

SPIDER Cs Ovens functional tests

M. De Muri^{1*}, A. Rizzolo¹, E. Sartori¹, S. Cristofaro², M. Barbisan¹, M. Fadone¹, D. Ravarotto¹, R. Rizzieri¹, R. Capobianco¹, P. Cinetto¹, B. Laterza¹, F. Rossetto¹, M. Rancan³, L. Armelao³, C. Taliercio¹, G. Serianni¹, D. Marcuzzi¹

¹ *Consorzio RFX, corso Stati Uniti 4, 35127 Padova, Italy*

² *Max-Planck-Institute for Plasma Physics, Boltzmannstr. 2, 85748 Garching, Germany*

³ *Institute of Condensed Matter Chemistry and Technologies for Energy (ICMATE), National Research Council (CNR), c/o Department of Chemical Sciences, University of Padova, via Marzolo 1, 35131 Padova, Italy*

**Corresponding author: michela.demuri@igi.cnr.it*

The SPIDER experiment is operating at the ITER Neutral Beam Test Facility PRIMA since June 2018 to study the operation of a full ITER-size negative ion source. In order to increase the negative ion extracted current and reduce the co-extracted electron current, caesium is evaporated inside the source. In fact, the adsorption of Cs onto the plasma grid, i.e. the first grid of the extraction system, lowers the surface work function, enhancing the probability of surface conversion of impinging hydrogen particles into negative ions. At the SPIDER test facility, Cs will be evaporated by heating a liquid Cs reservoir in three Cs ovens located at the back-plate of the ion source. The ovens must assure a controllable and reproducible evaporation. A SPIDER Cs oven prototype was procured and tested in the CAesium Test Stand (CATS) at PRIMA site to validate the design. Following the experimental tests, few improvements were introduced in the final design of the three Cs ovens for the SPIDER source. Then, the ovens were procured in 2019 and commissioned before their installation in SPIDER in 2020. This paper reports the functional tests of the SPIDER Cs ovens and of their embedded diagnostics. These tests were carried out at CATS, with the aim of characterizing the three ovens, in order to validate them for their use in SPIDER in the experimental campaign with caesium evaporation planned for the end of 2020. The ovens were successfully tested and characterized in terms of evaporation rate for different oven temperatures. Moreover, the three ovens show a good reproducibility and reliability.

Keywords: SPIDER, Beam Source, Cs evaporation, MITICA, PRIMA, Cs Oven functional tests, CATS test stand

1. Introduction

Negative ion sources [1] for the neutral beam injection system of the fusion experiment ITER (Cadarache, France) rely on the surface conversion of atomic hydrogen (or deuterium) and positive hydrogen (or deuterium) ions into negative ions at a low work function surface [2], namely the first grid of the extraction system (plasma grid). To lower the surface work function and enhance the negative ion yield, caesium is evaporated inside the source [3] by means of temperature controlled Cs ovens. The experiment SPIDER (Source for Production of Ions of Deuterium Extracted from RF Plasma) at the ITER Neutral Beam Test Facility (NBTF) at PRIMA (Padova Research on ITER Megavolt Accelerator) is equipped with a full ITER-size negative ion source and started operation in 2018 [4]. SPIDER accommodates three liquid Cs ovens [5] located at the back-plate of the ion source, and first Cs operation is foreseen for the end of 2020. A prototype of the ovens was first tested at the dedicated laboratory experiment CATS (CAesium Test Stand) at the PRIMA site [6]. The three Cs ovens were then procured in 2019 and were recently tested at CATS to verify their thermal behavior and to characterise the Cs emission as a function of the oven temperature, checking the system reliability, reproducibility and stability in view of their use in the SPIDER beam source. First investigations on one of the three ovens have shown reliable and stable Cs

evaporation [7]. In the present contribution, the performances of all the three Cs ovens are presented, including a comparison among the three ovens, the determination of the evaporation rates for different oven temperatures and the identification of proper operating procedures to be adopted in the SPIDER experiment.

2. Functional Tests

Figure 1 shows a CAD section view of the SPIDER Cs ovens, highlighting the position of the thermocouples (TC). The oven consists of a liquid Cs reservoir, a solenoid valve, an injection pipe and the nozzle. The valve has a mobile plunger which is operated by a custom solenoid, composed of an aluminium spool and a winding of at least 550 turns (nickel plated copper wire) with pure fibreglass insulation without impregnation; the magnetic circuit is made from pure iron, and the gasket of the custom plunger is made of Kalrez® 8900. This design allows to seal the caesium in the reservoir during the installation of the oven, and it is compatible with temperatures up to 350°C required for the operation. The thermocouples TC1 and TC4 control the heating of the Cs reservoir and of the injection tube, respectively. The oven nozzle is equipped with a Surface Ionisation Detector (SID) to monitor the Cs evaporation and to determine the evaporation rate [8, 9]. Several diagnostics are available at the CATS experiment [10] to study the

Cs evaporation: a laser absorption spectroscopy (LAS) system is installed to measure the line-of-sight integrated density of atomic Cs inside the evaporation chamber of CATS [11], [11] the vessel SID is located close to the wall of CATS and allows determining the flux of Cs redistributed in the chamber. Additionally a Residual Gas Analyser (RGA) is available and connected to the experiment. Several systematic tests and investigations were planned to study and characterize the Cs ovens behavior, aiming also to a comparison among the ovens. The following tests were performed:

1. Geometrical characterization of the oven SID.
2. Electrical characterization of the oven SID.
3. RGA records performed at the beginning of the experiment (before test 2) and after 1 day of oven heating at 250°C on the injection tube aimed at cleaning the oven, with the oven valve closed and before any Cs evaporation.
4. Thermal test.
5. Study of Cs evaporation.
6. Steady state test for long-term operation (only for oven 01).
7. Air exposure test and N₂ venting (only for ovens 01 and 02).
8. Restart of the Cs evaporation (only for ovens 01 and 02).

The testing schedule was optimized to incorporate more purposes in the same tests, to reduce the time necessary for testing each single oven. Moreover, at the end of the experimental campaigns, an inspection of the ovens was performed. Additionally, before the investigations on the last oven (Oven 03) the CATS vacuum vessel was cleaned.

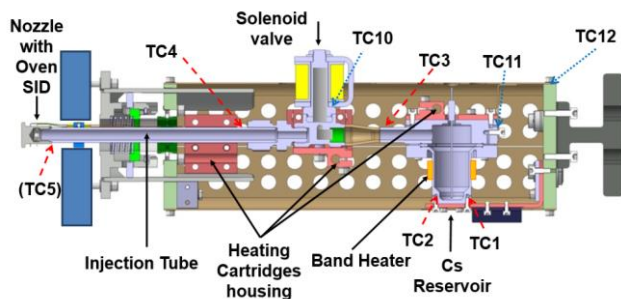


Figure 1 – Section view of SPIDER Cs ovens: thermocouple layout, permanent TC with red dashed arrows, auxiliary TC with blue dotted arrows, and Oven SID are shown.

The purposes and procedures of the different investigations are detailed hereafter, together with some preliminary results:

1. Geometrical characterisation: since the geometry of the SID filaments influences the measurements [9], it is important to know precisely these characteristics performing geometrical measures on the filaments. These measures were performed before the first use, reporting the geometry just after the realisation and installation of the filaments on board of the ovens, and after their use during the experimental campaign, to examine if distortions occurred due to heating and/or the heating/cooling cycles.

2. Electrical characterisation: this test regards the voltage/current characteristic and the Richardson emission to estimate the work function of the ioniser and collector filaments of the SIDs (4.6 eV for ionizer of Oven 02). This test was done with the valve closed and before any Cs evaporation.

3. RGA: in both cases of Oven 01 and Oven 02 (data of Oven 03 are not yet available), air and water were detected during the first heating of the oven with valve closed, but also mass 17 and 28 AMU could be identified, probably due to the use of a lubricant in the bayonet coupling, to facilitate the installation of the ovens inside CATS. As expected, the heating of the injection tube and the SID filaments increased the outgassing but no changes in the composition of the detected vacuum environment could be appreciated by RGA, hence an increase of the masses attributable to air and water was observed. The gas coming out from the inner volume of the reservoir during the opening of the valve caused an overpressure condition inside the vessel and the RGA acquisition was automatically stopped to protect the RGA. In this way, no RGA signal was recorded during the valve opening and it was not possible to evaluate if Ar was released in the vessel (since the Cs reservoir was filled in an Ar glove box).

4. Thermal tests were carried out varying the injection tube and reservoir set temperatures (Table 1), in order to: a) map for each oven the temperature distributions along the oven itself; b) demonstrate the repeatability of the temperature distributions; 3) make comparisons among the ovens. An example of temperature distributions is shown in Figure 2 for Oven 01 at 200°C on the injection tube and 130, 120 and 110°C of the reservoir. The temperature distribution along the oven allows to avoid Cs condensation points from the reservoir to the nozzle.

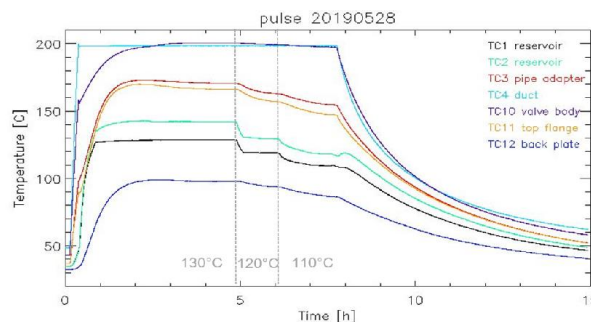


Figure 2 – Oven 01 – TD=200°C, TR=130/120/110°C

5. Study of Cs evaporation: the proportionality of the oven SID signal on the total Cs emission depends on the intercepted Cs vapour fraction and therefore on the SID geometry, which was determined with test 1. However, distortion of the filaments can occur during the operation, hence the Cs emission is studied by means not only of the oven SID, but also with the LAS diagnostic and the movable SID, so to distinguish between a real difference in Cs emission by the ovens or a Cs signal variation due to a distortion of the SID filament and so to the intercepted Cs fraction. These investigations were carried out for all the ovens for the temperature set of the injection tube and the reservoir shown in Table 1. This

investigation was aimed at: a) verifying the Cs flux dependence on the temperature of the reservoir; b) calibrating the oven SID also by correlating oven SID measurements with the ones from other diagnostics; c) verifying the Cs consumption; d) finding and verifying a map of Cs emission rates with respect to the reservoir temperature to be used in SPIDER. The results of these investigations are shown in section 3. In particular, it has to be noted that the procedures applied to Oven 01 and 02 (see tests 7 and 8 of this section) may affect the measurements, for this reason the Oven 03 was tested with a different procedure to guarantee the repeatability and accuracy (see Section 3.1 and 3.4).

T ref (TC4) injection tube [°C]	200	200	200	200	250	250	250	250
T ref (TC1) reservoir [°C]	100	120	130	140	130	140	150	160

Table 1 – Set of temperatures of the injection tube and of the reservoir applied for the current investigations on the Cs evaporation.

6. Steady state for long operation: this test, performed only for the Oven 01, was aimed to: a) verify the stability of Cs emission during several days; b) verify the correct behaviour of the solenoid valve after a large amount of Cs evaporated through the valve body; c) verify the correct behaviour of the SID after large amount of Cs deposited on the nozzle. The test was done at high temperature (injection tube at 250°C and reservoir at 140°C) to evaporate a conspicuous amount of Cs and to get confidence with the oven behaviour in preparation of the long experimental campaigns in SPIDER, without vacuum break and maintenance. This test was lasting 4 days during which the valve was left closed during the night and opened during whole working day. After the 4 days of steady state operation, pulse repetition was performed to observe the repeatability of the oven behaviour: the results showed that there is a good reproducibility of the measurements after the long term evaporation.

7. Air exposure test and N₂ venting: this test was carried out differently on the three Cs ovens. For Oven 01, after the previous tests, the CATS vessel was vented in nitrogen (as it will be in SPIDER) with the solenoid valve open. Then, CATS vessel was evacuated again to verify the performance of the oven after the reservoir being in contact with nitrogen (with possible reaction between Cs and N₂) and to check Cs emission repeatability and the solenoid valve functionality. At the restart of the Cs evaporation after N₂ venting, the signal from the oven SID was reproducible if compared with the previous measurements (test 4), indicating that the exposure of the reservoir to nitrogen does not affect to a great extent the evaporation of Cs from the oven. However, the Cs density from LAS and the SID current from the movable SID are slightly lower but comparable to the previous measurements. This can be due to the fact that both these diagnostics, aimed to characterize the evaporation chamber conditions and not the Cs emission rate, depend on the wall conditions, so a slight decrease of these signals could be expected. Then, Oven 01 was exposed to air for 22 days, with N₂ inside the reservoir and the valve closed. Finally, Oven 01 was installed in

CATS to perform Cs evaporation again after the break. In this way it was possible to evaluate the effect of diffusion of air inside the reservoir (details on the following point 8). Oven 02 was not subjected to N₂ venting, but it was stocked in air with vacuum inside the reservoir for 11 days and valve closed. Different procedures were adopted for Oven 01 and Oven 02 in order to choose the proper procedure for SPIDER. This test was not carried out on Oven 03: at the end of the calibration of Cs evaporation, the vessel was vented in Argon, with the valve open. Before the opening of the vessel, the valve was closed. Once the oven was extracted from the vacuum, the oven was put inside the Argon glove box (no air exposure test), ready for the inspection.

8. Restart of Cs evaporation. Regarding Oven 01, Cs density and SID currents measurements after air exposure showed a sudden drop down of the performance: all the signals decreased of at least one order of magnitude. In particular, the decrease of the oven SID current (directly correlated to the evaporation rate of the oven) showed that the oven did not deliver to the system the same amount of Cs for the same reservoir temperature as before air exposure; to achieve the same evaporation rates much higher temperatures were needed. The decrease of the performance, and the following oven inspection, suggest the formation of large crystals inside the oven might cause this. This has two implications, a depletion of pure Cs in the oven reservoir, and that the Cs flow is not necessarily conserved from reservoir to the nozzle, as it is consumed by the crystal growth along the oven. Regarding Oven 02, it was possible to evaluate the effect of diffusion of air inside the reservoir (initially under vacuum) by keeping the oven outside the glove box. Slightly lower movable SID currents were observed after air exposure, together with much lower Cs densities. Additionally, the Cs density seemed to saturate at high temperatures, not following anymore an exponential law (as usually observed by changing the temperature, see section 3). However, contrarily to the experience with the Oven 01, the oven SID currents measured after air exposure were reproducible with the ones measured before air exposure. This suggests that in this case the measurements from the movable SID and TDLAS were strongly affected by a loss of conditioning of the walls rather than a decreased Cs evaporation from the oven and the depletion of the Cs reservoir was not so severe, as it was confirmed by the following oven inspection (see section 3).

3. Discussion about main results

3.1 Determination of the evaporation rate

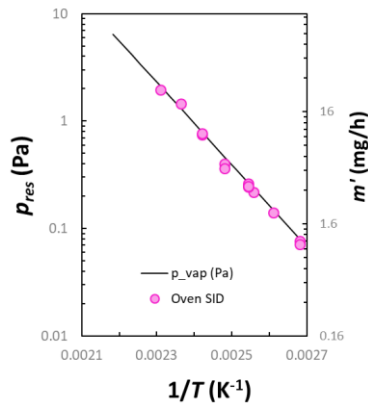


Figure 3 – Oven 03: vapor pressure obtained by analytical calculations (straight line) and experimental evaporation rate (circle markers) as functions of the reciprocal reservoir temperature.

During the investigations on the Cs evaporation for different reservoir temperatures, the evaporation rate was determined by means of the oven SID via the procedure described in [9] and after the geometrical characterization of the oven SID (test 1). Figure 3 shows an Arrhenius plot of the estimated evaporation rate of the Oven 03 at different reservoir temperatures (circle markers). The black straight line provides the calculation of the Cs vapour pressure for varying reservoir temperatures. The superimposed experimental points behave in line with the prediction based on a constant conductance of the injection tube of the oven. The cleaning of the vessel had an important effect on these measurements, appreciated on Oven 03 by the good fitting shown in Figure 3. As an example, the same graph regarding Oven 02 is reported in Figure 4 and did not show a so good agreement: as experimental days go by, the SID detects a higher current and consequently a higher evaporation rate is estimated. The arrow in Figure 4 roughly indicates the succession of the experimental days. Probably this behavior is due to other effects linked to Cs redistribution.

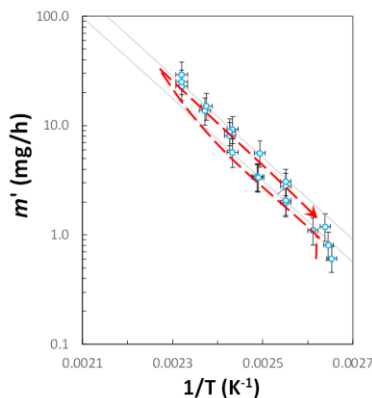


Figure 4 – Oven 02: vapor pressure obtained by analytical calculations (straight gray line) and experimental evaporation rate (circle markers) as functions of the reciprocal reservoir temperature.

3.2 Comparison and reproducibility of the ovens performance

Figure 5, Figure 6 and Figure 7 show a comparison of the performance of the three ovens in terms of Cs density (measured with the laser diagnostic) and in terms of SID currents for different reservoir temperatures, respectively. The first day of operation (highlighted in all these figures) shows lower signals, especially in terms of Cs density. This can be due to the fact that the conditioning of the CATS chamber requires some time in order to reach equilibrium of Cs adsorption and desorption at the walls. For increasing reservoir temperature, all the signals increase accordingly. Some campaigns were repeated in order to check the reproducibility of the signals, in particular of the oven SID current since it is not dependent on the conditioning of the walls but only on the evaporation rate of the oven (assuming a stable shape of the SID filaments during the experiments). As it can be seen in the figures, the data collected at the same reservoir temperature are in good agreement and the three ovens show very reproducible performance in terms of SID currents. The Cs density is much affected by the conditioning of the walls and by the measurement error, which increases at high reservoir temperature and explains the increased scattering of the data points. Another seen effect due to the cleaning is on the sticking coefficient of the vessel walls. This coefficient can be thought as the probability of a Cs atom to be captured by a wall after impact. In [12], this coefficient has been estimated through a comparison between a numerical model and experimental data obtained thanks to the LAS and the movable SID. With the oven prototype, a sticking coefficient value of 2% has been found, while for the third oven the value increased to 4%.

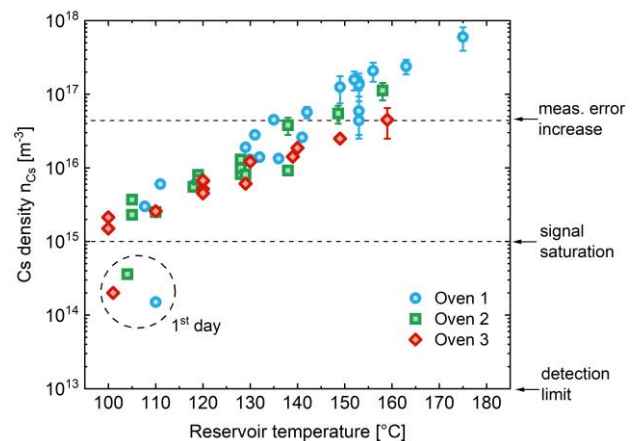


Figure 5 – Ovens comparison: Cs density from laser absorption spectroscopy for varying reservoir temperature.

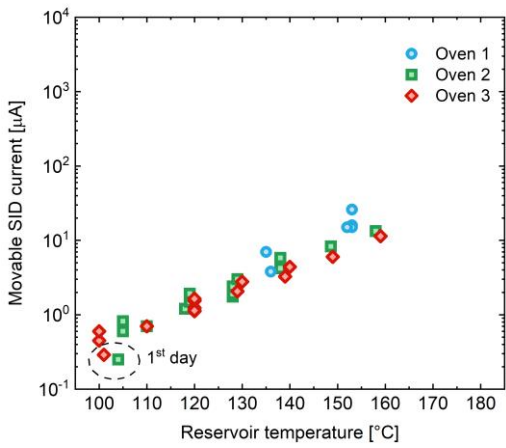


Figure 6 – Ovens comparison: movable SID current for varying reservoir temperature.

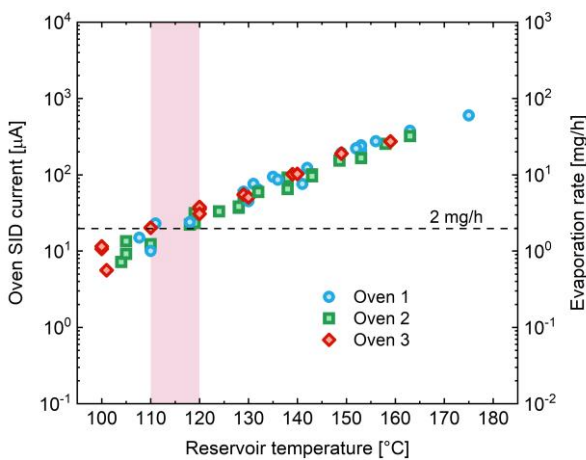


Figure 7 – Ovens comparison: oven SID current for varying reservoir temperature. The reference value of 2 mg/h [7] is also indicated, defining the operational temperature range (pink area) for application in SPIDER.

From the estimation of the evaporation rate by means of the oven SID, the current reference value of 2 mg/h per oven suggested for RF ITER-relevant ion sources [7] is achieved at a reservoir temperature between 110°C and 120°C.

3.3 Final inspection of the Cs reservoirs

The results of the final inspection performed in the argon glove box are reported in the following for each of the three ovens. The Cs residuals found in the reservoir of the Oven 01 are shown in Figure 8. All metallic Cs is consumed and almost no black/contaminated residuals are visible on the bottom of the reservoir. Nevertheless, Cs white residuals are visible on the CF interface flange (closure of the reservoir), on the valve body and on the plunger of the valve.

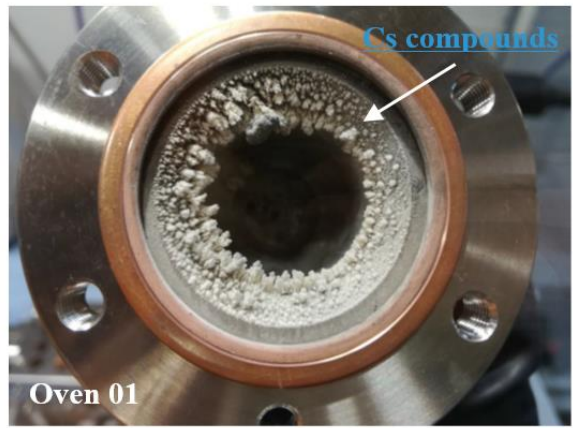


Figure 8 – Final inspection of the reservoir of the Oven 01.

The gasket of the valve plunger was hardened, difficult to be extracted from the plunger and its surface looked eroded or with a deposit over the exposed surface.

Figure 9 shows the reservoir of the Oven 02, where white compounds are visible in the upper part and some liquid Cs is still visible in the bottom part. Few Cs residuals were also visible on the valve body and on the plunger. The plunger gasket looked eroded.

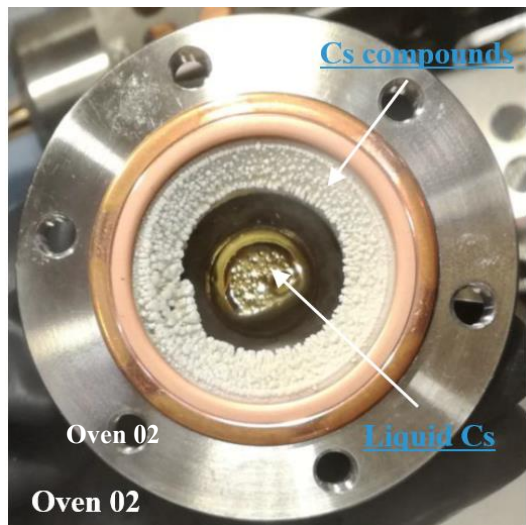


Figure 9 – Final inspection of the reservoir of the Oven 02.

Figure 10 shows the reservoir of the Oven 03, where only some liquid Cs is visible on the bottom part. No Cs residuals were found on the CF interface flange (closure of the reservoir), neither on the valve body nor on the plunger. This suggests that the formation of the Cs compounds for the Oven 01 and 02 took place during the air exposure test after their usage, while the Cs reservoir and valve were preserved for Oven 03 since there was no air exposure for this oven.

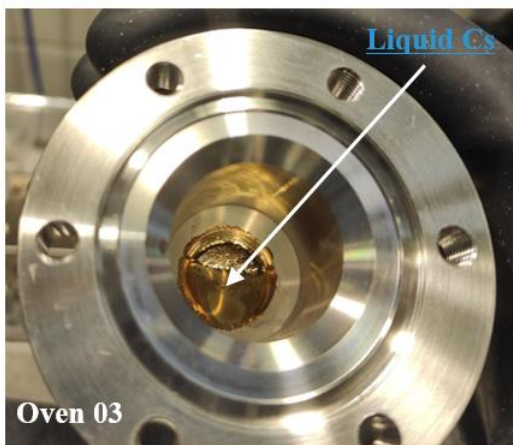


Figure 10 – Final inspection of the reservoir of the Oven 03.

With the aim to understand the nature of the contaminated residuals, and to confirm how and when they were formed, an investigation with X-ray Photoelectron Spectroscopy (XPS) was carried out at the ICMATE laboratories. Samples of Cs residual coming from the bottom and from the upper part of the Oven 02 reservoir were studied. The data analysis involved Shirley-type background subtraction, non-linear least-squares curve fitting adopting Gaussian-Lorentzian peak shapes, and peak area determination by integration [13]. The atomic composition was evaluated from peak areas using sensitivity factors supplied by Perkin-Elmer, taking into account the geometric configuration of the apparatus [14]. XPS survey showed the presence of the following elements: C, O and Cs. High resolution spectra have been collected for the regions corresponding to the transition C1s, O1s and Cs5d. The following considerations can be drawn: C1s clearly shows the presence of two different contributions; C1s region has been fitted with two components ascribed to C-C/C-H bonds (284.8 eV), and C=O bonds (aldehyde/ketone, 287.7 eV) both deriving from adventitious hydrocarbons [14]. O1s peaks have been fitted with two components: the most intense peak (531.4 eV) is ascribed to hydroxyl oxygen [14] while the less intense peak at higher binding energies (533.9 eV) is ascribed to oxygen atoms of adsorbed water [14]. Cs3d region is a multiplet with a well separated spin-orbit components ($\Delta BE = 13.9$ eV). The binding energy of the Cs3d_{5/2} peak (724.4 eV) is in agreement with a CsOH environment. The atomic percent of the elements and the binding energies show that the samples had similar composition and chemical nature. In particular, the ratio Cs:OH is close to 1 as expected for CsOH. The hypothesis that the analysed samples are mainly CsOH is also supported by the visual inspection and chemical behaviour of the powders. In fact, samples are white powders highly hygroscopic that readily deliquesce when exposed to air to form a basic solution (pH \approx 12-14). The deliquescent samples come back to white solid powders when evacuated in vacuum.

3.4 Estimation of Cs consumption

Oven	Initial Cs	Residual Cs from	Cs consumption	Cs evaporation
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	load	final weight	from weighing	from oven SID
01	2 g	0.4 g	1.6 g	0.9 g
02	1 g	0.74 g	0.26 g	0.3 g
03	1 g	0.85 g	0.15 g	0.16 g

Table 2 – Estimation of Cs consumption from nozzle SID data and weighing of the reservoir.

At the end of the experiment, inside the glove box, the reservoirs were weighed to estimate the Cs consumption: the remaining Cs amount was calculated by subtracting the weight of the clean reservoir to the weight of the reservoir dismantled just after the end of the experiments (Figure 8 - Figure 10). Table 2 shows the comparison between the estimation of evaporated Cs amount, obtained by integrating over time the evaporation rate that oven SID provided and obtained by the final weighing procedure. The differences on Oven 01 and Oven 02 results can be explained considering the presence of Cs residuals on several components along the oven that could not be weighed. As said before, slightly different conditions and procedure were applied to Oven 03: the CATS vacuum vessel was cleaned only before the investigations of this oven, the air exposure test was not performed and the ventilation of the valve was done with Ar instead of N₂. The agreement on the results of the Oven 03 can be explained by these considerations. The results of Oven 03 are the most indicative of the dependence of the emission rate on the reservoir temperature.

3.5 Evaporation chamber conditions

The conditions inside the evaporation chamber for the Oven 03, were studied both during evaporations and in between evaporations (oven hot but valve closed). A direct dependence of the Cs density over the oven emission rate was found as shown in Figure 11 top: the ratio was rather constant despite the increasing amount of Cs cumulated in the chamber. The effect of the Cs adsorbed to the wall was studied by closing the valve and measuring the density: Figure 11 bottom shows how the Cs density increases with the increasing of the total evaporated amount. LAS and vessel SID are in rather good agreement. The results of Figure 11 top are a further confirmation of the importance of a clean evaporation environment also for the characterization of the Cs ovens.

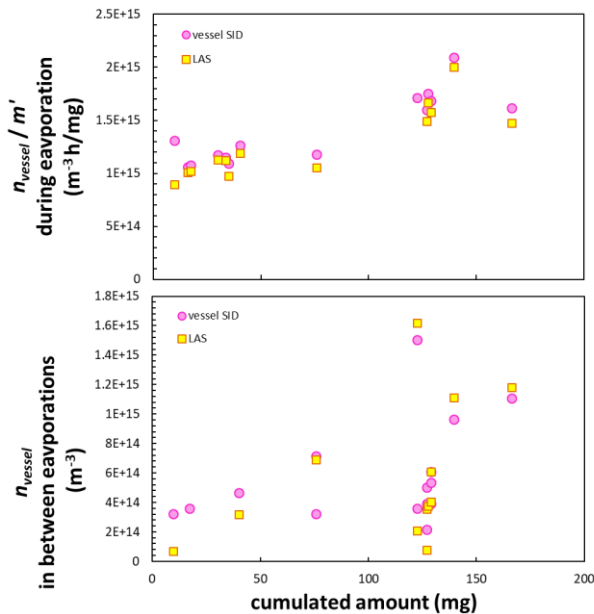


Figure 11 – Evaporation chamber condition for the Oven 03: ratio between Cs density and emission rate during evaporations (top) and Cs density and in between evaporations (bottom).

3.6 Issues on the valve of Oven 01

In two cases, the valve of the Oven 01 did not work properly, and the valve was stuck in closed position. These events however occurred in particular conditions: (a) after 4 days of steady state for long-term operation and (b) after N_2 venting and air exposure. In the first case, the large amount of Cs evaporated could have soiled the valve body. In this case, it was sufficient to increase the temperature of the injection tube up to 300°C (maximum allowed temperature to avoid damage on the plunger gasket): the valve could open again and no other sticking events occurred in the successive experimental days. In the second case, the valve sticking could be linked to N_2 venting and/or air exposure together with the large amount of Cs evaporated and deposited on the valve body. To solve this issue, the opening current on the solenoid of the valve was increased. Once the valve was unlocked, it could open again at the usual opening current. It should be noted that the plunger was never blocked in open position (this occurred during previous R&D tests on the prototype oven), thanks to the introduction of grooves at the rear side of the plunger.

4. Conclusions

The main important outcome of the reported functional tests on the three SPIDER Cs ovens is that a very similar behavior among the three ovens was verified.

In the first day of the oven operation, the ovens have a lower performance, so a relatively long time (about one experimental day) is necessary before reaching stability.

The oven SID signal gives important feedback on the Cs consumption, since it gives an estimation of the Cs evaporation rate. If the SID shape does not change during the oven operation, the SID signal can be directly correlated to the evaporation rate and an estimate of the Cs consumption can be done.

The reservoir temperature controls the Cs evaporation, while the temperature of the injection tube has no or very low influence on the emission rate. On the other hand, it is essential that the tube temperature is kept higher than the reservoir temperature to avoid condensation.

The solenoid valve is able to interrupt the Cs flux well and fastly, but if damage occur on the plunger gasket (as per Oven 01 and 02), the valve reduces its vacuum tightness but not such as to compromise the functionality of the ovens.

The steady state test for long term operation (Oven 01 only) demonstrated a very stable Cs evaporation and good performance of the oven: during the 4 days the oven SID signals were comparable with the ones of the first evaporation, LAS and movable SID signals were instead affected by the wall conditions. The large amount of Cs created problems to the oven SID: electrical leakages with respect to oven ground occurred but careful cleaning of the nozzle and SID with distilled water and fibre glass pencil improved the isolation.

After the venting of the vessel with oven valve open (only Oven 01), the signal from the oven SID was reproducible if compared with the previous measurements, indicating that the exposure of the reservoir to nitrogen does not affect the evaporation of Cs from the oven.

Long exposure to air should be avoided, otherwise the oven needs to be cleaned and refilled with new Cs.

Clean conditions while managing the Cs during the filling/refilling and while managing the ovens shall be guaranteed to avoid Cs contamination.

Thermal cycles caused the loosening on some screws of the ovens but the functionality is not compromised as soon as the oven is kept in vacuum.

During the functional tests, a few upgrades have been added to the ovens. The use of a custom piezoelectric accelerometer on the valve has been demonstrated to be effective for detecting the valve actuation, thus all the ovens have been equipped with an accelerometer.

F4E and ITER Disclaimers

The work leading to this publication has been funded partially by Fusion for Energy. This publication reflects the views only of the authors, and F4E cannot be held responsible for any use which may be made of the information contained therein. The views and opinions expressed herein do not necessarily reflect those of the ITER Organization.

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