY STUDIES (RED DOTS, FIG. 3)*

| **Location** | **Type of sample (mm)** | **Sample #**<br>**GTEMP-CPML03-** | **Environment** |
|---|---|---|---|
| *Upper part*<br>*304L*<br>*(219 mm diameter)* | Ø 40, THK ~ 3.76 | **02** | Ar gas at 400°C |
| | | **03** | Sodium free level zone :<br>Na / Steel / Gas |
| | | **04** | Liquid Na |
| *Lower part*<br>*316L*<br>*(159 mm diameter)* | Ø 40, THK ~ 4.5 | **05** | Liquid Na 550°C |
| | | **06** | Liquid Na 400°C |

*TABLE IV. AFTER DECONTAMINATION, ROD SAMPLING FOR MECHANICAL PROPERTY STUDIES (BLUE HATCHED ZONE, FIG. 3).*

| **Location** | **Type of sample (mm)** | **Sample # GTEMP-CPML03-XX** | **Environment / observation** |
|---|---|---|---|
| *Upper part*<br>*(219 mm diameter)*<br>*304 L* | Rectangle 2300 long and 50 width | **07** | To identify location for “sample #09” with maximum deformation |
| | Tube section Ø 219<br>L = 150 | **08** | Reference sample at ~400°C (Ar cover gas level) |
| | | **09** | Deformed sample at Na operation Level with (located with measurements on “sample # 07”) |
| | | **10** | Reference sample at ~550°C (liquid Na level) |

## 4.3. Implementation of the rod investigation program in collaboration with the dismantling teams

### 4.3.1. Sampling before decontamination

The rod had been removed from the reactor core vessel using a handling hood (*FIG. 4*). Between 2001 and 2012, it remained in a storage pit (*FIG. 5*) [8]. Once the rod was removed from the reactor, it was transferred into a special cell for the heat insulating jacket to be removed. A radiological mapping of the component was carried out before the washing process to check the possibility of pre-decontamination sampling. Dose rate and contamination results enabled the rod sampling to be performed in 2012 without washing or decontaminating its surface (*FIG. 6*).

*FIG. 4. Extraction of the rod.*

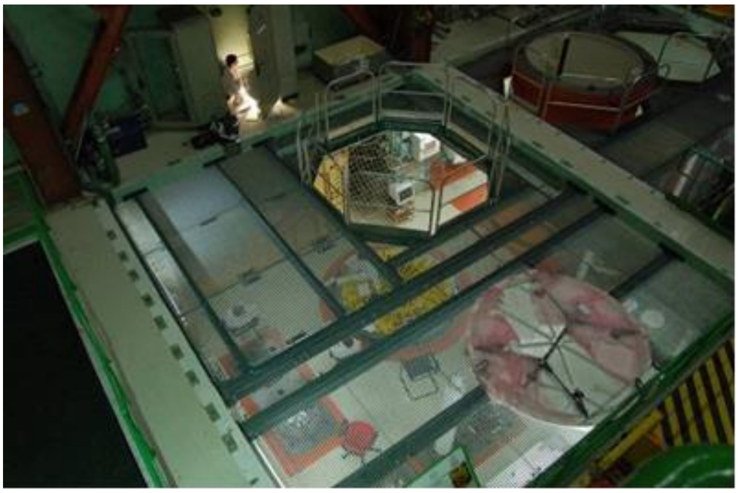

*FIG. 5. Storage pits.*

*FIG. 6. Sampling in nuclear suit in the work airlock, example of a sample before decontamination (sample #GTEMP-CPML0373-02, red dots, FIG.3).*

### 4.3.2. Sampling after decontamination

Once the samples were taken, the component was transferred to washing pits in order to remove sodium residues. Decontamination baths also helped to significantly reduce the contamination and therefore the surface activity. Sulfuric and phosphoric acids were used, and several decontamination cycles were performed. The component was delivered to the handling hall and tilted using a crane and a specially-designed tilter. After being tilted, the component was inserted into the work airlock and cut with a wire saw (*FIG. 7-8*).

*FIG. 7. Work airlock used to sample the rod (views from outside).*

*FIG. 8. After decontamination: view inside the work airlock during sampling using a wire saw for mechanical property studies (blue hatched zone, FIG 3.).*

Samples #2-6 were transferred to LMAC laboratory in CEA Marcoule for chemical compatibility studies, while samples #8-10 were shipped to LECI laboratory in CEA Saclay for mechanical property investigations.

The LMAC study results are currently being interpreted by the GTEMP experts and will be the subject of a future publication. The data obtained from the Phénix operating environment samples will be compared with CEA laboratory-scale R&D experimental results

## 5. Conclusion

The dismantling of Phénix components since 2009 has enabled a unique non-destructive and destructive examination of real components to be carried out. This is an opportunity to obtain design data and fundamental understanding for a robust demonstration of ASTRID. Sampling from a reactor undergoing dismantling after thirty five years of operation is a highly technical task and needs a good overall knowledge of the component studied.

In 2008, a CEA-AREVA-EDF working group GTEMP being preparing investigation guide sheets, key technical information enabling successful sampling and later investigations. They describe the expert investigation methodology to be implemented for Phénix NPP components, including the rod described here. The highly technical information included for example (a) Capitalization on historical data of the component, (b) Analysis of the data by the GTEMP expert group, (c) Proposition of an investigation program, (d) Implementation of the sampling program to be carried out during Phénix dismantling, (e) Carrying out expertise assessments, (f) Interpretations by the GTEMP group, and (g) Transfer of the expertise results/interpretations as input data for Astrid and for R&D working groups. The experts highlighted the great interest for future Gen IV reactors of some of Phénix NPP components. In the case of the rod, the objectives included studying chemical compatibility in liquid sodium and studying some mechanical properties of 304L and 316L steels after twelve years in full power operation.

In 2012, the Phénix operation teams began dismantling the CPML0373 rod. Thanks to the preliminary work carried out by the GTEMP, coupons were successfully sampled from the rod, the fruit of an effort shared by all the Phénix plant teams. The samples were transferred to the host laboratories (LMAC, CEA Marcoule and LECI, CEA Saclay) in order to be characterized using SEM, XRD. The first results showed that the sampling approach without prior decontamination had enabled all the characterization techniques planned to be implemented. The rod project is demonstrating the feasibility of R&D groups working in cooperation with dismantling teams in order to obtain feedback relevant for future reactors.

After the successful sampling from the Phénix NPP rod, the objective now is to carry out characterizations and compare the results with the literature data available. A detailed comparison will be made in order to attempt to link the sample microstructural observations with the reactor rod operating phases and its environmental exposure conditions (liquid sodium during operation from 1974 to 2001, and inert gas during storage 2001-2012). The final objective will be to be able to discriminate among the types of damage observed and characterized by the expert laboratories, depending on the different environments. For example, the formation of chromite can be encountered, but is usually difficult to characterize, together with dissolution of the steel that leads to the formation of a ferrite layer, or the carburization-decarburization phenomena that are some of the corrosion mechanisms that could be observed after a long residence time in sodium [9-10-11].

Carrying out examinations of real components is a unique opportunity to obtain design data, fundamental understanding and material damage data for the qualification of the ASTRID project. These data will be useful in terms of the design rules RCC-MRx code but also to justify an extended 60 year lifetime for such reactors.

## Acknowledgements

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