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LyoLearn Tech Brief

Determining The Completion Of Primary Sublimation During Freeze Drying

ABSTRACT:

Optimization of freeze-drying requires a method to determine that sublimation during the primary drying phase of freeze drying is complete so that the lyophilization process can be safely advanced to the secondary drying or desorption phase. This technical brief will discuss two preferred primary drying endpoint determination methods which are available on freeze dryers equipped with advanced process control systems.

The first method entails comparison of parallel pressure readings between a Pirani gauge and a capacitance manometer. When freeze drying, a capacitance manometer always gives a true pressure reading in the freeze drying product chamber. The Pirani gauge, a gauge commonly used in freeze dryers, will give a false high reading in the presence of water vapor. When the Pirani pressure reading decreases and approaches the true pressure reading of the capacitance manometer, little or no water vapor is present in the freeze dryer and it can be concluded that the primary drying stage of freeze drying is complete.

The second method is available with freeze dryer designs that have external condensers. An isolation valve can be added to the vapor port that connects the product chamber to the condenser. This valve can be closed for a short period of time and the subsequent rise in pressure in the freeze dryer's product chamber can be measured. When this pressure rise approaches zero, no more water vapor is being generated and the sublimation phase of freeze drying is complete.