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EXPLORAVAC CHAMBER BAKEOUT AND DEGASSING

APPLICATION NOTES

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1. INTRODUCTION

The purpose of this document is to educate and instruct the ExploraVac operator about degas and bakeout procedures. These two processes are used to clean the chamber and items within it at a molecular level far beyond what can be achieved with simple solvents and scrubbing. Non-Exploravac users can also benefit from this information.

Vacuum chambers must be kept very clean in order to efficiently obtain and maintain desired vacuum levels. Molecules, including water, organic compounds used in experiments or processes, reaction products, grease from fingerprints, backstreamed oil from pumps, and film left over from cleaning solvents such as isopropyl alcohol (IPA) and acetone become stuck on all surfaces inside the chamber. These residual contaminants create a vapor pressure which works against the vacuum pump, slowing pump down and causing the pump to be unable to reach its ultimate base pressure.

Both degassing and bakeout procedures use a combination of heat and vacuum to strip off these undesirable contaminants which are then drawn through the vacuum pump and eliminated through the pump's exhaust.

The term "degassing" is normally used to describe the process of removing these molecules from articles inside the chamber. The term "bakeout" is normally used to describe the process of stripping these molecules from the chamber itself.

The amount of degassing or bakeout needed depends on the cleanliness required. The ability to degas or bakeout to the desired level depends on the vacuum pump's capability, the type and amount of heat that the materials, including the chamber seals, can tolerate, and the chemistry of the molecules to be eliminated.

Degassing and bakeout both require the ExploraVac to have either a rough or rough and high vacuum pump, a thermal platen or resistive or infrared chamber heating, or both. The use of purge gas further improves the effectiveness of either procedure.

The chamber should always be thoroughly cleaned with solvents such as Isopropyl alcohol (IPA), acetone, or other degreasers, and physical scrubbing to remove gross contaminants, prior to performing any experiment, process or procedure.

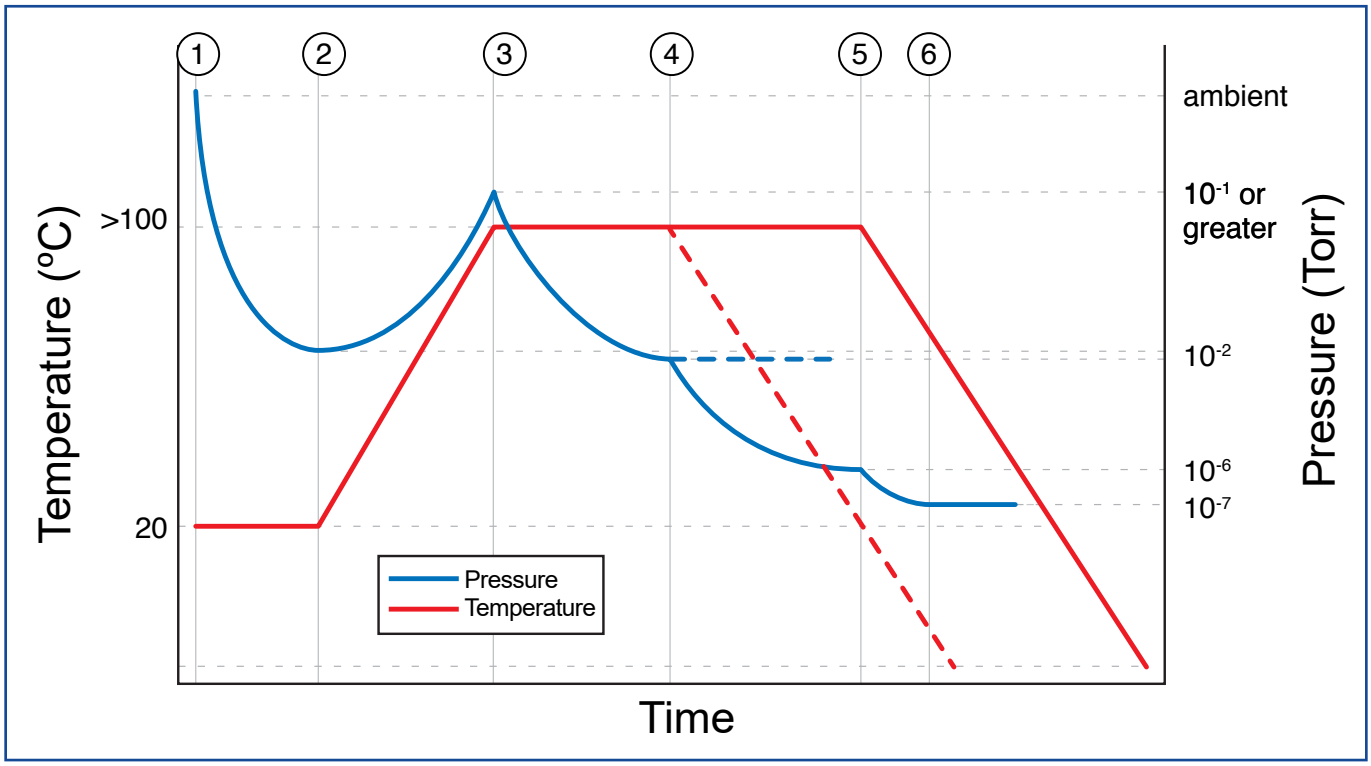
Any objects, including any fixtures, that go into the chamber should be similarly cleaned prior to them being placed inside the chamber.

Although degassing and bakeout procedures can be completed manually by allowing ample time for each step of the process to complete, a much better and faster method is to watch changes in the chamber pressure as the process progresses. The fastest and easiest method to degas or bakeout an ExploraVac chamber is to use the premium version of AutoExplor to write a recipe which progresses as certain pressure and temperature conditions are met. The recipes can be stored and run whenever needed.

The general methodology of degassing and baking out a chamber is discussed in Section 2. Sections 3 and 4 provide step-by-step instructions for generic or typical degassing and bakeout procedures. These procedures may be modified for the user's particular requirements and the Exploravac system configuration they have. The procedures outlined can be used to build AutoExplor recipes for repetitive use.

2. GENERAL METHODOLOGY

The graph below illustrates the chamber temperature and pressure during a degas or bakeout procedure. All values are approximate and the graph is not to scale. Actual results vary.



(1) The chamber is at ambient atmosphere and temperature. Roughing begins.

(2) The roughing pump reaches its base pressure which may be its rated ultimate pressure or somewhat above due to the vapor pressure of contaminants. Platen or chamber heating begins. As volatiles are cooked off, their vapor pressure rises raising the chamber pressure.

(3) The temperature reaches >100ºC and is maintained (soaked). Chamber pressure increases to 10^{-1} Torr or higher depending on the amount of volatiles present. As volatiles are removed, their vapor pressure is diminished and chamber pressure begins to decrease.

(4) The Roughing pump pressure returns to its original base pressure, or slightly below when all volatiles that can be removed at rough vacuum have been pumped out of the chamber. If the ExploraVac does not have a high vacuum pump, roughing continues while the platen or chamber is cooled or heat is turned off (dashed red line). The pressure stabilizes at or close to the roughing pump's rated ultimate pressure (blue dashed line).

If the Exploravac has a high vacuum pump, temperature continues to be maintained and high vacuum is turned on. As the pressure decreases, more volatiles are expunged.

(5) The high vacuum pump reaches its base pressure (≈10^{-6} Torr), typically one decade above its rated ultimate pressure. The platen or chamber is cooled or turned off. The high vacuum pump remains on. As cooling proceeds, the vapor pressure of any remaining volatiles decreases thereby reducing the chamber pressure by as much as a decade, to its ultimate pressure.

(6) When the high vacuum pump's ultimate pressure is reached, the chamber pressure stabilizes. When the platen temperature reaches room temperature, the procedure is complete.

3. DEGASSING PROCEDURE

Objects to be degassed should be directly affixed to the platen for maximum thermal conduction.

It is not required for the chamber to have wall or infrared heaters to degas objects. In fact, when the platen is heated under vacuum, volatiles will naturally move from the higher temperature of the platen to the cooler temperature of the chamber walls. If the contaminants are not removed through the pump, they get deposited on the chamber walls.

Objects can be solids, such as metal parts destined for use in semiconductor applications, or a combination of solids and liquids, such as epoxy and composite materials.

For ExploraVac systems with high vacuum and a heated platen:

Clean items and platen with IPA and scrub as necessary.

1. With chamber at ambient pressure and temperature, affix items to platen.
2. Turn on ROUGH.
3. Make platen setpoint temperature as hot as the items or process can tolerate at MAXimum ramp speed.
4. When pressure is below 50 mTorr, turn on PLATEN TEMP.
5. When the platen reaches the temperature setpoint, soak (maintain) the temperature until the pressure decreases and becomes stable (<25 mTorr), typically 2-12 hours.
6. Turn on HI VAC.
7. Continue soaking platen temperature until the pressure decreases and becomes stable at $<1 \times 10^{-6}$ Torr or for 2-12 hours.
8. Maintain high vacuum and cool or turn off platen until it reaches room temperature.

For ExploraVac's that also have pressure control and the purge gas option:

If there are stubborn contaminants or deep crevasses in the objects, it may be helpful to perform multiple pump-purge cycles. The purge gas can help desorb and strip contaminants away from object surfaces at higher pressures more effectively than with pure vacuum.

Pump-purge procedure:

1. Rough vacuum to 100 mTorr
2. Purge nitrogen (or other) gas to 700 Torr
3. Repeat Steps 1 and 2 at least three times
4. Rough vacuum to 25 to 50 mTorr
5. High vacuum to desired pressure

Some users find that there is an ideal pressure to remove contaminants where the vacuum is sufficient to remove them but the presence of purge gas is still helpful for its scrubbing action.

In this case, the active purge pressure mode can be used. Active purge continuously flows purge gas through the chamber and out the pump exhaust at the maximum possible sustainable rate while maintaining a set pressure inside the chamber. Active purge works in the roughing vacuum regime only.

Active purge procedure:

1. Active purge to predetermined optimal pressure.
2. Continue active purge for X hours.

   The length of time for active purge to continue cannot be determined by pressure measurement the way previous methods have been described. The duration must be experientially determined. The length of time needed will depend on several factors including how contaminated the chamber is initially, the type of contamination, the target pressure, and the pumping speed.

   To determine how long active purge should be continued, start active purging for a number of hours. Then, stop active purging and run normally to determine if the ultimate roughing pump pressure is realized, or if the pumpdown speed is acceptable. If not, restart active purging and continue it for a few more hours.

3. Rough vacuum to 25 to 50 mTorr.
4. High vacuum to desired pressure.

Use the gas ballast to desorb water:

Water can accumulate in the rough pump gas ballast and reduce performance.

If the items have a lot of adsorbed water on them, it may be helpful to turn the roughing pump gas ballast knob on to its open position either during the pumpdown or once rough vacuum has been achieved for more than 30 minutes.

This is not necessary when utilizing pump-purge or active purge procedures.

4. BAKEOUT PROCEDURE

Bakeout can be performed with the chamber empty, or with items inside. When items are inside the chamber, the bakeout also degasses the items within.

To get the best and fastest bakeout, the Exploravac must have high vacuum capability, a thermal platen, resistive or infrared chamber heating, and the premium version of AutoExplor software. The bakeout procedure below describes the procedure with an Exploravac so equipped. Note that baking out a chamber does not require high vacuum or chamber heating. In this case, the bakeout will not eliminate as many contaminants (which may be accpetable) and will be somewhat slower since it uses the platen to heat the chamber walls.

Bakeout with ExploraVac chamber empty, high vacuum, and chamber heating:

1. Remove all items. Clean the chamber with IPA and scrub as necessary.
2. Rough vacuum until pressure reaches 0.025 Torr.
3. Continue rough vacuum and turn on Platen heating to 200°C.
4. Turn on chamber heating to 140°C.
5. Run until platen and chamber have both reached final temperature and the pressure is < 0.022 Torr.
    a. These temperatures may be adjusted. At any rate, heat to at least 100°C..
    b. During a bakeout, it is good practice to ramp up the platen temperature while at rough vacuum to avoid getting gunk in the high vacuum pump. If the chamber is already clean, this is not necessary.
6. Platen and chamber heating remain on. Turn on high vacuum until pressure is < $5x10^{-6}$ Torr.
    a. Reduce this pressure if this level of vacuum is not required.
    b. Once the temperature drops in the next step, the vacuum level will drop with it, by about one decade.
7. High Vacuum remains on. Chamber heating is turned off. Platen temperature control is set to 25°C. Run until the chamber and platen temperature have reached room temperature.

This recipe will clean any fingerprints, lint, or other volatile material out of the chamber. When the chamber is vented or purged, it is desirable to use nitrogen or dry air if possible. The dry gas provides a short window of protection (1-2 minutes) while samples are loaded into the chamber, preventing water from adsorbing back onto the chamber surfaces.

If the chamber has organic deposits on surfaces, even after cleaning and scrubbing, bakeout may leave non-volatile carbon residue or gunk on these surfaces. It is best to clean, scrub with lint free cloth or plastic scraper, and rebake the chamber again to eliminate that material.

For any items which degas, the user will normally want to degas, bakeout, insert the next item, and repeat. Bakeout may not need to be done every cycle, but often enough to keep the chamber clean enough to efficiently reach target pressure.

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