MILLROCK TECHNOLOGY®

Innovators in Freeze Drying

Freeze Drying / Lyophilization Tech Note

Vacuum Pressure Measurement Gauges

Freeze drying / lyophilization depends on vacuum to accommodate sublimation. The measurement and control of vacuum is therefore critical to the success of the process. There are several types of gauges that are used, their range and accuracy should be understood to perform a consistent freeze drying cycle.

Typical pressures in freeze drying are maintained well below 1000 mT and must be measurable down to 1 mT.

A ***pirani gauge*** is used for a high vacuum because it measures from atmospheric down to approximately 1 millitorr. The word approximately is used because pirani gauges tend to be accurate to 2% of the actual reading. When requiring better accuracy a ***capacitance manometer*** would be the choice. **Capacitance manometers** are typically accurate to 0.25% to 0.5% of the actual reading.

In the ***Pirani Gauge*** design, a sensor wire is heated electrically and the pressure of the gas is determined by measuring the current needed to keep the wire at a constant temp. The thermal conductivity of each gas is different so the gauge will measure the relative vacuum. Due to the fact that it consists of an exposed wire, a pirani gauge can't be used when dealing with solvents.

The ***Capacitance manometer*** operates by measuring the absolute pressure. The design consists of a moveable tensioned metal diaphragm with one side exposed to the gas and the other side containing an electrode assembly in a sealed high vacuum reference cavity. The gauge has the ability to detect the smallest changes in the pressure because of the smallest movements of the diaphragm. This construction also makes it immune to contamination so it can be used in working with corrosive products, products with solvents, and steam sterilizable applications.

A method to determine the 'end of the primary drying' is to monitor the **Pirani gauge** vs. the **Capacitance manometer**. When no unbound moisture is left in the product both gauges will read the same and therefore it can be considered the end of primary drying.

Vapor Pressure Table

Temp C	Press mT	Press mB
0	4579	6.1
- 4	3280	4.37
- 8	2326	3.1
- 12	1632	2.17
- 16	1132	1.51
- 20	930	1.24
- 24	526	.7
- 28	351	.467
- 32	231	.307
- 36	150	.199
- 40	96.6	.128
- 44	60.9	.081
- 48	37.8	.050
- 52	23.0	.030
- 56	13.8	.018
- 60	8.0	.0106
- 64	4.6	.0061
- 68	2.6	.0034
- 72	1.4	.0018
- 76	0.77	.00102
- 80	0.40	.00053
- 84	0.20	.00026
- 88	0.10	.00013

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