



## OIL TECH SERVICES, INC.

800 Wilcrest, Suite 100  
Houston, TX 77042-1359  
(713) 789-5144  
Email:

[sales@oiltechservicesinc.com](mailto:sales@oiltechservicesinc.com)

Website: [www.oiltechservicesinc.com](http://www.oiltechservicesinc.com)

5 January 2012, Rev. 1

### **Temperature-programmed Desorption (TPD) Bake-Out Process**

The vacuum bake-out process is a combination of several operations performed to achieve a high vacuum sealed within the annular space between the inner and outer tubes of a VIT assembly. This high vacuum including the multiple thermal radiation barriers wrapped around the inner tube becomes the thermal insulation of the VIT joint.

The processes necessary in the vacuum bake-out are:

- Cleaning (Grit Blasting)
- Assembly (Getter Installation)
- Vacuum Bake-Out (Temperature Programmed Desorption)
- Sealing

Cleaning: To reduce off gassing during the vacuum bake-out process mill scale, oils, greases, and other volatile hydrocarbons and contamination are removed by grit blasting of the inside diameter of the outer tube and the outside diameter of the inner tube.

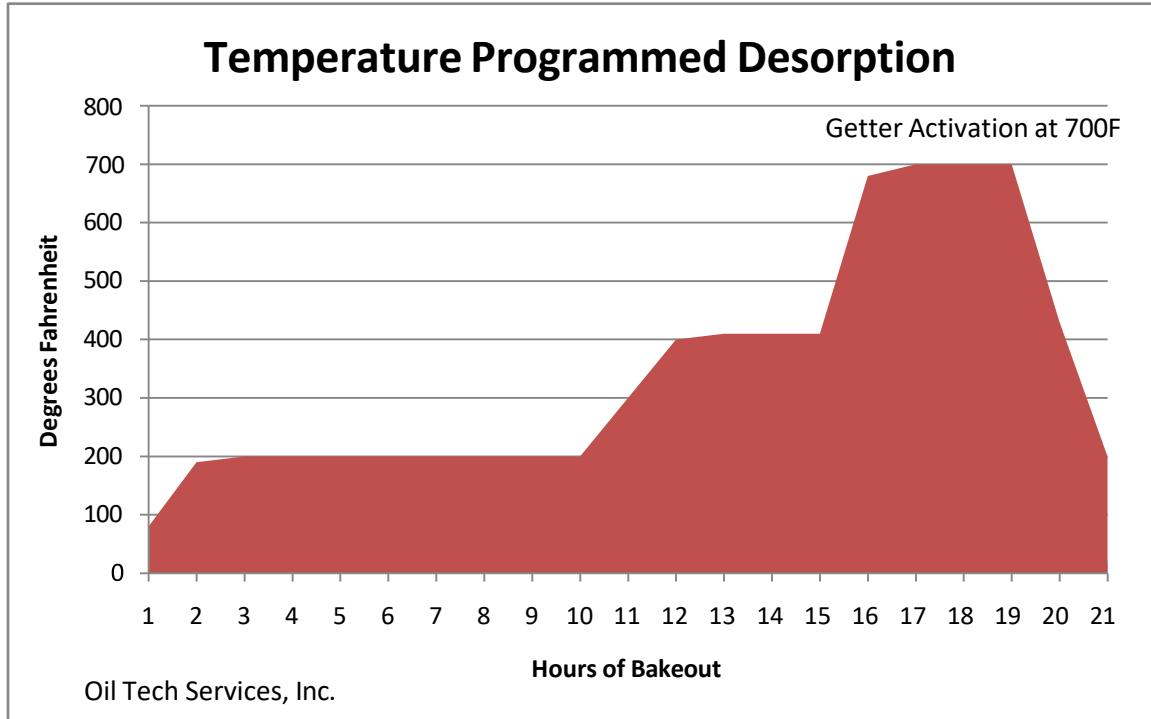
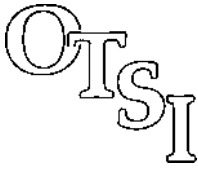
Getter Installation: During assembly (wrapping of multiple layers of aluminum foil separated by ceramic paper and fiberglass cloth) the Getter is installed within the closest aluminum foil wrap to the OD of the inner tube.

Vacuum Bake-Out (Temperature Programmed Desorption): After the assembled VIT joint is welded at both ends sealing the vacuum and connecting the inner tube to the outer tube, the full length of the VIT joint, including the vacuum fixture, is positioned inside a bake-out oven. The VIT joint has a vacuum port drilled into the outer tube approximately 18-inches from the tube end. A special fixture is installed over vacuum port and attached to the manifold / vacuum pump system. The mechanical vacuum pump is started, the oven is turned on and a programmed time-temperature cycle takes over control of the oven and process.

This programmed temperature desorption cycle causes the mechanical (through the vacuum pump) removal of gases sorbed and occluded into the steel surface and held within the inner and outer tube internal matrix structure. Oven temperature is slowly increased to maximize removal of these sorbed gasses from the carbon steel inner and outer tubes: This time period is approximately 12-15 hours.

Vacuum levels are continuously measured through the fixture covering the vacuum port. Once the mechanical pump has lowered the annulus pressure to a medium vacuum level, the oven temperature is quickly increased causing the passivation layer protecting the Getter from premature activation to be released allowing any remaining gas molecules within the annulus to react with the Getter alloy thereby chemically pumping the vacuum to approximately 15 milli-Torr.

Once the desired vacuum level is obtained, sealing of the vacuum port is completed. A seal plug pre-installed within the vacuum port fixture is pressed into the vacuum port (hole) and a metal-to-metal seal is confirmed using a Helium leak detection process. Once the metal-to-metal seal is confirmed, the fixture is removed, and the plug is seal welded around the plug circumference.



Additional comments: One or more insulated tubing manufacturers do not use a bakeout process in their manufacturing. The following information is presented to help the engineer understand the importance to this step in the manufacture of a high vacuum system.

Getters are quite forgiving in terms of the activation process and how they are activated. If an insulated tube was to rely on the first steam cycle to activate the getter, steam temperature will need to be 200°C (400°F) or hotter. According to SAES Getters general activation recommendations the suggested temperature of activation is 450°C (845°F) for a period of 10-15 minutes and longer periods at lower temperatures.

This bake-out process is a common practice for all high vacuum systems and a critical process to cause the passive film protecting the Getter from premature activation to diffuse into the bulk of the Getter making the Getter surface active for sorption and insuring maximum residual Getter capacity. It is essential to heat the Getter under a medium vacuum of  $1 \times 10^{-3}$  Torr or better to prevent the accumulation of gases from building up another passivation layer and stopping the gettering action.

The getter's task is to maintain the vacuum in the annulus of the insulated tube: The better the bake out the lower the amount of residual gas. The challenge for the manufacturer is to have sufficient getter material to exceed the amount of gas evolving from the inner and outer tubes during the life of the product. A clean inner tube OD and outer tube ID (such as a grit blasted surface) makes this easier. An insulated tube manufactured without a bakeout cycle needs substantially more Getter and assurance there is sufficient temperature and time during the initial steam cycle to properly activate the Getter avoiding contamination of the Getter surface with a new passivation coating stopping the gettering action.