Technical Information Bulletin
Number AL-119

PRODUCT NO. **Z10,361-6**

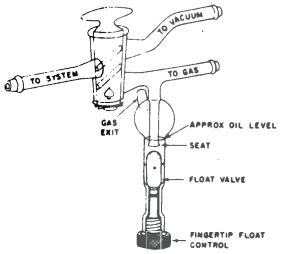
The Firestone* Valve

A Rapid-Purge Valve for Controlled-Atmosphere Work

- **No air suck back (liquid seal)**
- **No pressure monitoring necessary**
- **No mercury used**
- **No reducing valve needed on gas source**
- **No high-vacuum source necessary**
- **No fear of pressure buildup**
- **No watching once purge is completed**

VALVE, Rapid-Purge, Firestone*

A rapid, efficient and foolproof purge valve for 100% replacement of air in reaction vessels with any desired gas (N_2, H_2, Ar, Cl_2, etc.). No need to watch manometers, hand-control gas flow, or install warning systems to prevent accidents. Expensive gases are conserved because, once purging is complete, the flow can be cut to almost zero. This valve is so inexpensive, it is possible to run all reactions under nitrogen as easily as not.



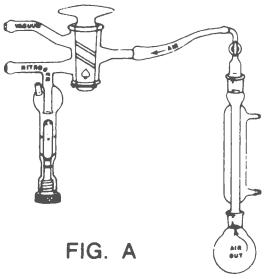


FIG. A

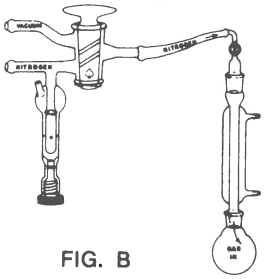


FIG. B

Connect reaction vessel, house vacuum, and purge gas to valve *via* 10-mm o.d. connections. On vacuum cycle (Fig. A) air is removed. On purge cycle, (Fig. B) the float valve first closes. When filling is complete, the float valve reopens to prevent pressure buildup. Thus, a simple half-turn of the stopcock alternates the reaction system from vacuum to gas flow as fast as desired. In small systems, a complete cycle takes as little as 1-2 sec. With only 0.5-atmosphere house vacuum, ten cycles removes all but 0.5^{10} atmospheres of air. About 70 cycles will get you down to the last molecule of oxygen. After purging, the system is kept under slight positive pressure indefinitely, with a slow bubbling of the gas to prevent diffusion of air past the joints. If the reaction evolves a gas, no pressure builds up, and it may even be collected and measured while maintaining a controlled atmosphere. Proper selection of the liquid for the seal allows purging with any gas that does not react with glass. (Liquid is not supplied.)

Operating Instructions

1) Clamp the valve securely using a standard laboratory clamp.
2) If not already assembled, insert fingertip float control into place. Make sure float is NOT against seat. A distance of 1-2 mm is usually adequate depending on oil used, gas flow, etc.
3) Select a non-volatile oil that is somewhat viscous. Mineral oil works well. For reactive gases, a fluorocarbon oil can be used.
4) With dropper, add enough oil through gas exit so it rises a few millimeters above seat. With some heavy oils such as silicone, float may actually "float."
5) Connect tubing from vacuum line, purge-gas line and vessel to proper arms of valve (see Fig. A).
6) Turn stopcock on valve so bore is open to purge-gas line. Start purge gas. Bubbling should occur at float vlave as excess gas escapes through gas exit. Start vacuum pump.
7) OPERATION: Turn stopcock 180° to vacuum line (Fig. A). Air will be removed from vessel. After a few seconds, turn stopcock 180° to purge-gas line (Fig. B). The float valve will first close to prevent air from entering system while the purge gas fills the vessel. When filling is complete, float valve automatically opens allowing excess gas to escape and thus prevents pressure buildup. Repeat cycle as often as necessary for a thorough purge. After purging, leave stopcock open to the purge-gas line and reduce gas flow to very low. The system will remain protected from the atmosphere indefinitely.
8) If excessive oil squirts up the tube during closing of the valve while going from vacuum to gas-purge, reduce the free play of the float valve slightly by screwing in the fingertip control. If gas has difficulty escaping, increase the free play.
9) To clean, remove fingertip control and flush with solvent. Reassemble, using fresh oil.

*Designed by Dr. Raymond Firestone, Merck, Sharpe & Dohme Research Labs.
(U.S. Patent 4,131,129)