pti science of quality

CASE STUDY

Helium Leak Detection vs. Dye Leak Testing

THE CHALLENGE

A global pharmaceutical entity approached Leak Detection Associates (LDA, now a PTI company) with early package failures from stability studies for a new drug product. The package was a cold-formed foil blister card that contained a wafer type product. During stability packaging, samples were leak tested using dye ingress methods and no leak failures were noted. However, after a very brief period under accelerated storage conditions, packaged samples were failing at an alarming rate. Since the product was highly sensitive to moisture, it was easy to determine failures by just shaking the blister. Once moisture entered the cavities, the wafer would dissolve into a small hardened "ball" that would rattle within the cavities.

THE SOLUTION

Stability blister card samples sent to LDA were leak tested by utilizing a SIMS helium leak detector. Since there was no helium sealed inside the cavities, each sample card had to have each cavity flushed with helium prior to performing the vacuum leak test. By using small gage syringe needles, helium was purged through the lidding and these puncture sites were sealed using an adhesive backed foil disk. This eliminated the possibility of any leak signal caused by the puncture sites. Helium leak rate data was collected from apparent well sealed blisters (no rattles) and failed blisters (product rattles).

THE RESULT

With the leak rate data from the blister samples analyzed, it was very clear to understand why many blister samples had failed in storage. Helium leak rates in the low/mid 10-4 mbar-L/sec, considered a gross helium leak, lead to the ingress of moisture to the cavities, transforming the product wafer into a hard ball. Even at these relatively high helium leak rates, dye ingress testing was not sensitive enough to detect seal quality issues. Some blister samples had leak rates significantly lower (10-7 and lower); these contained intact wafer product.

The data and results obtained from this case was one of the early proving grounds for using a helium mass spectrometer for pharmaceutical packaging applications, with LDA leading the industry promoting this novel and highly sensitive leak test method.