101b Using Process Analytics to Monitor Drying of an Organic Monohydrate

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In this paper, we discuss the development of a drying protocol for an Active Pharmaceutical Ingredient (API) with a hydrate final form. The goal was to develop a drying protocol that reduced the plant drying times and had minimal risk of changing the hydrate form. Lab studies were done using in-line monitoring of both the headspace gases and of the drying cake. A contact near-infrared (NIR) probe was used to determine the wet cake composition and confirm form of product. Off-gas readings of the humidity and composition were also taken using a dew point meter and a mass spectrometer, respectively. All of these measurements were taken simultaneously in lab studies. The data from the humidity meter and mass spectrometer were correlated to the wet cake composition. These studies demonstrated a good correlation between the drying end-point, determined by NIR of drying cake, and off-gas conditions. Using this information it was possible to determine the drying endpoint while retaining correct hydrate form under a variety of drying conditions. Through the process understanding gained using off-gas monitoring in the lab, pilot plant batches ranging in scale from 10-70 kg were dried successfully.