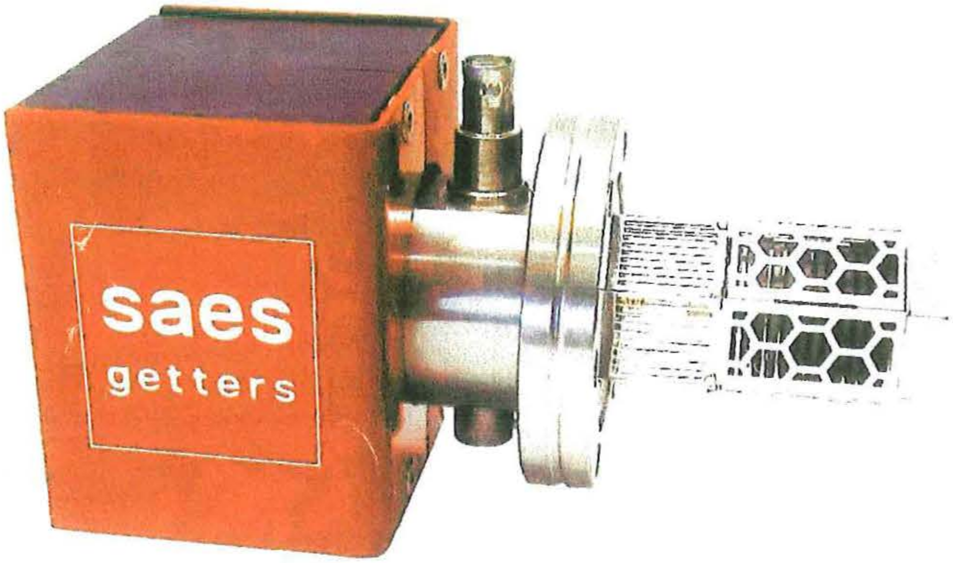
OPERATING INSTRUCTIONS

NEXTORR D 100-5 PUMP



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www.saesgetters.com

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1. PRODUCT GENERAL INFORMATION

The NEXTORR D 100-5 Pumps combines in a very compact and unique design NEG and ion pumping technologies.
The getter element provides very large pumping speed and capacity and acts as the main pump for the active gases, leaving to a small ion pump the ancillary task of removing noble gases and methane which are not pumped by the NEG.
This approach is effective since methane and rare gases are a very small percentage of the gas composition of UHV-XHV systems, generally dominated by hydrogen and oxygenated gases. Just a small pumping speed is thus required for the ion pump to sorb them effectively. The ion pump also provides a pressure reading, which can be used for vacuum and process monitoring. 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.

The NEXTORR pump can be used alone or in combination with other vacuum pumps. It allows reducing the total weight of the pumping group, which has great value in equipments like electron microscopes, semiconductor inspection and review tools where the presence of large masses on the electro-optical columns may introduce vibrations and limit ultimate achievable performances. A variety of UHV-XHV systems like surface science and MBE equipment, electron/ion spectrometers as well as high energy physics machine like synchrotrons and colliders can also benefit from the NEXTORR design. These systems are generally very crowded with instrumentation, flanges and service ports. The NEXTORR can easily fit in a small space providing very large pumping speed in a compact volume, freeing space for additional instrumentation and experiments.

2. NEXTORR D 100-5 PUMP

A typical NEXTORR D 100-5 pump is shown schematically in *Fig. 1*.
The essential components are:

1 - NEG element
2 - ION element
3 - NEG element connector
4 - ION element connector
5 - Connecting flange

1. NEG element

The NEG element consists of a special AISI 304 support that contains sintered porous getter material in the shape of disks suitably stacked in a high efficiency structure. A heating element is inserted inside the disks to allow the proper getter activation. An AISI 304 protective screen surrounds the getter material to avoid possible unwanted contact, contamination or damage of the getter disks. Composition, porosity and specific surface area of the getter disks, as well as their arrangement, have been optimized to significantly increase sorption capacity and speed. The heater consist of a tantalum wire inside an alumina insulator.

2. Ion element

A diode sputter ion pumping element is integrated with the NEG element. It removes rare gases and methane, not pumped by the NEG element and provides a pressure reading. The integration of the getter and the ion elements has been studied and optimized to enhance the pumping performances of the ion element.
In fact, gases released by the ion section during the operation, are intercepted and removed by the getter element, with a substantial reduction of back-streaming effects. For the same reasons, increased pumping efficiency for H_2 and CH_4 are obtained, as well as better argon stability. This allows the achievement of better base pressure in a shorter time. Fine titanium particles which may be sputter-emitted by the penning cells during operation are also effectively trapped by the pump geometry, reducing the potential contamination of the vacuum system.

3 & 4. NEG/ion connectors

These two connectors serve to connect the power cables from the NEXTORR pump to its dedicated power supply unit (NEXTORR POWER SUPPLY NIOPS-03). Cables can withstand a maximum working temperature of 150°C. In the case of baking treatment at higher temperature cables must be removed to avoid being damaged. See *Cap. 3.4* for major information.

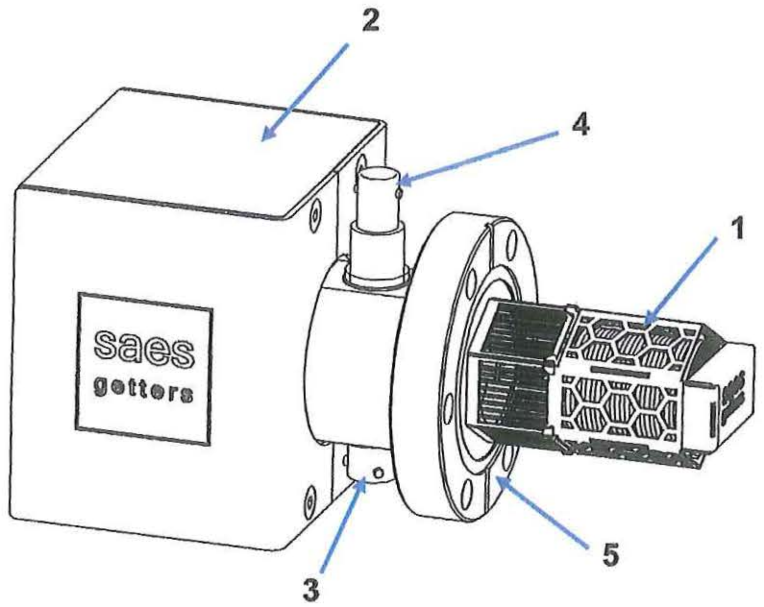


Figure 1

5. Connecting flange

The connecting flange is a standard rotatable DN 40 (or 2¾") ConFlat type flange (AISI 304). The flange assembly is leak tested.

<u>IMPORTANT NOTICE:</u> the getter cartridge, the support flange and the ion pump are, by design, one single piece. Replacement of the cartridge is therefore not possible. At the end of its operation life, when the getter or the ion element is exhausted, the complete NEXTORR pump must be replaced.

2.1. PUMP DIMENSIONS

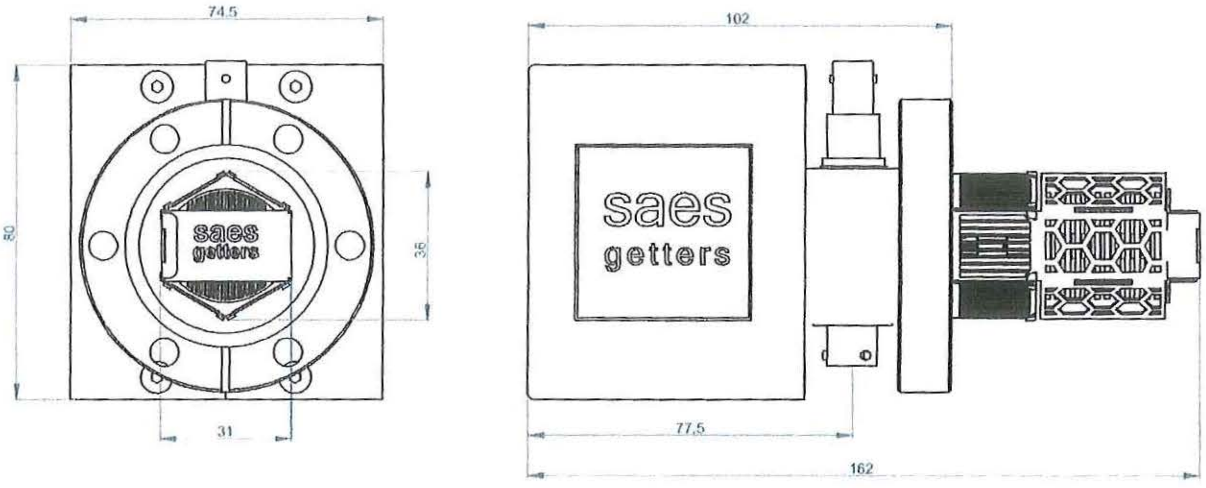


Figure 2 (All measures are in millimetres)

2.2. MAIN TECHNICAL FEATURES OF NEXTORR D 100-5

Initial pumping speed (l/s)	Gas	NEG activated	NEG saturated
	O_2	140	5
	H_2	100	-
	CO	70	6
	N_2	40	5
	CH_4	20	9
	Argon[1]	6	6
Sorption capacity (Torr·l)	Gas	Single run capacity[2]	Total capacity[3]
	O_2	7	>500
	H_2	135	N/A[4]
	CO	0.6	>120
	N_2	0.3	>25
	CH_4	40	50,000 hours at 10^{-6} Torr
NEG section	Getter alloy type		St 172
	Alloy composition		ZrVFe
	Getter mass (g)		13.5 g
	Getter surface (cm^2)		114
ION section	Voltage applied		DC +5kV
	Number of Penning cells		4
	Standard bake-out temperature		150°C

[1] Measured at $3x10^{-7}$ Torr.
[2] Capacity values with the NEG element at room temperature, corresponding to a drop of the pumping speed to 10% of its initial value. A drop to 15% and 50% has been considered in the case of N_2 and CH_4.
[3] Total capacity values for each single gas obtained after many reactivations (getter fully consumed). Capacity values for the various gases are not additive (a getter fully reacted with one gas specie will not sorb another gas).
[4] After the getter element has reached its room temperature H^2 capacity (135 Torr·l) it can be "regenerated". Through the regeneration process it is possible to extract the hydrogen stored in the getter. After a full regeneration process, the pump can start pumping hydrogen as from time (see *Sect. 4.3*).

3. INSTALLATION OF THE NEXTORR PUMP

The NEXTORR pump can be easily mounted connecting its DN 40 flange to the port of the vacuum system. The pump can operate in any mounting orientation.

3.1. Unpacking

The NEXTORR pump is sealed under dry argon. It also contains a copper gasket for flange connection.

⚠ **CAUTION**

Do not open the package until final assembly. The getter material may be harmed by a long exposure to the environment in presence of high humidity levels.

3.2. Precaution during Installation

As for all UHV type equipment, clean, lint free gloves should be used for handling the parts of the pump exposed to vacuum. All operations should be conducted on a clean dust-free bench.

3.3. Installation

The NEXTORR pump not required for the installations in a vacuum system to remove same parts. To use a copper gasket for the DN 40 (or 2 ¾") ConFlat flange connection.

3.4. Bake-out

If a chamber bake-out is carried which can bring the ion element at temperature higher than 150°C, magnets have to be taken out.

⚠ **WARNING**

The maximum baking temperature of the NEXTORR pump with the magnets, the covers and the cable of the NEG pump mounted is 150 °C. The maximum temperature of the ion cable is 60 °C.

To baking the NEXTORR up 150 °C (max 250 °C) remove the cables and the magnets.

1) remove the two cables
2) remove the four assembly screws
3) remove the three covers
4) remove the support with the magnets

See the *Fig. 3*:

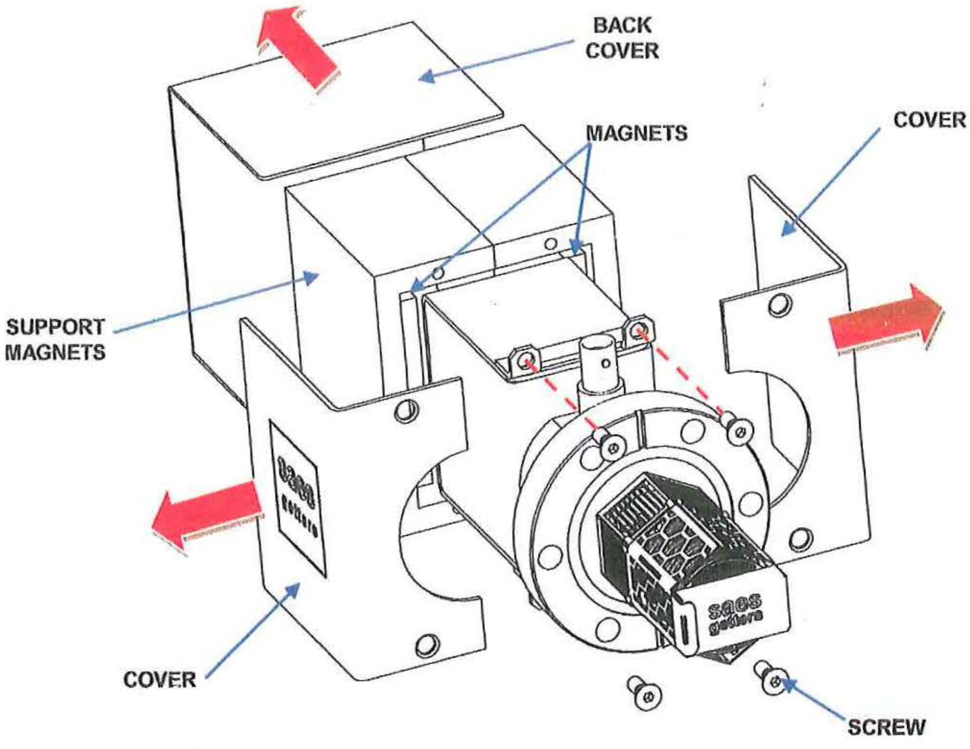


Figure 3

3.5. Powering the pump

To power the pump, the two dedicated cables must be connected to the pump connectors and then plugged to the dedicated NEXTORR NIOPS-03 power supply. This power supply is specifically designed to operate independently and simultaneously the getter and the ion pumping elements. Do not use different type of power supply. Specific information and procedures to use the power supply are given in the:
"User's Manual NEXTORR Pump Power Supply NIOPS-03" (document: M.HIST.0058.23)

4. OPERATING OF PUMP

After having mounted the pump, the following sequence is recommended:

- Check the vacuum conditions. If the pressure is below 10^{-5} Torr, switch on the ion element using the dedicated NIOPS-03 power supply.
- A bake out of the ion element is in general advisable to ease degassing and achieve better final pressure. This bake-out can be carried out along with the chamber bake-out or, in a separate moment, if no bake-out of the chamber is planned. Standard heating tapes or other heating means can be used to heat the ion element. During chamber and ion element bake-out the ion element must be turned off.
- After the bake-out of the ion element and before starting the NEG activation process, flash the ion pump to further clean it. It is suggested to flash the ion element before the NEG activation to avoid that possible contamination coming from the flashing process may partially saturate the getter. During the activation the ion element must be turned off.

- Start the activation of the NEG element (see following section for details).
- Once the getter element is activated, the ion element can be turned on. At this point the NEXTORR pump will develop its full pumping speed, removing both inert and active gases from the vacuum environment.

During normal UHV-XHV operations the NEG element sorbs gases at room temperature and does not need power to operate. Under these conditions, only the ion pump element of the NEXTORR pump needs to be powered.

After having pumped a significant amount of gases, the surface of the NEG element of the NEXTORR pump will progressively become saturated. When the pumping speed drops below an acceptable value (generally set at 10% of the initial pump speed), the NEG element needs to be reactivated. This procedure, like the first activation, restores the initial pumping speed of the pump (see *Sect. 4.1* for more details)

In presence of a high gas or during a long bake-out it may be useful tom keep the temperature of the getter at 250÷350 °C. Doing so, the gases trapped on the surface and chemically reacted will progressively diffuse from the surface to the bulk of the getter material freeing a significant fraction of the surface sites for additional chemisorptions. As a result, the pump will be able to sustain a significant speed for a longer time. This is helpful to improve the achievable base pressure during the pump down or to reduce the bake-out duration. During this process, depending on the pressure in the system the ion element can be kept on or off. For pressure higher than 10^{-7} Torr if is advisable to keep it off, to reduce ion element partial saturation for hydrogen. Hydrogen as in fact the (main) gas released by the getter when kept at 250÷350 °C (see *Sect. 4.1*)

When pumping high purity hydrogen, operation at room temperature is also possible with high gas loads, due to the relatively large diffusivity of H_2 at that temperature. In this case, a careful evaluation of the gas load and the quantity of gas sorbed is necessary to avoid the embrittlement of the getter material, which takes place when hydrogen concentration exceeds 20 Torr·l (H_2)/g of getter are reached. For this reason, to stay on the safe side, it is recommended not to exceed the value of 10 Torr·l (H_2)/g (getter).

4.1. ACTIVATION OF THE NEG ELEMENT

The non-evaporable getter materials used in the NEXTORR getter cartridges (Zr-V-Fe, St 172®) develops its pumping characteristics after an **activation** process, i.e., a heat treatment carried out at moderate temperature under vacuum for a sufficiently long time.

The heat treatment remove the thin surface layer formed on the getter surface due to air exposure during manufacturing process (mainly oxides and carbides).

The activation process "cleans" the getter surface which is then able to sorb gas molecules present in the vacuum system in which it is operated.

The efficiency of this activation process is related to the diffusion coefficient of the specific getter material, which in turn depends on an exponential function of the temperature: $D = D_0 e^{-E/T}$.

It is also related to the square root of time, as are all diffusion processes.

In first approximation, the activation efficiency, which is related to the quantity of passive species removed from the surface of the getter particles, can be expressed by the following formula:

$$\text{Activation efficiency} \propto \sqrt{Dt}$$

The suggested current/time combinations for the best (100%) activation for NEXTORR pumps with St 172 alloy getter material is:

Activate @ → **9 V - 5 A (45 W) for 60 minutes**

With this current, the getter temperature for the pump in "nude" configuration is about 450°C (see **APPENDIX A**). When the pump is used inside a jacket or in a small size chamber, the chamber wall will act as thermal shield allowing the getter material to reach higher activation temperature. Lower current values might therefore be sufficient to reach 450°C.

⚠ **CAUTION:** the maximum applicable current is 5 Ampere.

The activation process should be carried out after the pump down of the vacuum system to a pressure of 10^{-4} Torr or less. After having reached this pressure, the heater can be energized to reach the desired temperature.

During the heating phase there is a desorption of gases from the getter material. This is due to the physisorbed and weakly chemisorbed gases which form the external monolayers covering the surface of the getter material, while the internally chemisorbed layers are diffused into the bulk of the getter material. Some hydrogen, in solid solution in the alloy lattice, will also be released. Hydrogen is very quickly re-adsorbed as soon as the getter cartridge cools down (see also *Fig. 4*).

heat tape on arm,

Desorbed gases generally include H_2, H_2O, CO, CO_2 and CH_4. In order to minimize this gas evolution, baking the system at temperature in the 150÷250°C while keeping the getter cartridge maintained at a slightly higher temperature (e.g. 200÷300°C) has been found to be effective. This procedure minimizes the migration of the gases desorbed from the wall of the system toward the getter cartridge which has an effective surface area much larger than the system walls themselves.
During activation it is advisable not to exceed pressures in the 10^{-3} Torr range to avoid phenomena of corrosion of the heater wire and partial saturation of the getter material due to the high gas load and the consequent sorption of active gases.

For this purpose and depending on the backing pump pumping speed, it may be advisable to activate the getter cartridge by successive steps, not applying the full power to the heater at the beginning of the activation process. In this case, reaching the activation temperature may take a few hours compared to 60 minutes usually needed if full power is applied at the beginning.

How long activate? + bake? — until $p < 10^{-4}$ torr.

4.2. REACTIVATION OF THE NEG ELEMENT

The reactivation of the getter material of a NEXTORR is necessary when the pump is exposed to air or when its pumping speed falls below acceptable limits. In either case the surface of the getter material becomes covered by a passivation layer of mainly carbides and oxides.
If reactivation is preceded by an air exposure and a successive pump down cycle, it should follow the same procedure of first activation and has the same characteristics.
If the reactivation follows normal operation in vacuum without air venting, it may be shorter and carried out at a lower temperature (approximately 25% lower). Moreover, during a reactivation which follows normal operation in vacuum the only gas released is hydrogen.

Through successive reactivations it is possible to use the entire capacity of the getter material. When the pumping speed no longer recovers sufficiently after reactivation, the pump must be replaced. One way to determine if the getter cartridge is exhausted is when the following quantities of gas have been sorbed:

$$q_{CO} + q_{CO_2} + \frac{1}{5} q_{O_2} + \frac{1}{5} q_{H_2O} + q_{N_2} = 9\ torr \cdot liter / g \qquad (1)$$

4.3. REGENERATION OF THE NEG ELEMENT

4.3.1. General concept

When the NEXTORR pumpis exposed to high hydrogen loads, large amount of hydrogen may be sorbed by the getter material. It is however possible to remove this hydrogen from the NEG element through a special procedure called "regeneration". This is possible thanks to the reversible nature of the hydrogen sorption on St 172®.

Regeneration is necessary when:

- the pumping speed for hydrogen or hydrogen isotopes has fallen below acceptable limits because the equilibrium pressure has been approached

- equilibrium is far from being reached but the hydrogen or hydrogen isotopes quantity pumped is approaching the safety limit of 10 Torr·l/g.

Embrittlement of the material takes place when the quantity of hydrogen sorbed in the getter material is high enough (20 Torr·l/g) to modify the mechanical characteristics of the alloy causing it to flake in small particles. For this reason it is advisable not to exceed the safety limit of 10 Torr·l/g. The process of hydrogen sorption and successive desorption (regeneration), can be visualised on the Sievert's plots (see *Fig. 4*).

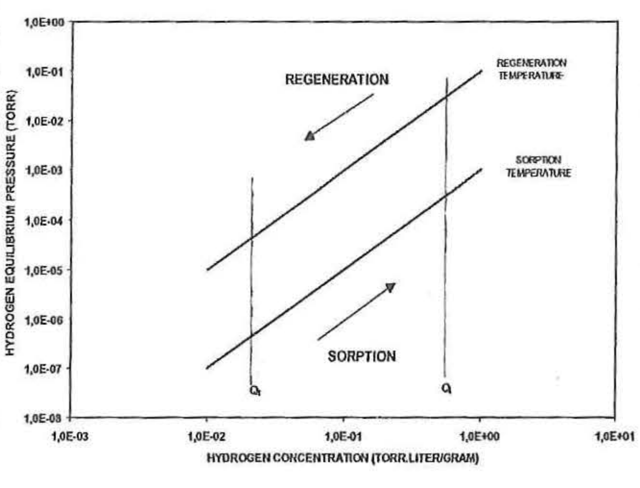


Figure 4

The temperature increase of the getter alloy establishes a high hydrogen equilibrium pressure, allowing hydrogen removal by means of a standard backing pump. From the plot in *Fig. 4* one can see that the regeneration process will be more efficient at higher temperature.
The time necessary for the regeneration process is given by the expression:

$$t = \frac{M}{F}\left(\frac{1}{q_f} - \frac{1}{q_i}\right) 10^{-(A - \frac{B}{T})} \qquad (2)$$

where:

t = regeneration time, in seconds
M = mass of getter material, in grams
F = pumping speed of the backing pumps, in liter/s
q_f = final H_2 concentration, in Torr·liter/g
q_i = initial H_2 concentration, in Torr·liter/g
A = 4.45 for St 172 alloy
B = 5730 for St 172 alloy
T = regeneration temperature, in K

Due to the exponential shape of the regeneration curve, a significant amount of time is saved when the regeneration is not programmed to be complete (100% of the sorbed hydrogen released) but only partial (for example 90%).
Thus if a given amount of hydrogen must be sorbed in each cycle (for instance 5 Torr·l/g), it is much better to operate in the q_f=2 - q_i=7 mode, than in the q_f=0 - q_i=5 one.

The Sievert's law for St 172 getter material is given by the expression:

$$\text{Log } P = 4.45 + 2 \text{ Log } Q - 5730 / T \qquad (3)$$

where :

P = equilibrium pressure, in Torr
Q = sorbed quantity, in Torr·liter/g
T = temperature, in °K

The equilibrium pressure curves of hydrogen for St 172 getter material are shown in **APPENDIX C**.

4.3.2. Regeneration procedure

To carry out the regeneration process of the NEXTORR pump the following simple steps have to be taken:

- Switch off the ion element (to avoid its exposure to relatively high pressure of hydrogen).
- Provides a turbo-molecular pump (or other baking pump) with pumping speed *F*, able to remove the hydrogen released by the getter element.
- Heat the getter element up to the desired regeneration temperature *T*.
- Keep the getter element at this temperature for the time *t* under turbo-molecular pumping.
- From formula (*2*) it is possible to calculate the amount of hydrogen removed and the amount still store in the alloy. Should this value acceptable switch the heater off and let the getter cool down.
- When the pressure is sufficiently low (e.g. < 10^{-7} Torr), switch the ion element on.

4.4. OPERATION OF ION ELEMENT

- Instruction for the proper use of the ion element are contained in the NEXTORR POWER SUPPLY user's manual.

- After the activation, reactivation or regeneration of the NEG element, the ion element can be started by pressing the pushbutton ON/OFF in the section ION of the power supply
- When the ion element is operating properly, the current drawn is proportional to the pressure as shown in **APPENDIX D**. A reading of a current or of a pressure on the power supply display indicates that the ion element discharge has started.

WARNING

Before removing the high voltage connector of the cable from the power supply unit, be sure the main power is removed from the power supply unit. Wait at least 10 seconds after removing the main power from the power supply unit, to allow capacitors to discharge completely

4.5. SPECIAL INSTRUCTION

4.5.1. Air venting

Venting to air the NEXTORR pumps must be performed only when the getter material is at room temperature or, at least, below 50°C (for St 172 getter). If dry nitrogen is used, the temperature of the getter element can be higher (100°C). After each air exposure, a new reactivation of the getter cartridge is required. A progressive reduction of pumping speed for hydrogen and active gases is observed after successive exposures to air. If dry nitrogen is used instead of air, the pumping speed reduction, after the same number of exposures, is significantly smaller. This is because, for the first cycles, the active gas diffusion effect of the reactivation which follows a nitrogen exposure is greater than the new contamination caused by the exposure to nitrogen. Further improvement is obtained when pure argon is used as a protective gas during maintenance operations.

4.5.2. Vacuum failure during activation or regeneration

During the activation phase of the activation or regeneration process of NEXTORR pumps, air must not be allowed to suddenly enter the vacuum system. Such an occurrence can cause the oxidation of the getter material if the temperature of the getter, at the moment of vacuum failure, is above 100°C and the pressure of oxygen higher than 0.1 Torr.

For higher getter temperature and oxygen pressures, the oxidation can take the form of a "burning" reaction, which will be in any case slow and progressive, not explosive. Should a serious vacuum failure take place when the temperature of the cartridge is high, although below the above indicated values, permanent damage of varying degrees will occur, according to temperature, but not burning. In this case, pumping characteristics of the getter material would be affected to a greater or lesser extent depending on temperature and time.

4.5.3. Mechanical shocks

Due to the mechanical characteristics of the getter disks on the pump and of the insulating elements of the heater (ceramic components) particular care must be observed in handling during assembly (and removal) of the pumping system. Accidental dropping and similar mechanical shock could result in permanent damage of the getter disks or the heating elements.

5. SERVICE

For a request of return of the pump please contact SAES Customer Service.

Sales & Service Locations:

Europe, Middle East and Africa:

SAES Getters S.p.A.
Viale Italia 77
20020 Lainate (Milan) - Italy
Ph. +39 02 93178 1 - Fax +39 02 93178 320

European Customer Relations:
Ph. +39 02 9317 8402 - Fax +39 02 93178320
E-mail: CRM_SALES@saes-group.com

Asia and Oceania:

SAES Getters S.p.A. - Japan Technical Service Branch Office
2nd Gotanda Fujikoshi Bldg.
23-1 Higashi Gotanda 5-Chome
Tokyo 141, Japan
Ph. +81 3 542 00431 - Fax +81 3 542 00438

SAES Getters (Nanjing) Co.,Ltd.
56 Xingangdadao, Xinshengwei
Nanjing Economic & Technical Development Zone
Nanjing 210038, Jangsu Province, P.R. of China
Ph. +86 25 8580 2335 - Fax +86 25 8580 1639

SAES Getters Korea Corporation
7th Fl. Dongwon Bldg. 143-28
Samsung-dong, Gangnam-gu
Seoul 135-877, Korea
Ph. +82 2 3404 2400 - Fax +82 2 3452 4510/11

North and South America:

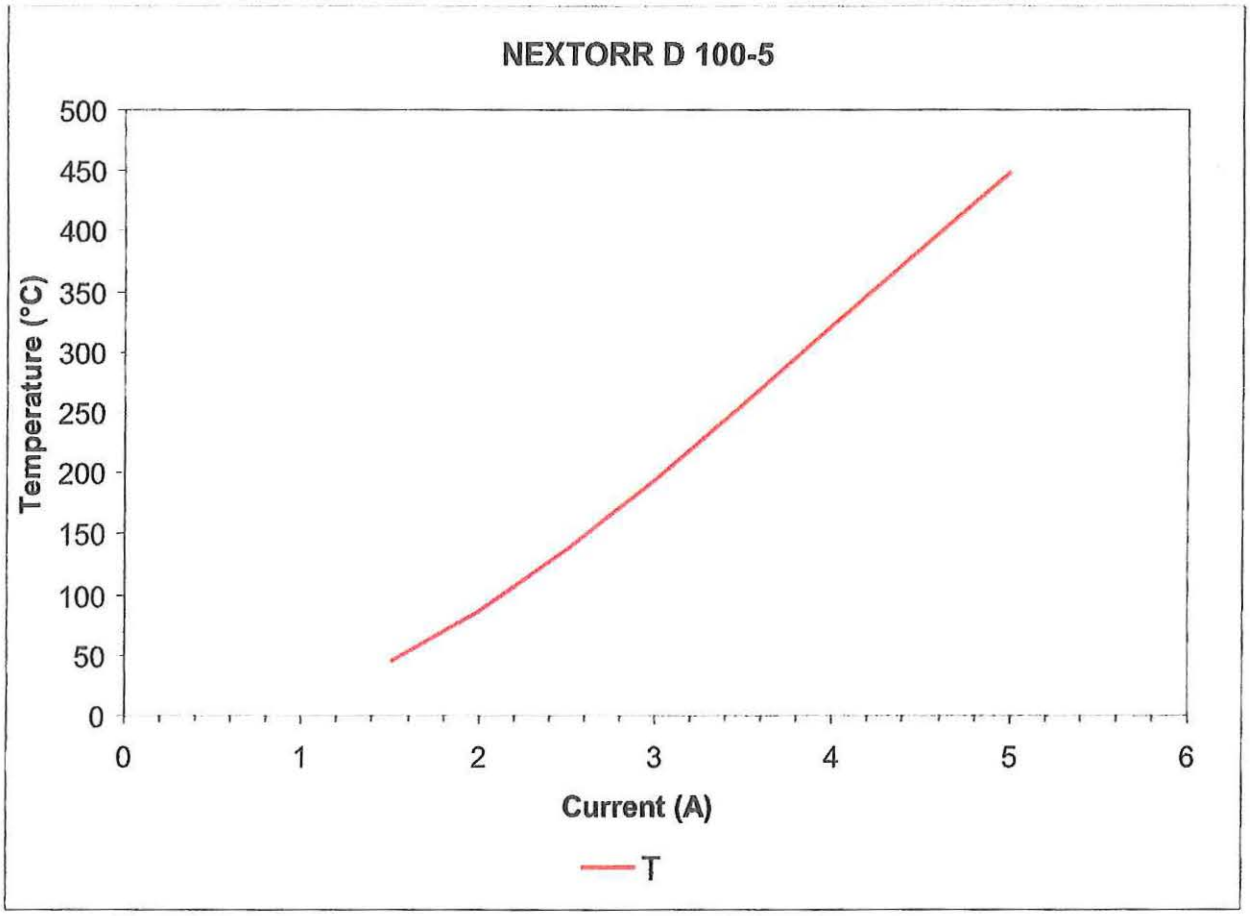
SAES Getters USA, Inc.
1122 East Cheyenne Mountain Blvd.
Colorado Springs, CO 80906 - USA
Ph. +1 719 576 3200 - Fax +1 719 576 5025

Remember that SAES cannot accept any pump which contains biological or chemical hazards or radioactive substances. Please clearly inform SAES Customer Service should this have happened during pump use, so to discuss adequate solutions.

APPENDIX A

NEXTORR D 100-5

Typical Heating Curves

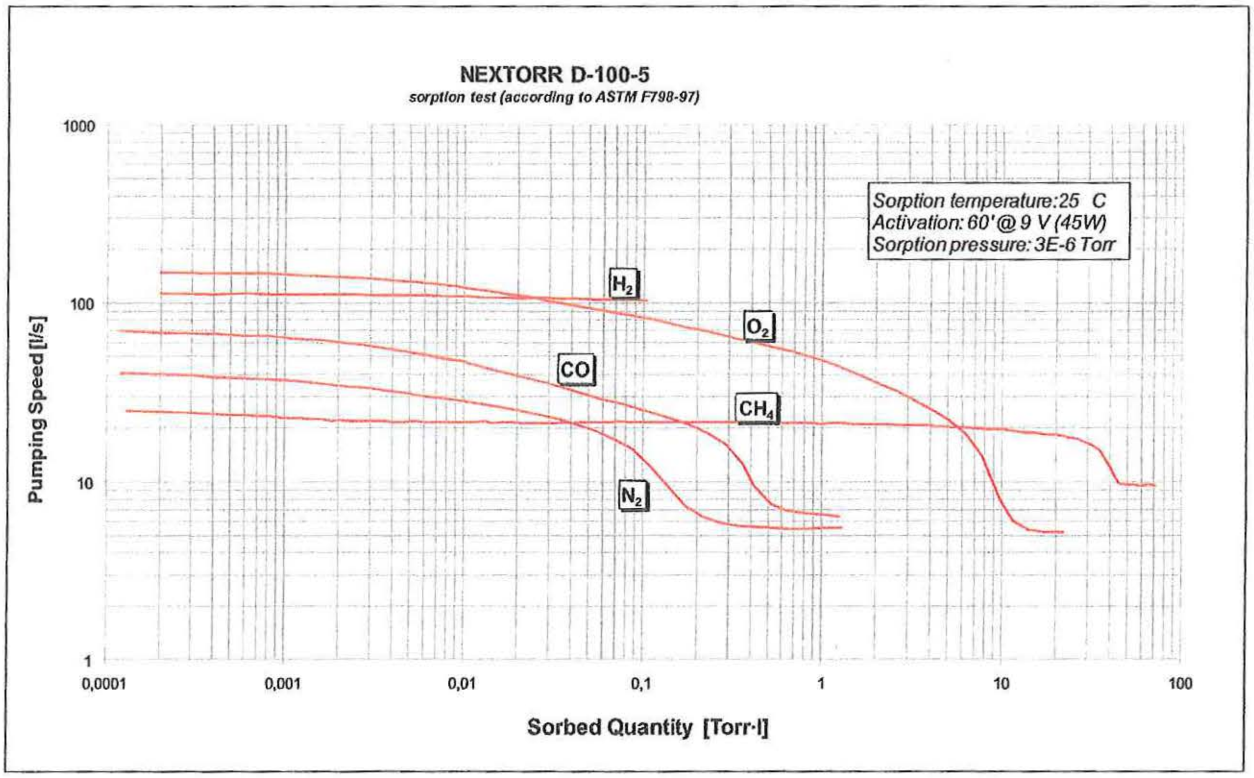


Current as a function of the average temperature in the getter cartridge.

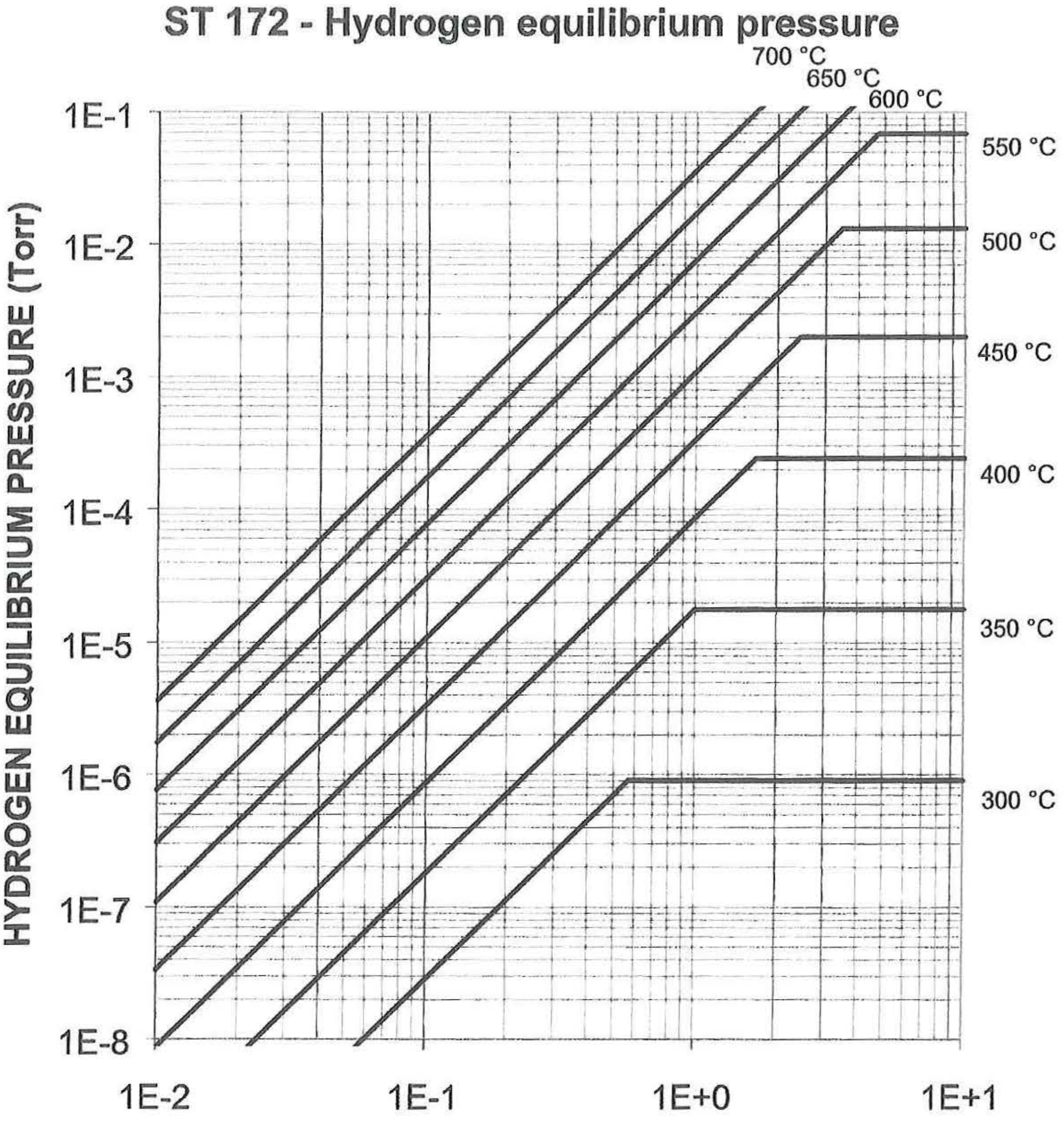
APPENDIX B

NEXTORR D 100-5

Typical Sorption Curves



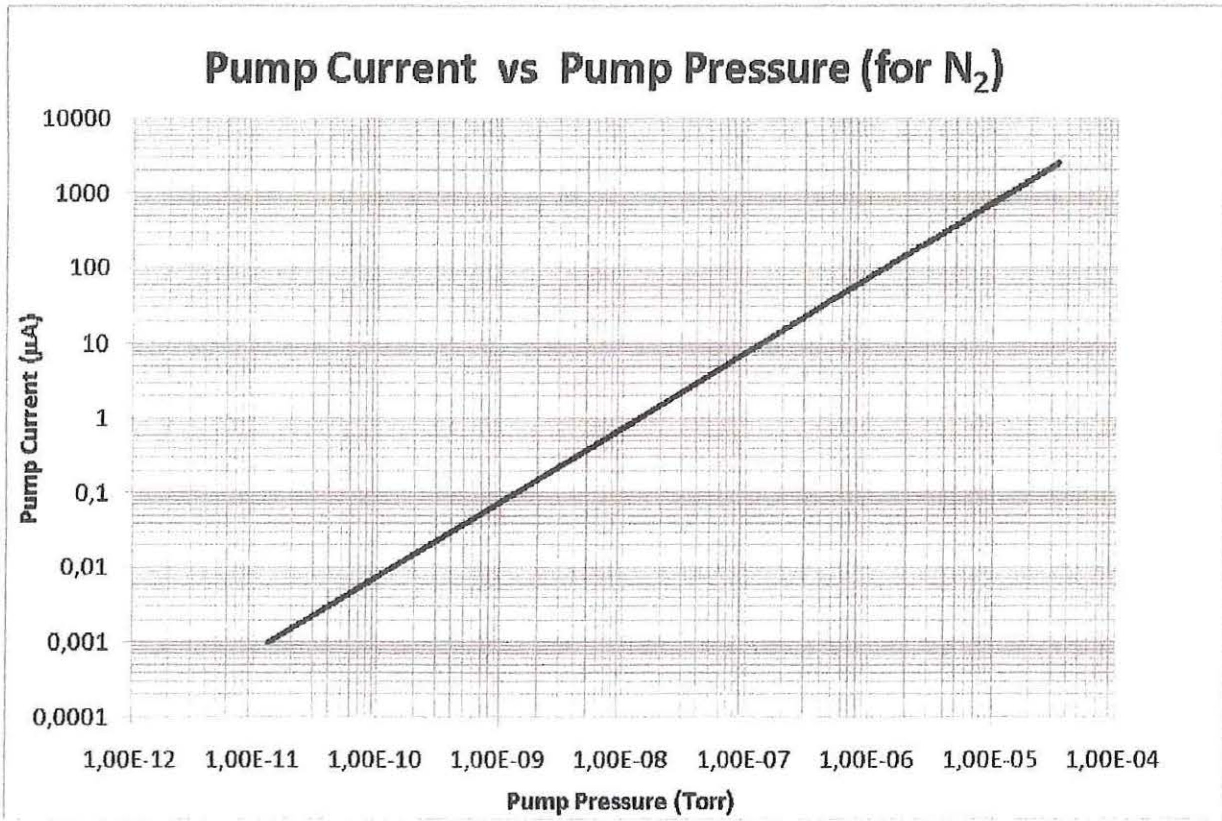
APPENDIX C


ST 172 - Hydrogen equilibrium pressure
700 °C
650 °C
600 °C
550 °C
500 °C
450 °C
400 °C
350 °C
300 °C
HYDROGEN EQUILIBRIUM PRESSURE (Torr)
1E-1
1E-2
1E-3
1E-4
1E-5
1E-6
1E-7
1E-8
1E-2
1E-1
1E+0
1E+1
HYDROGEN CONCENTRATION (Torr*l/g)

APPENDIX D

NEXTORR D 100-5

Typical Current vs Pressure Curves



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