OPERATING INSTRUCTIONS FOR

SORB-AC® CARTRIDGE PUMPS

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SORB-AC Cartridge Pumps are a type of vacuum pump manufactured by SAES ADVANCED TECHNOLOGIES S.p.A., which sorb active gases with a non-evaporable getter material. They are used alone or in combination with other vacuum pumps in several applications in the high and ultrahigh vacuum field (HV and UHV).

DESCRIPTION OF SORB-AC CARTRIDGE PUMPS

A typical SORB-AC Cartridge pump is shown schematically in fig. 1.

The essential components are:

Cartridge containing the getter material **(C)**

Heater assembly **(H)**

Heater support flange **(F)**

Removable pump body (water cooled version available) **(W)**

Thermocouple **(T)***

Electrical connector **(E)***

* The thermocouple and the electrical connector are not available on the GP 50 pump.

Cartridge

The cartridge is made by coating the getter alloy onto metal strips which are then pleated in concertina fashion and formed into cylinders which make up the cartridges.
Iron or constantan strip are used for St 101 strip, while only constantan is used for St 707 strip. A stainless steel tube is inserted in the cartridge to facilitate the correct positioning of the thermocouple.

Heater

The heater consists of a tantalum wire spiral wound on alumina elements supported by stainless steel rods. The heater assembly also includes the cartridge positioning and securing components.

Flange

The support flange is a standard CF type flange in which two current feedthroughs for the heater and a miniconflat nipple are TIG welded. The nipple is used to connect the thermocouple assembly.

Pump Body

The optional pump body is a simple double flanged nipple. If desired, the pump can be mounted nude inside the vacuum system to fully exploit its pumping speed. The pump body can be supplied with a water cooled jacket, but it should be noted that water cooling is not necessary for the pump to function efficiently, except when using the high activable getter alloy St 101. Both the pump body and the pump flange are leak tested to 1.5×10^{-10} scm^3/s.

Thermocouple

The thermocouple is a standard K type insulated and embedded in a stainless steel sheath. The thermocouple sheath is vacuum brazed to a miniconflat blank flange assembled on the support flange.

Electrical connector

The electrical connector allows the connection of the pump to the power supply and control unit. A single MS circular connector is used for the GP pump models equipped with the thermocouple. The thermocouple connections are compensated. The connector assembly can be removed if the pump needs to be baked.

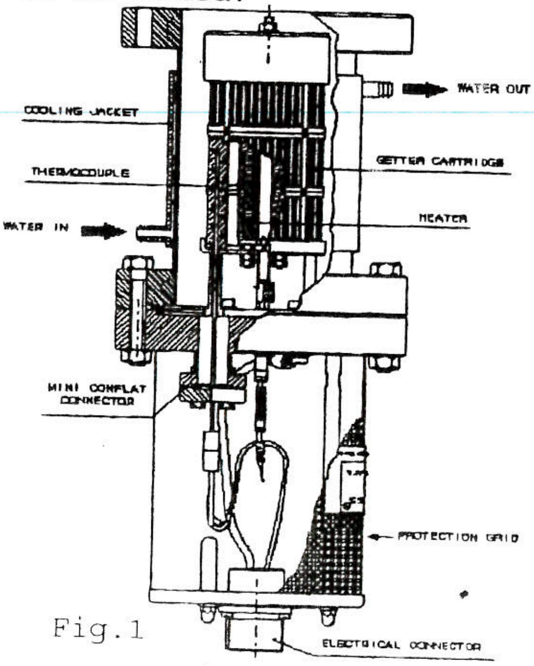


Fig.1

INSTALLATION OF A SORB-AC CARTRIDGE PUMP

The SORB-AC Cartridge Pumps can be mounted on any suitable CF type flange, in appendage to the vacuum system to be pumped. In addition the pump could be mounted "nude" inside the vacuum system removing the flanged body that could be optionally supplied with a water cooling jacket, and directly connecting the heater supporting flange to the port to the vacuum system.

A) Unpacking

The pump is contained in two packs: one for the pump body and the second for the corresponding SORB-AC cartridge sealed under dry nitrogen. The pack which contains the pump may be opened immediately on arrival. It also contains copper gaskets for flange connection (one or two respectively for a nude or an enclosed pump).

CAUTION: 1) Do not open the cartridge pack, until final assembly. The getter material may be harmed by a long exposure to air, specially if stored for several days in a humid atmosphere.

B) 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 operation should be conducted on a clean dust-free bench. All tools used for the assembly should be cleaned before use.

(GP W and GP B only) disassemble the heater flange from the water jacket.

CAUTION: The copper gasket already in this flange assembly has been used as a spacer for transport. As the flange assembly has not been tightened, the gasket should be used for the final assembly.

Stand the pump vertically with the heater to the top.

Now refer to fig. 2.

GP 100, GP 200 and GP 500, remove the nut (1) and lift off the washer (2) from the heater central rod.

GP 50, remove the nuts (11) and bolts (10), take off the two halves of the split clamp (9) and lift off the bakable spring washers (7).

GP 100 and GP 50, lift off the ceramic insulating spacer (8).

Lift off the cartridge centering cap (3 or 12).

Open the cartridge pack and place the cartridge over the heater, taking care not to damage the heater assembly and ensuring that the base of the cartridge goes within the base centering ring (4), and the thermocouple (5) fits in the thermocouple tubulation (6) provided in the cartridge (the GP 50 is not provided with a thermocouple).

Replace the centering cap (3 or 12).

GP 100 and GP 50, replace the ceramic spacer (8) ensuring that the narrow part is in the centering cap.

GP 100, GP 200 and GP 500, replace the washer (2) and the nut (1) on the heater central rod.

GP 50, replace the spring washers (7) with

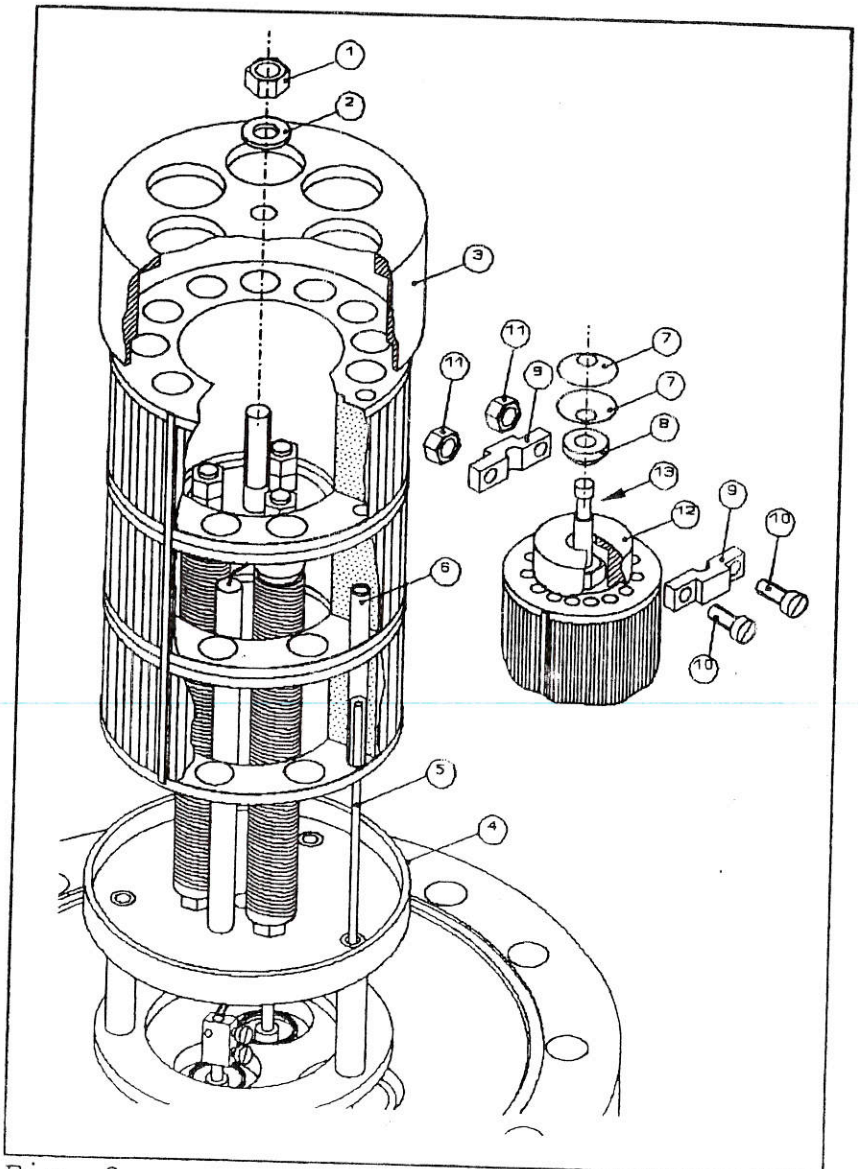


Fig. 2

their cavities alternating, replace the split clamp (9) with its bolts (10) and nuts (11), ensuring that it locates on the relieved part (13) of the central rod.

Check that the cartridge is properly seated.

Check, with an ohmmeter, at the electrical connector (see fig. 3) or at the current feedthroughs for the GP 50, that the heater shows continuity and is insulated from the flange.

GP W and B only. Reassemble the heater flange to the pump body, using one of the gasket supplied.

<u>CAUTION</u>: The water cooled envelop W 50 of the GP 50 2F W pump is not symmetrical. Care must be taken to reassemble the pump with the shorter neck next to the heater flange.

GP W and B only. Assemble the free flange of the pump body to the vacuum system using the other gasket supplied.

GP only. Assemble the heater supporting flange to the vacuum system using the gasket supplied.

C) Water Cooling

For all pumps fitted with a water jacket, the minimum cooling water flow is 0.5 liter/min. The water should not be connected (after baking) until the pump body has cooled to below 100°C.

D) Powering

The pump models from GP 100, GP 200 and GP 500 can be powered using the dedicated SORB-AC Pump Control Unit, for details see the instruction manual that comes with the Control Unit.

In case the Control Unit is not available, other DC or AC power sources can be used. Details of

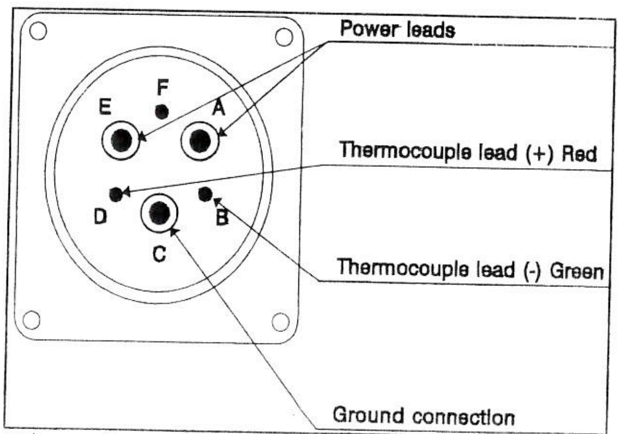


Fig. 3

the pump connector terminals are shown in Fig. 3.

In case the temperature cannot be recorded, as in the case of the GP 50 pump which is not provided with a thermocouple, it is possible to refer also to the heating curves in **Appendix A**.

ACTIVATION OF SORB-AC CARTRIDGE PUMPS

The non-evaporable gettering materials used in the getter cartridges develop their pumping characteristics after an **activation** process, i.e., after heating them to a suitably high temperature under dynamic vacuum for an appropriate time.

The heat treatment diffuses the thin protective layer, formed on the surface of the getter particles by air exposure during manufacture, into the bulk and makes the surface of the getter clean and able to sorb the gas molecules of 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: $D=D_0\exp(-E/T)$. It is also related to the square root of time, as all the diffusion process.

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

$$\mathit{Activation\ efficiency} \propto \sqrt{D\,t}$$

Fig. 4 gives the suggested time/temperature combinations for the best (100%) activation for both the St 101 (700° C for 45 minutes) and St 707 (450° C for 45 minutes) alloy based cartridges.

The curves show that in order to obtain the same final efficiency a shorter duration must be compensated by a higher temperature of the treatment and vice versa. Both these parameters are limited by practical and physical reasons to the range of values indicated in the figure.

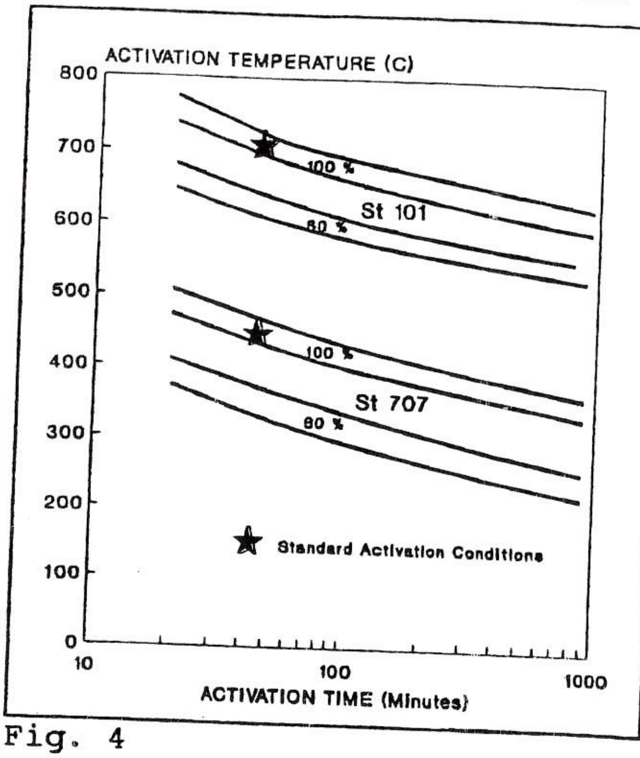


Fig. 4

However, in practical cases it is not always possible to use the "ideal" time/temperature combinations. Suitable operation modes can be programmed where only a "partial" activation is considered sufficient. This results in a condition where the getter surface develops less than full pumping speed. The condition which gives 60% of the optimum pumping speed is also shown in fig. 4.

The activation process should be carried out after a 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 cartridge. This is due to the physisorbed 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.

Desorbed gases include H_2, H_2O, CO, CO_2, CH_4, and eventually H_2, due to the behavior of the getter material toward this gas.
In order to minimize this gas evolution, baking the system, with the getter cartridge maintained at a relatively higher temperature than the other components of the system, 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 a real 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, r.f. discharges between the heater and other pump elements and contamination of the gettering material due to the sorption of active gases during the activation process.

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 30 minutes usually needed if full power is applied at the beginning.

OPERATION OF SORB-AC CARTRIDGE PUMPS

When activated, the SORB-AC cartridge pump can be operated at various temperatures according to the load of active gases, taking into account two main facts: First, the higher the temperature, the higher the diffusivity of the sorbed gases into the bulk of the getter material. Second, the higher the temperature, the higher the H_2 equilibrium pressure.

High temperature operation is preferred when high gas loads are present. In this way it is possible to maintain a high diffusion rate and consequently a constant pumping speed for all the active gases. Temperatures in the range of 280° C and 400° C are typically suggested for the st 707 and St 101 materials respectively. High getter temperature is usually not compatible with UHV operation due to the desorption of hydrogen from the getter material. For example the use of an St 707 cartridge at 280°C will cause a hydrogen equilibrium pressure on the getter material in the range of 10^{-7}-10^{-8} torr due to the residual H_2 content of the alloy and depending on the presence of additional pumping devices.

Room temperature operation is indicated when the gas load is low, and for the reason mentioned above, it is mandatory when the operating pressure is below 10^{-8} torr. When pumping high purity hydrogen, operation at room temperature is also possible with high gas loads, due to the high diffusivity of H_2 at that temperature. In this latter case, a careful evaluation of the gas load and the quantity of gas sorbed is necessary to avoid possible embrittlement.

The pumping speed curves of the various pump models are shown in **Appendix B**. The curves refer to the pump configuration with water cooled body, standard activation conditions and are referred to the St 707 alloy cartridges. The initial pumping speed values of the pump models which use St 101 alloy cartridges are typically 10 % higher than the equivalent St 707 models. On the other hand, the room temperature capacity values of the pump models which use St 707 cartridges are 15 % higher than the equivalent St 101 models.

REACTIVATION OF SORB-AC CARTRIDGE PUMPS

The reactivation of the getter cartridge of a SORB-AC Pump is necessary when the cartridge is exposed to air or when its pumping speed falls below acceptable limits. In both cases 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 and has the same characteristics of the first one.
If the reactivation follows normal operation in vacuum without air venting, it may be shorter and carried out at a lower temperature (indicatively 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 cartridge must be replaced.
One way to determine if the getter cartridge is exhausted is when the following quantities of gas have been sorbed:
$q(CO) + q(CO_2) + q(O_2)/5 + q(H_2O)/5 + q(N_2) = 9$ q=liter torr/g

REGENERATION OF SORB-AC CARTRIDGE PUMPS

Hydrogen and hydrogen isotopes sorbed by the cartridge can be released from the getter material through a regeneration treatment.

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 "embrittlement limit" of 20 liter torr/g.

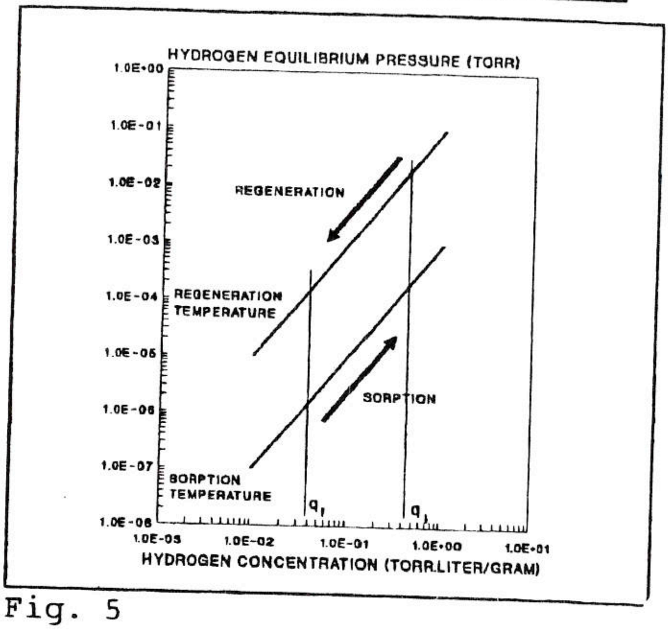


Fig. 5

Embrittlement of the material takes place when the quantity of hydrogen sorbed in the getter material is high enough to modify the mechanical characteristics of the alloy causing it to flake in small particles from the substrate.
The process of hydrogen sorption and successive desorption (regeneration), can be visualized on the Sieverts' plots of each specific alloy (see fig. 5).
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. 5 one can see that the regeneration process will be more efficacious at higher temperature.

The time necessary for the regeneration of a cartridge is given by the expression:

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

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 liter torr/g
q_i = initial H_2 concentration, in liter torr/g
A = 4.82 for St 101 alloy cartridges
4.8 for St 707 alloy cartridges
B = 7280 for St 101 alloy cartridges
6116 for St 707 alloy cartridges
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 again) but only partial (for example 90%).
Thus if a given amount of hydrogen must be sorbed in each cycle (for instance 10 torrl/g), it is much better to operate in the q_f=2 - q_i=12 mode, than in the q_f=0 - q_i=10 one.

SPECIAL INSTRUCTIONS

Thermal fatigue

The reactivation and regeneration processes can be performed many, but not an infinite number of times. Due to thermal fatigue in the getter coated strips, peel-off of the gettering powder eventually occurs, which mainly depends on the temperature and on the duration of the heating process.

Fig. 6 shows the number of permissible thermal cycles, at 700°C, 600°C and 500°C as a function of the hold time, before peel-off starts to occur.

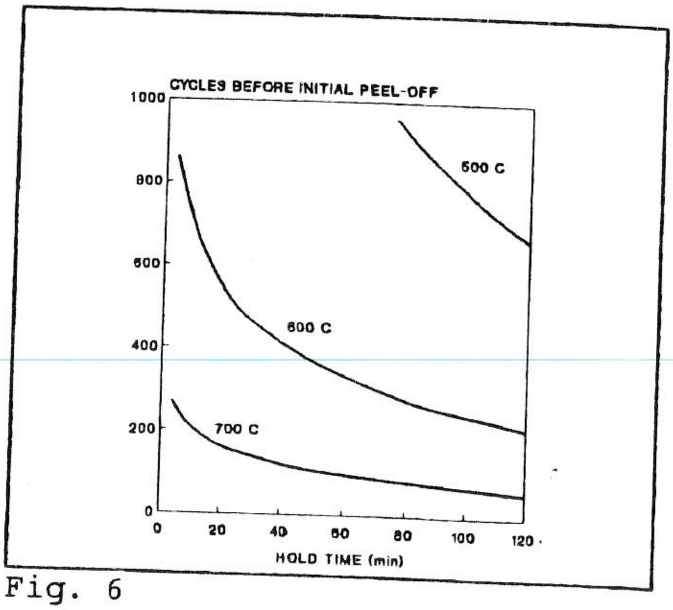


Fig. 6

Air venting

Opening the vacuum chamber where SORB-AC pumps are mounted should be performed only when the getter material is at room temperature or, at least, below 50°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.
After about 30 air exposures at room temperature followed by reactivation, St 101 cartridges at 400°C and St 707 cartridges at 280°C still have a pumping speed for hydrogen of about 40% of the original pumping speed.

If dry nitrogen is used instead of air, the pumping speed reduction, after the same number of exposures, appears to be smaller (residual pumping speed still about 80% of the initial value, see fig. 7). 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.

Vacuum failure during activation or regeneration

During activation or regeneration of SORB-AC cartridge pumps, air must not be allowed to suddenly enter the vacuum system. Such an occurrence could cause "burning" (i.e. a quick oxidation) of the coated strip. This happens if the temperature of the cartridge at the moment of vacuum failure is above 450°C in the case of St 101 cartridges and above 200°C in the case of St 707 cartridges.

The burning is 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. For example, only negligible damage would result in St 101 cartridges if the air exposure occurred at 300°C for a maximum of 2 to 3 minutes. Also, in these cases, a more efficient recovery of gettering efficiency can be obtained by using a reactivation procedure at a temperature higher than the normally adopted value.

Excessive heating of the cartridge

The non-magnetic cartridges which use constantan as a substrate material, should not be heated to temperature over 750°C to avoid possible copper or manganese evaporation from the material. At temperatures over 850°C, it is possible to have the formation of eutectic compounds between copper and zirconium which could cause melting of the cartridge.

Mechanical shocks

Due to the mechanical characteristics of the insulating elements of the heater (ceramic spacers) particular care must be observed in handling during assembly (and removal) of the pumping system. Accidental dropping and similar mechanical shock could result in breakage of the insulating elements with possible short circuits of the electrical path.

MAINTENANCE OPERATION

Electrical Connector Removal

The Electrical Connector used for SORB-AC Cartridge Pumps must be removed if, during a baking treatment, it has to withstand temperatures over 100°C. The electrical connector must be removed also to allow the replacement of the thermocouple.

For this operation refer to fig. 8.

Remove the three grommet nuts (1) and lift off the washers (2).

Lift off the external ring (3) and the protection grid (4).

Remove the socked-head screw (5) and the washer (6) used to fix the dead earth cable (7).

Lift off the thermocouple female connector (8) from the male one.

Remove the 3 nuts (9) and lift off the female connectors (10) from the current feedthroughs.

Lift off the electrical connector disk (11).

Only for the thermocouple replacement

CAUTION: The getter cartridge can not be exposed to air at temperatures over 35° C when still active.

Remove the bolts (13), the washers (14) and the nuts (12) used to fix the miniconflat flange on which the thermocouple sheath is vacuum brazed.

Remove the faulty thermocouple from the pump.

Replace the gasket (16) and insert the new thermocouple in the tubulation in the cartridge (taking care not to damage the thermocouple sheath).

Reassemble the thermocouple flange to the pump.

Electrical Connector Assembly

After the baking treatment or the thermocouple replacement it is necessary to replace the electrical connector.
For this operation refers to fig. 8.

Replace the electrical connector disk (11) and insert the female connectors (10) into the current feedthroughs.

Fix the three nuts (9).

Insert the thermocouple female connector (8) into the male one.

Fix the dead earth cable (7) to the pump flange using the washer (6) and the socked-head screw (5).

Insert the protection grid (4) and the external ring (3) and fix it with the proper grommet nuts (1) and washers (2).

Check with an ohmmeter, at the electrical connector, the continuity of the heater and of the thermocouple.

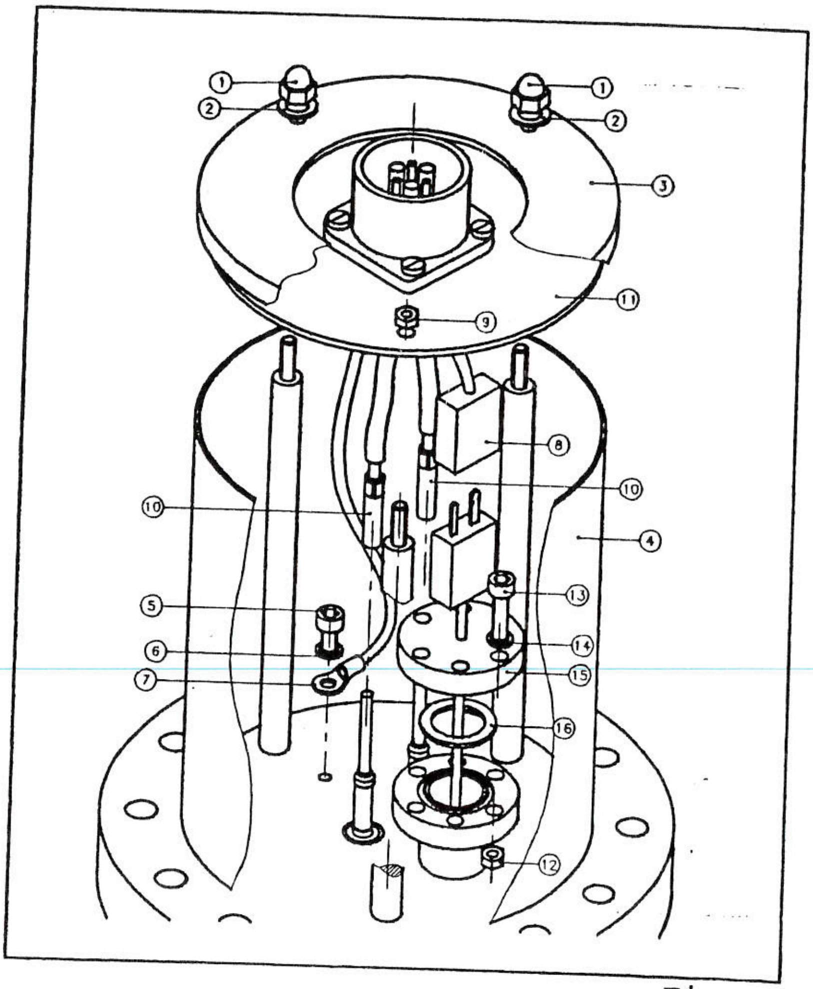


Fig. 8

Check with an ohmmeter that the heater and the thermocouple are insulated from the flange.

Heater substitution

When the heater needs to be substituted, proceed according to the following instructions and referring to Fig. 9.

Remove the terminals (1) between the heater filament and the copper feedthroughs by loosening the terminal screws (2).

Remove completely the 4 socket head screws (4) and washers (3) which fix the heater base to the bottom of the flange.

Lift off the heater (5) from the flange taking care not to damage the sheath of the thermocouple (6) (not present in the GP 50 pump) which must slide out of one of the heater base supporting spacers (7).

Position the new heater on the base flange, inserting the thermocouple (6) in one of the heater base supporting spacers (7), and the copper feedthroughs in the terminals (1) connecting the heater filament.

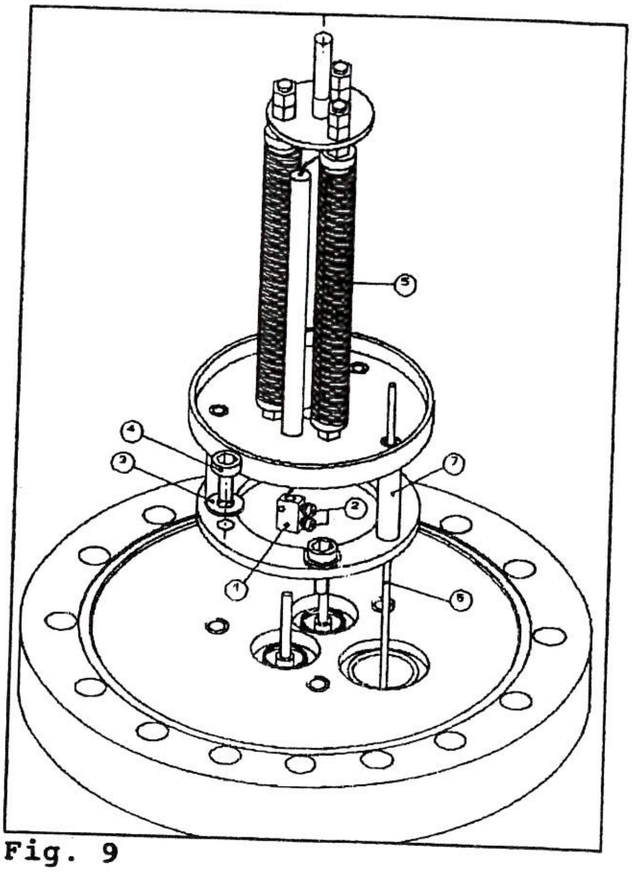
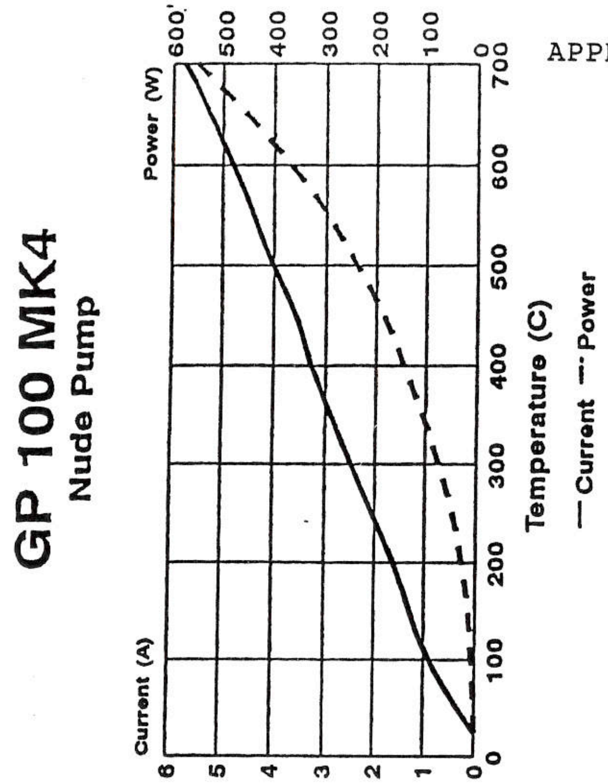


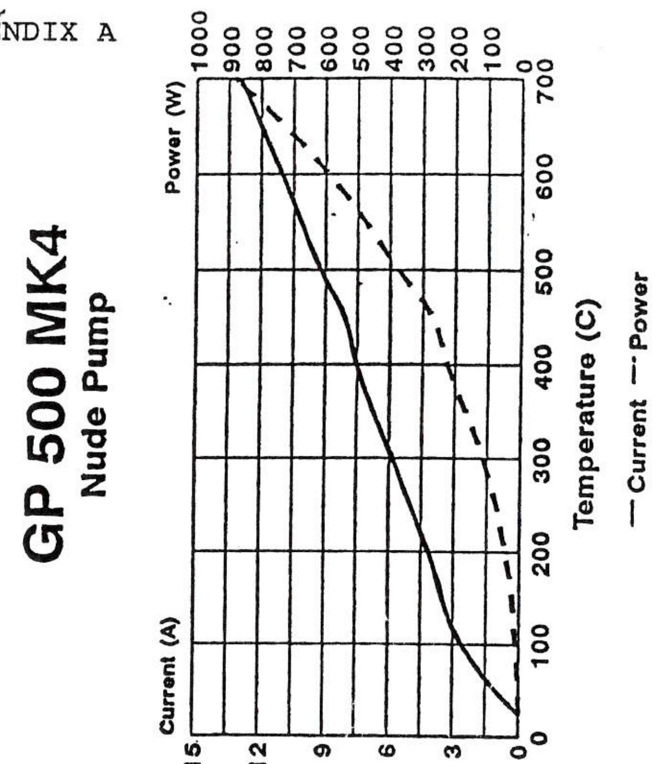
Fig. 9

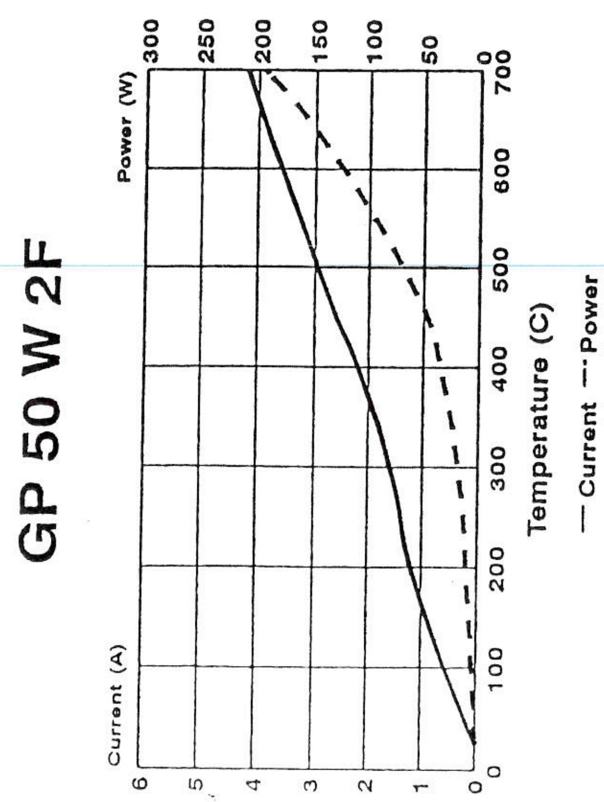
Tighten the screws (2) of the terminals.

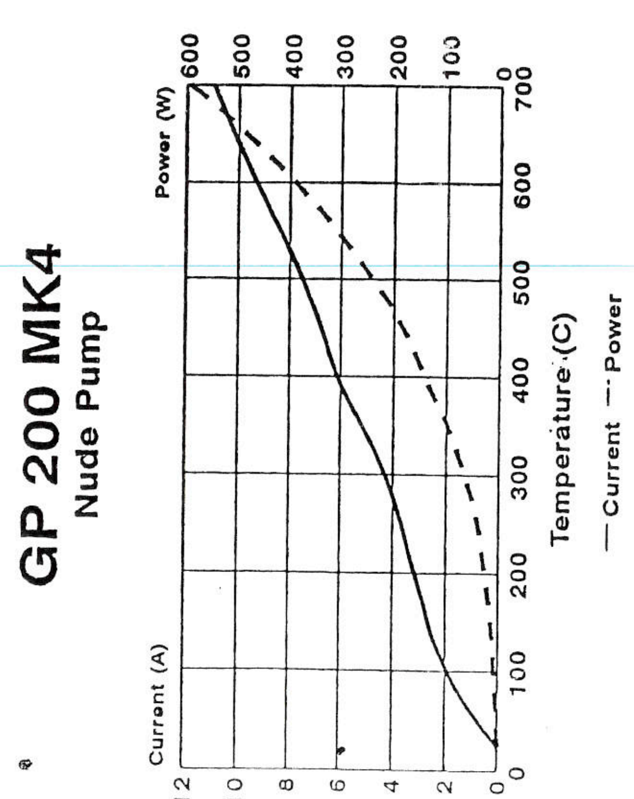
Insert and tighten the 4 washers (3) and socked head screws (4) to fix the base of the heater (5) to the bottom of the flange.

APPENDIX A

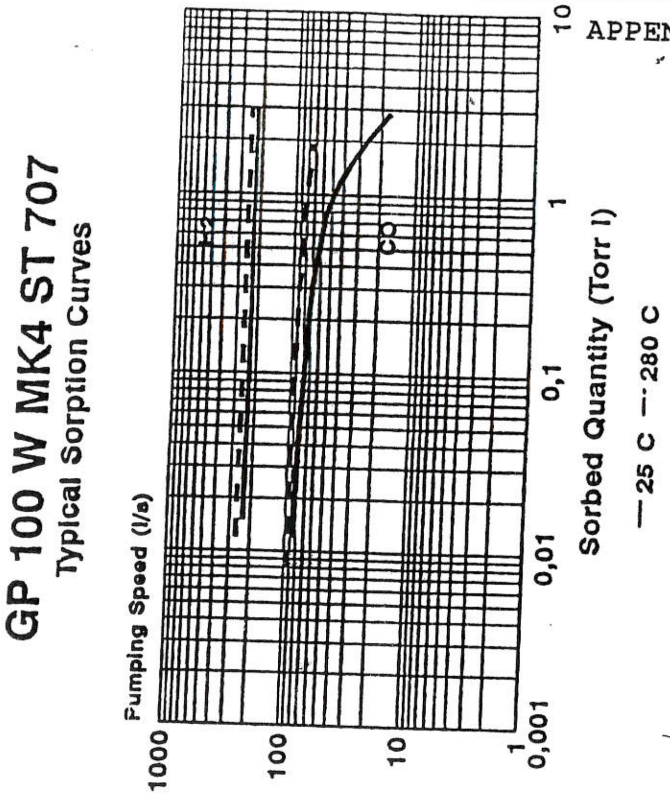

GP 100 MK4
Nude Pump
Current (A)
Power (W)
Temperature (C)
— Current - - Power

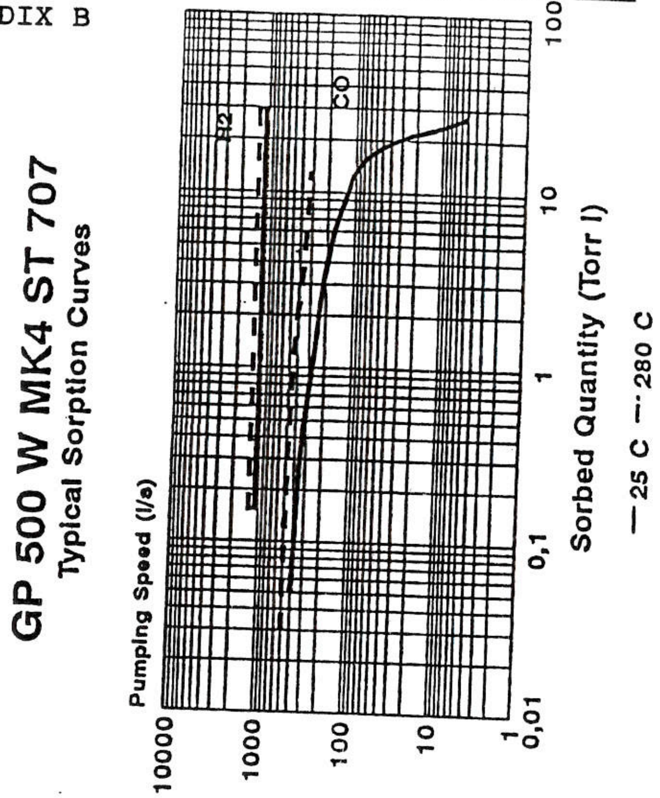

GP 500 MK4
Nude Pump
Current (A)
Power (W)
Temperature (C)
— Current - - Power

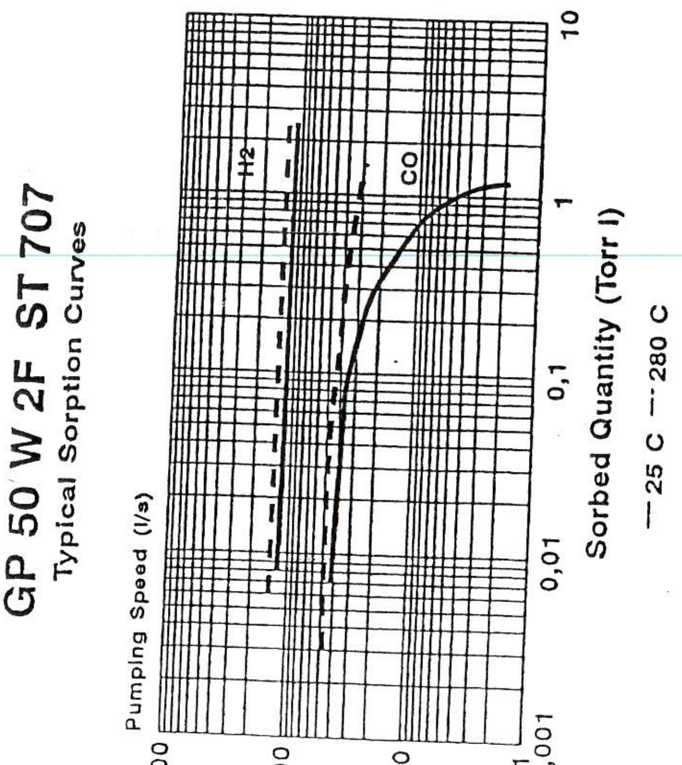

GP 50 W 2F
Current (A)
Power (W)
Temperature (C)
— Current - - Power

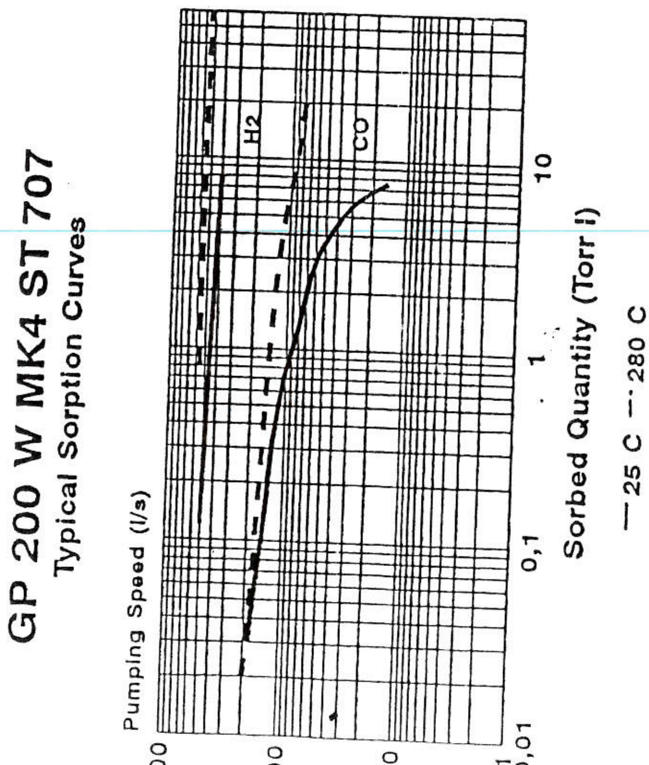

GP 200 MK4
Nude Pump
Current (A)
Power (W)
Temperature (C)
— Current - - Power

APPENDIX B


GP 100 W MK4 ST 707
Typical Sorption Curves
Pumping Speed (l/s)
1000
100
10
1
0,001
0,01
0,1
1
10
H2
CO
Sorbed Quantity (Torr l)
— 25 C —·280 C


GP 500 W MK4 ST 707
Typical Sorption Curves
Pumping Speed (l/s)
10000
1000
100
10
1
0,01
0,1
1
10
100
H2
CO
Sorbed Quantity (Torr l)
— 25 C —·280 C


GP 50 W 2F ST 707
Typical Sorption Curves
Pumping Speed (l/s)
1000
100
10
1
0,001
0,01
0,1
1
10
H2
CO
Sorbed Quantity (Torr l)
— 25 C —·280 C


GP 200 W MK4 ST 707
Typical Sorption Curves
Pumping Speed (l/s)
1000
100
10
1
0,01
0,1
1
10
H2
CO
Sorbed Quantity (Torr l)
— 25 C —·280 C