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Tech Brief: An Explanation of Leak Rate

By: Charles D. Dern, P.E.

Introduction

Personnel involved with freeze-drying technology must deal with evacuated vessels and the testing and criteria that assure that a vessel is free of significant leaks. Traditionally, industries that are involved with vessels subjected to evacuation have measured the relative "tightness" of these vessels by one of two criteria: "rate of rise" or "leak rate." At first glance, it may appear that these are simply two different terms for the same thing. However, a closer look reveals that this is not the case.

Basic Definitions

Rate of rise is the amount of pressure change in an evacuated vessel over a given period. (i.e. millitorr per minute, mbar per second, etc.) For example, if one evacuates a vessel to 100 mTorr, closes the vacuum valve, and then observes that after one minute, the pressure is 105 mTorr, then the rate of rise is quite simply 5 mTorr per minute. Mathematically the formula is:

$$\textbf{Rate of Rise} = \frac{\text{Finish Pressure - Start Pressure}}{\text{Elapsed Time}}$$

Rates of rise can be performed at any pressure below the local ambient pressure and can be done for any length of time. The best pressure at which to test is at the expected working pressure of the vessel. Also, in most cases, the longer the elapsed time, the more assurance one will have of obtaining an accurate result.

However, rates of rise, no matter how carefully done, are not an accurate basis for comparing tightness among vessels of various sizes. This is because rate of rise does not account for the volumes of the vessels in question. If a 10 ft^3 vessel and a 100 ft^3 vessel have the same rate of rise, a greater amount of gas must leak into the 100 ft^3 vessel to raise the pressure the same amount. (In fact, ten times as much.)

To do an accurate comparison therefore, one must account for the respective volumes of the vessels. This is accomplished by a "leak rate." Obtaining a leak rate involves multiplying "rate of rise" by the system volume. Thus, if a rate of rise is expressed in millitorr per minute (mTorr/min.), then a leak rate is expressed as millitorr x cubic feet per minute (mTorr-ft^3/min.) The general formula is:

$$\textbf{Leak Rate} = \frac{\text{(Finish Pressure-Start Pressure) x Volume}}{\text{Elapsed Time}}$$

OR

$$\textbf{Leak Rate} = \text{Rate of Rise x Volume}$$

Example: Assume that vessels of 10 ft^3 and 100 ft^3 are both evacuated to 100 millitorrs and are maintained at a constant temperature. At this pressure, the 10 ft^3 vessel will contain .00132 standard cubic feet (SCF) of gas and the 100 ft^3 vessel will contain .0132 SCF of gas. Assume further that each vessel has an identical leak that allows .001 SCF of gas in one minute into each vessel. At the end of one minute, the 10 ft^3 vessel would contain .00232 SCF of gas and be a pressure of 176 mTorr for a rate of rise of 76 mTorr/min. The 100 ft^3 vessel will contain .0142 SCF of gas and be at a pressure of 107.6 mTorr for a rate of rise of 7.6 mTorr/min. Both chambers have the same leak yet the smaller chamber has the greater rate of rise.

However if the rates of rise are multiplied by the respective chamber volumes one obtains:

$$10\ ft^3 \times 76\ mTorr/min. = 760\ mTorr\text{-}ft^3/min.$$

and

$$100\ ft^3 \times 7.6\ mTorr/min. = 760\ mTorr\text{-}ft^3/min.$$

The vessels have identical leak rates!

It is important to visualize that even though the 100 ft^3 vessel has ten times the evacuated volume of the 10 ft^3 vessel, as long as the vessels are at the same pressure and have identical leaks, virtually the same amount of gas will enter into each vessel. This is because the orifice of each leak "sees" approximately the same suction. Furthermore at very low pressures, flow velocity through any small leak path likely will be "choked," that is, limited by the speed of sound at the conditions in the leak path.

Industry Standards

The Parenteral Society Technical Monograph No.7, *Leak Testing of Freeze Dryers* notes that a frequently specified Leak Rate for new, clean, dry and empty freeze dryers is 2×10^{-2} mbar-liters/second (31.8 mTorr-ft3/min).[i] However, practices in industry for freeze dryers in use vary widely even to the point of accepting equivalent leak rates 50 times less stringent than the Parenteral Society Monograph suggests.

Influencing Factors

The above considerations also lead to the important observation that the starting pressure of either rates of rise or of leak rates must be specified. At high vacuum, there is a greater suction through a leak than at low vacuum. Therefore, one should expect lower leak rates and rates of rise at lesser vacuums (higher pressures) and higher leak rates and rates of rise at higher vacuums (lower pressures). In fact, one can obtain a rate of rise or leak rate of "0" with any chamber at atmospheric pressure!

A major concern for those measuring rates of rise or leak rates is the presence of what are called "virtual leaks." As the name implies, virtual leaks are not real or actual leaks caused by a breach in the vessel's walls or seals. Materials or pockets contained within the vessel can cause a greater rate of rise or leak rate than

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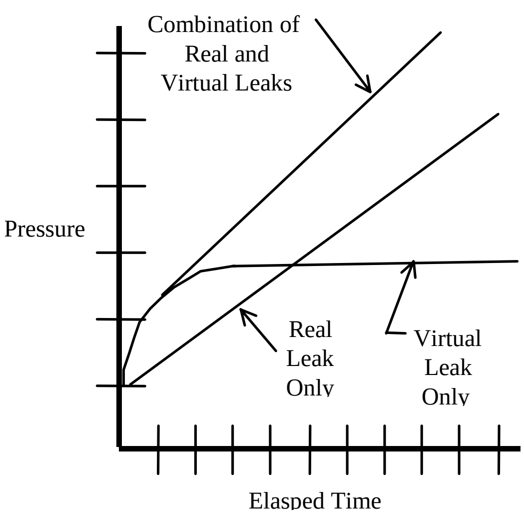
through outgassing would otherwise be obtained. Thus, one may be led to believe that there is a defect in the vessel's physical structure when in fact there is not.

One cause of virtual leaks is humidity and/or fluids within the vessel. If the vessel to be tested is not clean, dry and empty, pressure increases caused by the vaporizing of water and/or solvents contained within the vessel can occur. As the fluids vaporize, the pressure within the vessel increases at least in part due to the vaporization and not due to any real problem with the containment components. The out-gassing of volatiles from polymers and/or other substances can also have a similar effect.

A third type of virtual leak occurs when air (or other gas) is trapped in an annular space that has no opening to the outside of the vessel and a pinhole (i.e. very small) opening to the inside (e.g. a cavity within a weld). While the main vessel evacuates rather quickly, the gas trapped in the annular space evacuates much more slowly. Thus, while the vessel will appear to have been evacuated to the desired pressure, there will remain gas that is trapped in the annular space. When a leak rate or rate of rise measurement is attempted, a false reading will occur due to the gradual leakage of the gas from the annular space into the main vessel.

One indication of virtual leak is a decrease in the rate of rise. As shown on the graph below, when a virtual leak is present, the rate of rise will taper off as time progresses. In the case of outgassing, the rate at which vapor is emitted from the out-gassing substance slows and eventually stops as the pressure within the vessel becomes greater than vapor pressure of the substance. In the case where there is gas trapped in an annular space, the rate of rise will slow as the pressure of the gas equalizes with the pressure of the main vessel.



One possible remedy to these problems is to evacuate the vessel for an extended length of time. This will allow some vapors to be driven off outgassing substances and/or time for gases to evacuate from annular spaces. However, only trial and error experimentation can determine if virtual leaks are present.

Pressure increases due to virtual leaks can be reduced by ensuring that substances with vapor pressures lower than that of the operating conditions are used and/or by ensuring that the vessel is constructed without voids.

Vessels that contain refrigeration coils and/or other components which can become very cold can compensate for the above problems. If a surface within the evacuated vessel can be made cold enough such that out-gassing volatiles or gas from a void will condense onto the surface, then the effect of the virtual leak can be reduced if not completely abrogated. Most important, one must be consistent by always using refrigeration at the same temperature (because different temperatures will condense different amounts of gas) or by never using refrigeration. Furthermore, it is inaccurate to compare the leak rate of a vessel performed without refrigeration to the leak rate of similar vessel performed with refrigeration.

The Parenteral Society Monograph recommends that freeze-dryer condensers be kept at -40°C or colder to protect the vacuum pumps. Common practice in the United States is to keep the shelves at or below ambient (=<20°C) while allowing condensing plates to attain their minimum temperature (about -70°C for two-stage systems using refrigerant R507). Again, as long as conditions are maintained consistently from test to test, these conditions will yield validatable results.

Finally, specifying leak rates or rates of rise at pressures well below that of the system's normal operational parameters is unnecessary and potentially costly for several reasons. Components that satisfactorily contain vacuum at the operating condition can fail at the test condition. In addition, volatiles in substances that do not outgas at the operating condition may do so under the test conditions. As such, a large amount of time, money and effort can be wasted attempting to solve a “problem” which does not exist at actual operating conditions.

Conclusion

In summary, rates of rise cannot be used to compare performance of vessels of differing sizes; a leak rate must be specified. Second, when comparing leak rates (or rates of rise between vessels of equal volume), the starting pressures must be approximately equal. Third, whether using leak rates or rates of rise, consideration must be given as to how long the vessel is evacuated and the possible presence of virtual leaks. Last, refrigeration can be used, but must be used consistently to yield realistic comparisons and cannot be used to compare results against data obtained without the use of refrigeration. Of course, any leak rate done with refrigeration should have the cold trap temperature specified.

If you have further questions regarding leak rate testing, vacuum technology or freeze drying, please contact the SP Industries sales department at 1-800-523-2327.

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Common Units of Leak Rate

International:
millibar-liter per second (mbar-l/sec)

United States:
milliTorr-cubic foot per minute (mTorr-ft^3/min.)

Some Useful Conversion Factors

1 mbar-l/sec = 1592.97 mTorr-ft^3/min.

1 standard atmosphere = 760 torr
= 760 mm Hg
= 29.92 in Hg
=1010 mbar
= 14.7 psia

1 torr = 1 mm Hg
= 1000 millitorrs
= 1.33 millibars
= 133.3 Pascals (N/m^2)

1 cubic foot = 28.32 liters

[i] The Parenteral Society, *Technical Monograph No.7: Leak Testing of Freeze Dryers,* (Wilshire, England: The Parenteral Society, 1995), 9.