

Cold and Ultra Cold Vial Helium Leak Testing

THE CHALLENGE

A global pharmaceutical entity (Client B) approached Leak Detection Associates (LDA, now a PTI company) with vials for helium leak testing services. The vials were of the same size; however, one was of glass composition and the other was a thermoplastic material. Each contained the same neck finish to accommodate a press-fit integral crimp/stopper component. They were interested to see how various temperature storage conditions might affect their helium leak rates. The temperatures of interest were refrigerated (2 - 8°C), -40°C, -60°C, and -80°C.

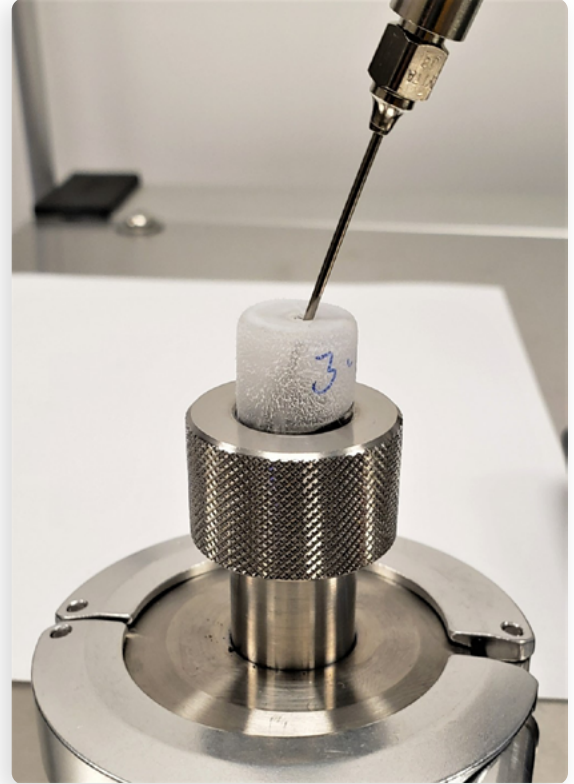


THE SOLUTION

Client B provided LDA one hundred (100) 2R size glass and plastic vials that were prepared by first drilling a hole (3.2mm dia.) in the base of each sample. After cleaning residual debris, the crimp/stoppers were applied to the finish using a small arbor press. The purpose of the hole was to supply 100% helium gas during the leak test cycle. This procedure is known as the 100% Helium Flow or Continuous Helium Flow method. This was a client stipulated method since they did not want to introduce potential leaks by puncturing the stoppers to allow helium into the sealed vials. Each vial set (25 samples) were allowed to condition at their test temperature for 24 hours prior to helium leak testing.



After the conditioning period, vial samples were individually analyzed utilizing a SIMS helium leak detector machine. Once removed from the cold environment, the sample was immediately placed on the vacuum test fixture and the leak test cycle was initiated. Approximately 20 seconds into the leak test, a tube was inserted into the exposed hole in the vial base and a flow of helium gas (25cc/min) continued for 10 seconds. The initial test cycle allowed for the leak rate to stabilize prior to introducing the helium gas. Any increase in the helium leak rate would indicate a leak pathway from the crimp/stopper area since this was the only section of the vial exposed to the helium leak detector. Once the full test cycle was completed, the next vial sample was removed from cold storage and the same test procedure was repeated until all 25 vials were completed. Subsequent leak tests of the other cold storage conditions were carried out by first allowing the 24 hour conditioning period of the vial sets. Helium leak rate data was collected from all four vial sets.



THE RESULT

After collecting and tabulating the helium leak rate data from each of the four temperature storage conditions, it was of interest to note that all of the vial samples did not exhibit any helium leakage – all were in the low/mid 10-9 mbar-L/sec. This leak rate level is well below the Kirsch helium leak rate criteria (10-6 mbar-L/sec) ensuring that these vials would most likely not encounter microbial or sterility problems during their intended shelf life. This study confirmed to the client that their press-fit vial crimp system was extremely robust, even well beyond the glass transition temperatures of most rubber stopper compounds.

