

New Approach to meet vacuum requirements in UHV/XHV systems by Non Evaporable Getter Technology

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Abstract. UHV/XHV users are increasingly demanding vacuum pumps with high performances with smaller package and weight. This requires a more efficient combination/integration of the currently available pumping technologies and, possibly, the development of new approaches in vacuum pumping systems. Non Evaporable Getter (NEG) pumps represent one of the most appealing option to UHV/XHV pumping, thanks to his large pumping speed and sorption capacity for several active gases (H₂, H₂O, CO, O₂, N₂, ...). NEG pumps can also reduce the pump down and bake-out time and can keep a stable pressure level in UHV conditions even if the other pumping systems are switched off. However, NEG pumps are unable to sorb, in UHV/XHV systems, the small amount of noble gases and methane which, on the other hand, can be removed by sputter ion pump (SIP). For this reason, extensive studies have been carried out to combine NEG and SIP technologies. Some results of the vacuum characterization of such combination are discussed in this work and compared with traditional pumping approaches based on large SIP. A further step forward is the NEXTorr^{®3} pump which is an integration of a NEG pump (pumping speed ranging between 100 to 500 l/s for hydrogen) with a SIP (pumping speed of 6 and 10 l/s for Ar and CH₄ respectively) into a single small package unit. Examples of applications of this new approach in vacuum technology will be given to demonstrate the simplification of the design and operation of UHV/XHV systems.

1. Introduction

The design and operation of UHV/XHV systems requires the development of vacuum groups able to deliver higher pumping performances with a smaller package. In this framework, Non Evaporable Getter (NEG) pumps can satisfy these requirements due to the ability to chemically and permanently remove active gases in vacuum system [1]. However NEG pumps are unable to adsorb methane and noble gases and for this reason they are used together with sputter ion pump (SIP) [2]. The classical approach is to combine a large SIP with a compact NEG pump of comparable pumping speed. However, the analysis of the residual gases in UHV systems shows that hydrogen is the main gas, while methane and other noble gases are several orders of magnitude smaller. Therefore the most efficient approach to achieve UHV condition may be based on the use of high efficiency pumps to remove hydrogen backed by smaller pumping systems to remove the small amount of methane or noble gases. The use of performing pumping system to remove hydrogen could bring additional

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benefits into the UHV system. In fact if NEG pumps are activated and heated at moderated temperature during the bake out process, a reduction of either the baking time or the baking temperature can be achieved, obtaining even better pressure level at the end of the process in a shorter time [3].

In this framework, recently a new combination pump has been developed: the NEXTor[®] pump. The NEXTor[®] is the combination of a NEG pump (100÷500) l/s for hydrogen with a 5 l/s (N₂) SIP pumping speed.

A critical overview of the pumping performances obtained by using several types of SIP and NEG pumps is presented. In addition, a characterization of the pump down properties of NEXTor[®] will be given and compared with SIP.

2. Experimental details

Tests 1 and 2: the tests were carried out on an 8.8 l volume AISI 304 steel chamber having a total inner surface area of 3000 cm². The chamber was fitted with an extractor gauge (detection limit is 10⁻¹² mbar) and was leak checked down to 10⁻¹⁰ mbar l / s using a helium leak detector. The extractor gauge and the RGA were calibrated using a spinning rotor gauge. Primary vacuum was achieved by means of a 60 l/s (N₂) turbomolecular pump (TMP) backed by a 1.8 l/s scroll pump. In test 1, a 60 l/s (N₂) SIP was connected to the bottom flange of the chamber and the NEG pump (Capacitor^{®4} D 400-2) was integrated into the SIP. In test 2, the 60 l/s SIP was replaced with 10 l/s SIP fitted directly to the test chamber. The NEG pump was also connected to the chamber directly. In both tests, pressure profiles were achieved first with the SIP alone (baseline) and then adding the NEG pump. During the tests, the vacuum chamber was first pumped with the TMP to less than 10⁻⁶ mbar. The SIPs were then switched on and the bake-out started. The bake-out was carried out at 150 °C for 18 h, with 1 h to ramp up and 2 h to cool down. At the beginning of the bake-out, the NEG pump was activated and then kept at 200 °C. At the end of the baking, when the chamber was still moderately warm (60–80 °C), the NEG pump was re-activated.

Test 3 and 4. The two tests were carried out mounting two NEXTor models (NEXTor D100-5 and NEXTor D200-5 respectively), in an AISI 304 test chamber equipped with a 210 l/s (N₂) TMP and a 3m³/h rotary pump. The chamber was fitted with an Extractor gauge (detection limit is 10⁻¹² mbar) and a quadrupole mass spectrometer (0-100 a.m.u.), which was calibrated against a certified Bayard Alpert gauge for H₂, CO, N₂, CH₄, O₂, Ar, CO₂ and He. In the NEXTor design (see figure 1), NEG cartridges are integrated with a small ion pump (red box in figure 1) into one, very compact, functional unit. The getter cartridge provides large pumping speed and capacity, acting as the main pump for the removal of active gases. The ion pump has the task of removing ungetterable gases like argon and methane. Tests were carried out as follows. The test chamber was pumped with the mechanical pumps for 30 minutes down to 10⁻⁶ mbar and helium leak checked. Baking of the chamber, including the NEXTor pump was started. In the first test the baking was carried out at 170°C for 18 hours, while in the second test the baking conditions were 190°C for 18 hours. In both series, after one hour from the start of the bake, the NEG elements of the NEXTor pumps were activated at 500°C x 1h. Afterwards the NEG element was kept hot at 300°C. Two hours after the end of the baking, while the system was cooling down, the ion element of the NEXTor was degassed, the NEG pump re-activated, to recover its full pumping speed, and the ion element of the NEXTor switched on. In both tests, pressure profiles were achieved first with the SIP alone, featuring 120 l/s (N₂) and 200 l/s (N₂) of pumping speed respectively (baseline).

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Figure 1. Picture of NEX Torr[®] D100-5 (2011-2013 © SAES Getters S.p.A.)

3. Experimental results

The pump-down results in test 1 and 2 are showed in figures 2a and 2b. In both experiments, the achieved pressure values are lower than 3×10^{-11} mbar. Compared to the experiments performed with only the SIP (baseline), an improvement of the pressure by factors of 6 and 30, respectively, was obtained.

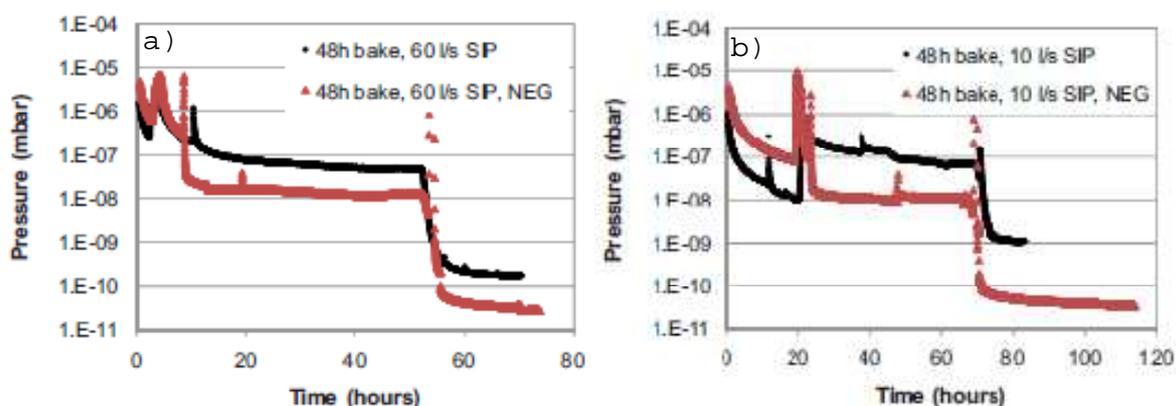


Figure 2. Pump down curves with (red curve) a without (black curve) the NEG pump in the chamber baked at 150°C for 48 h with SIP of a) 60 l/s and b) 10 l/s.

The improvement of the pressure in both experiments can be ascribed to 1) the activation of the NEG pump at the very beginning of the bake out process and 2) keeping the NEG at $\approx 300^{\circ}\text{C}$ during the baking (conditioning mode). The conditioning temperature is sufficient to promote surface to bulk gas diffusion keeping a fresh getter surface which adsorbs all active gases degassed during the baking [4]. In addition, in conditioning mode, the pumping speed of the NEG pump will be higher, reducing in this way the achievable ultimate pressure. It must be noted that under the study conditions the used getter capacity during the bake-out process is a negligible fraction of the pump getter capacities (see table 1).

Table 1. Gas capacities of the NEG pump used into the test 1 and 2. The capacity of H₂O, CO and N₂ is relative at single run of adsorption. The sorbed quantity of each gas has been calculated considering the either the H₂ or the H₂O as the main gas species.

Gas	Capacity (mbar·l)	Sorbed capacity (mbar·l)
H ₂	900	0.86
H ₂ O	80	0.57

This approach may be particularly advantageous in synchrotrons and accelerators, which can enhance the users operation. These results demonstrate that the getter can be used as the main UHV-XHV pump keeping the overall pump size sufficiently small. The weight vs pumping speed for NEG and SIP pumps is provided for reference in figure 3.

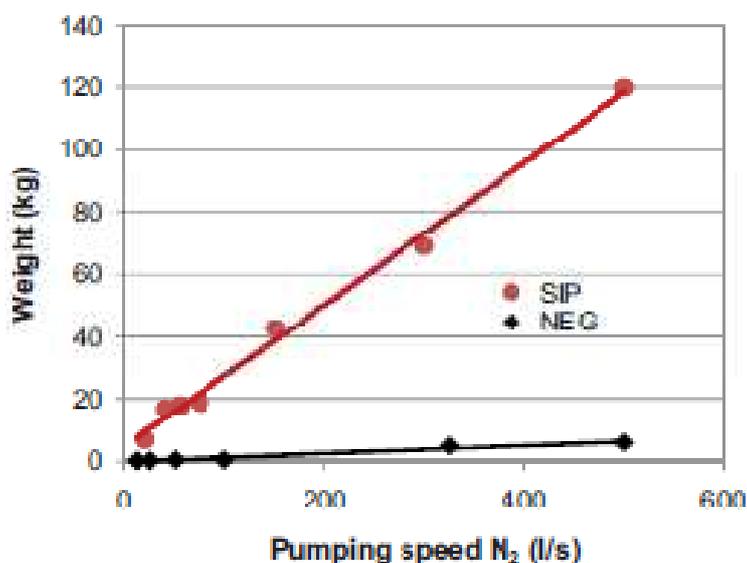


Figure 3. Weight comparison between NEG and SIP pumps as a function of their pumping speed for N₂.

This result is not totally new. In particular, Benvenuti et al. achieved [5, 6] XHV conditions ($<10^{-13}$ – 10^{-14} mbar) by using the NEG strip as the main system while the SIP is the secondary pumping system in order to remove the tiny amount of the other active gases that NEG pump are unable to adsorb.

The concept of combining large NEG with smaller SIP fully applies in the NEXTorr pump family. In particular in the present study, the NEXTorrD100-5 and NEXTorr D200-5 have been tested. The two pumps are a combination of a SIP of 5 l/s pumping speed and NEG pumps of 100 l/s and 200 l/s of pumping speed for H₂ respectively. They have been compared with 65 and 200 l/s(N₂) SIP

respectively (tests 3 and 4). The setups of the two experiments are shown in figures 4 and 5 respectively.

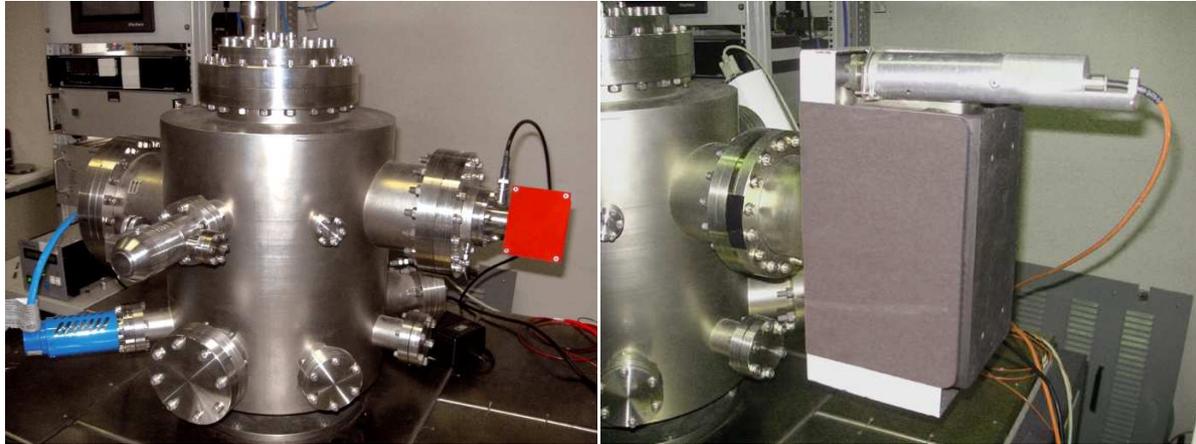


Figure 4. Experimental setup of tests 3 by using NEX Torr D100 (left) and SIP featuring 120 l/s of pumping speed (right).

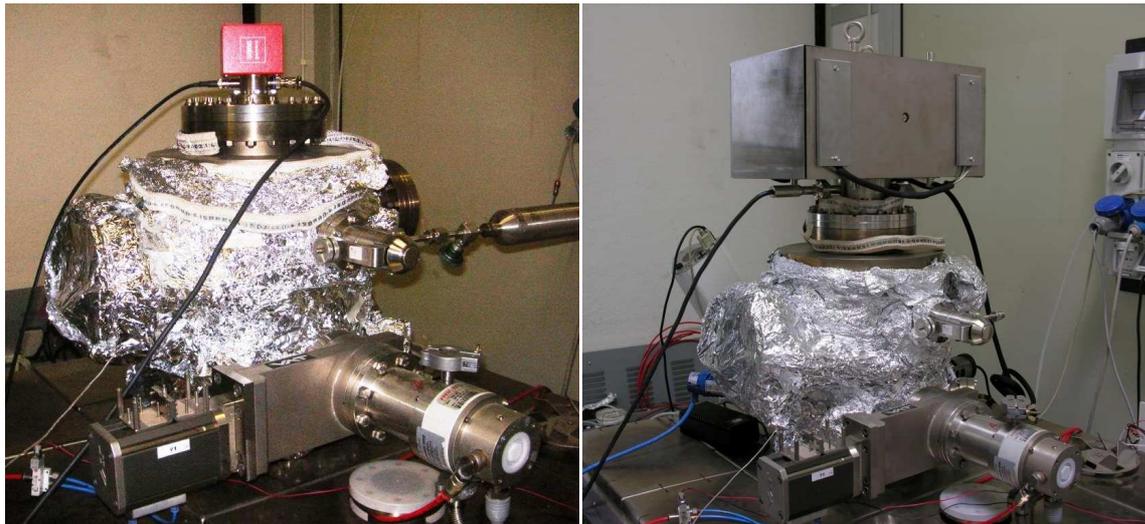


Figure 5. Experimental setup of tests 4 by using NEX Torr D200 (left) and SIP featuring 200 l/s of pumping speed (right).

The base pressure achieved by NEX Torr D100-5 in the pump down experiment (test 3) was a factor 2-3 lower than that obtained by the ion pump (see figure 6). This is an important result considering the much smaller size and weight of the pump. Similar results have been achieved using the NEX Torr D200-5 (see figure 7). Interestingly, if the bake out time is reduced from 18 h to 7 h, the vacuum level obtained by using the NEX Torr D200-5 does not change. Therefore even in the case of the NEX Torr pump, the use of the NEG element during the bake out process can allow to reduce either the ultimate pressure value and/or the baking time.

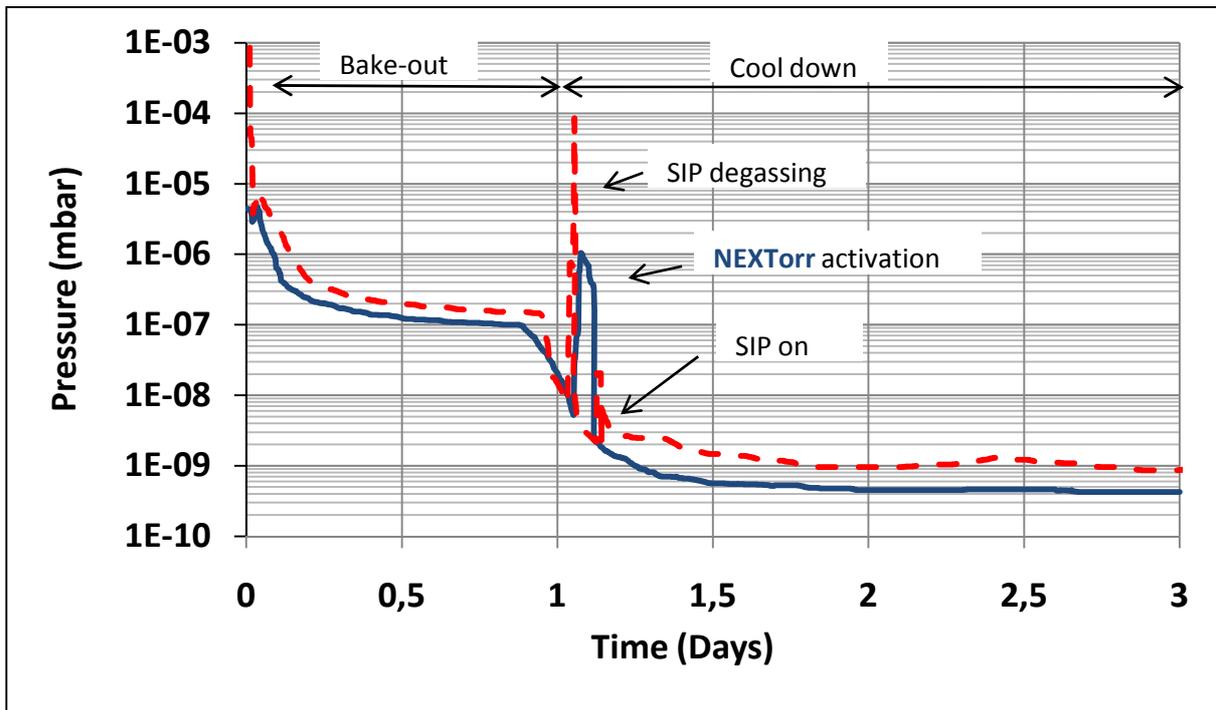


Figure 6. Pumpdown curves by using NEX Torr D100-5 (solid line) and SIP (120 l/s) (broken line) in the chamber baked at 170°C for 18 h.

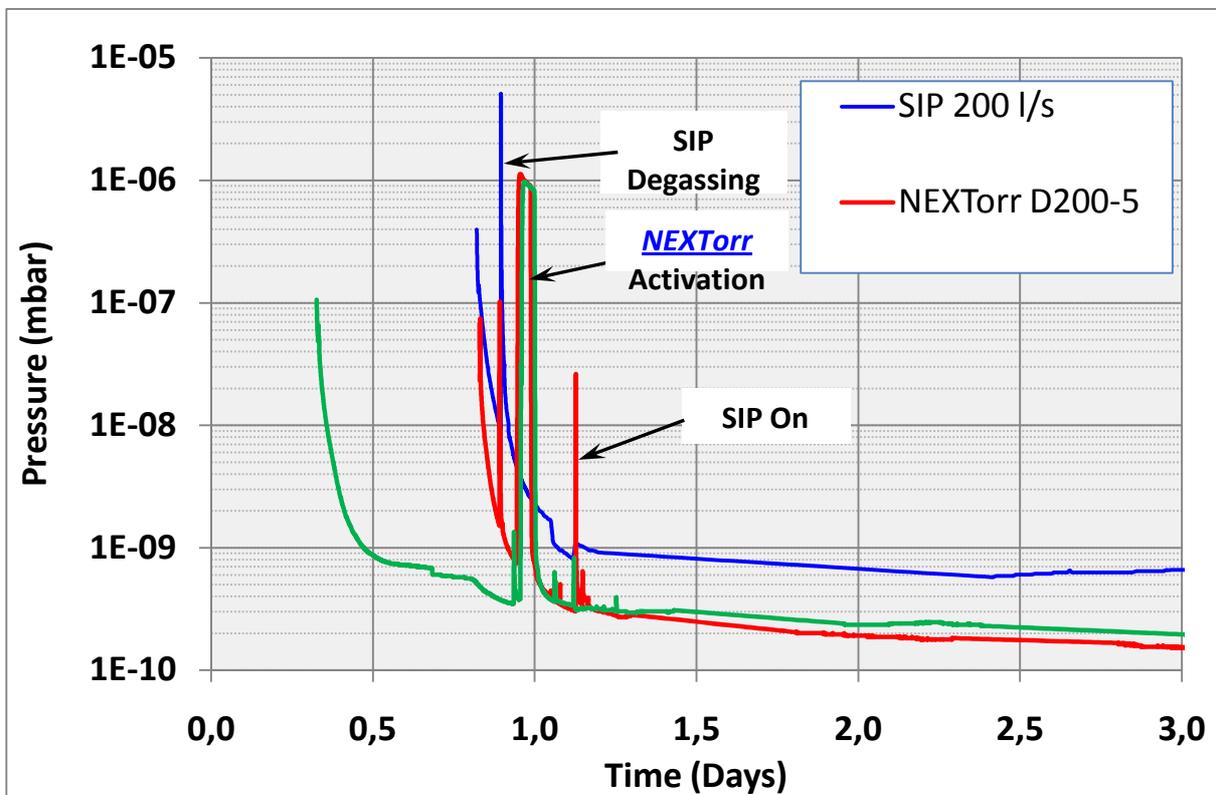


Figure 7. Pumpdown curves by using NEX Torr D200-5 (red and green curves) and SIP (200 l/s) (blue curve)

The reasons of such results can be explained by considering the partial pressure profile of all active gases during the pumpdown experiments as a function of the time. In this experiment the bake out temperature has been fixed at 250 °C. Partial pressure curves of all active gases are smaller in the case of the test using the NEX Torr (see figure 8), mainly due to the large pumping speed of the NEG element.

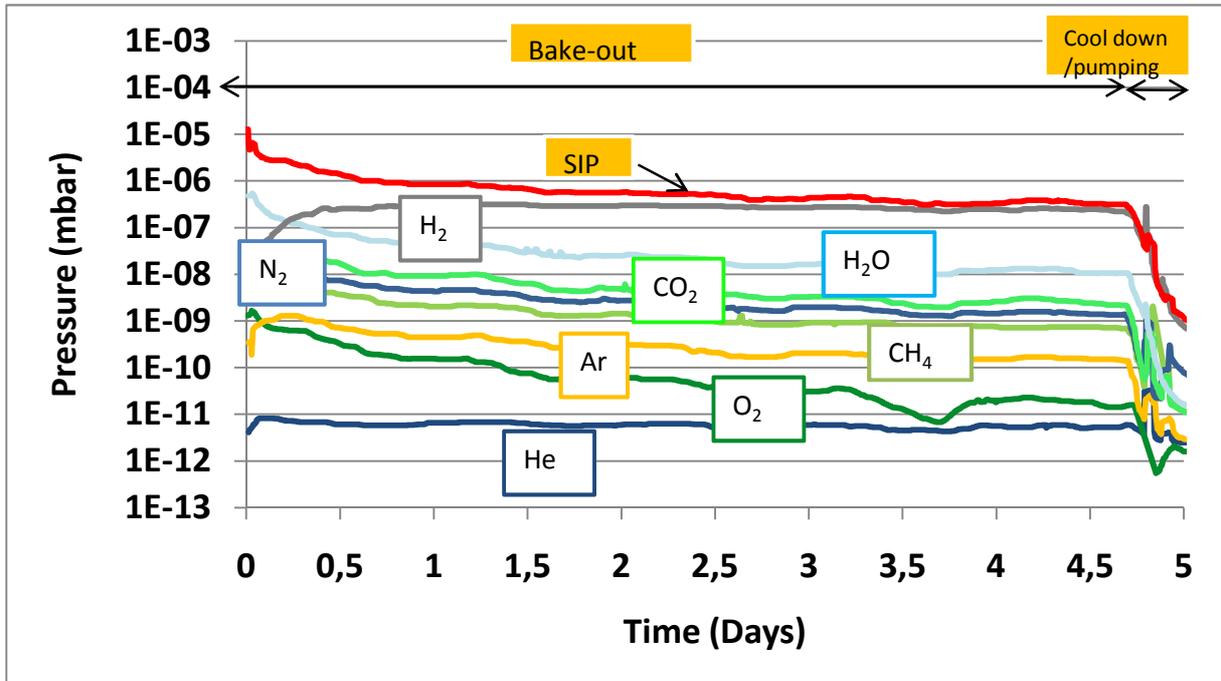


Figure 8. Partial pressure profile of all active gases by using SIP 120 l/s (N₂).

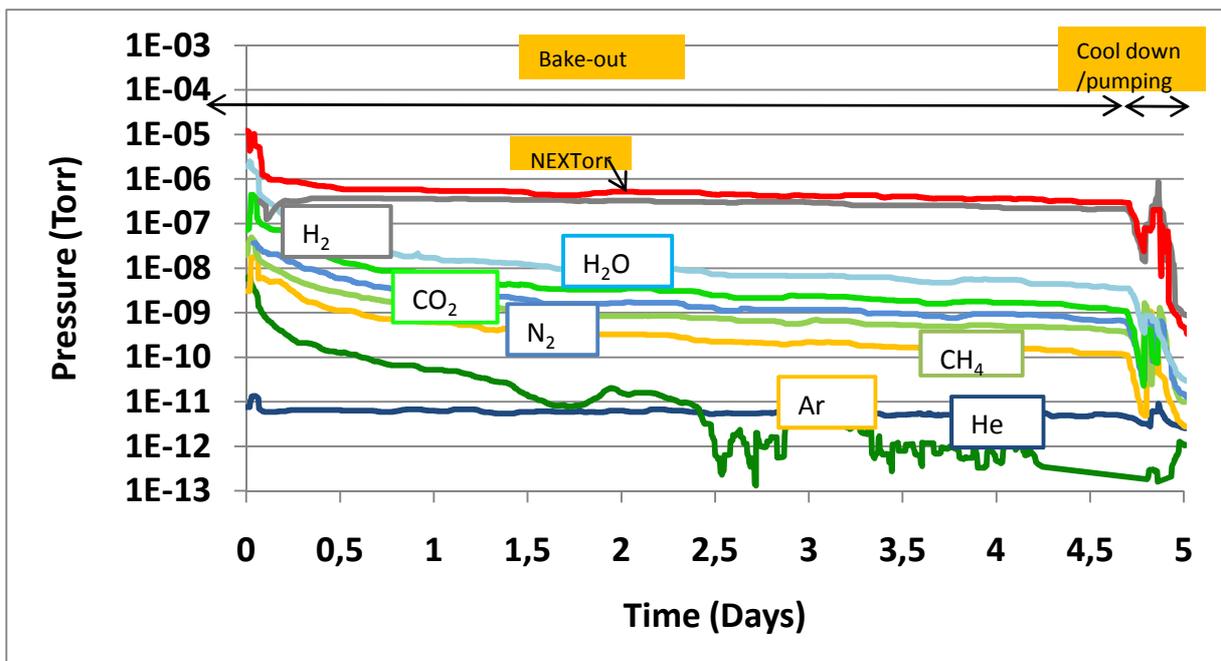


Figure 9. Partial pressure profile of all active gases by using NEX Torr D100-5.

4. Conclusions and perspectives

UHV-XHV systems can be effectively pumped using NEG and SIP pumps combined together. Being methane and argon a small fraction of the residual gas spectrum small ion pumps can be used keeping the NEG pump as the main pump to achieve the ultimate pressure. Such an approach is also used in the case of the NEXTorr a new combination pump integrating a NEG and SIP element in one small package pumping unit Results of pump down tests under different baking conditions shows that the NEXTorr compares favorably with larger and heavier Sputter ion pumps. Additional advantages, particularly appealing for application in large vacuum systems, are the ability of using the getter element of the pump to improve bake-out efficiency. This approach radically removes the main limitations of NEG pumps, providing in a very compact design a superior product in term of pumping speed, capacity, power consumption, reliability. A variety of UHV-XHV systems like surface science and MBE equipment, electron/ion spectrometers as well as high energy physics machines like synchrotrons and colliders can also benefit from this pump design. In fact these systems are generally very crowded with instrumentation, flanges and service ports. The NEXTorr can easily fit in a small space, freeing space for additional instrumentation and experiments.

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