

Helium Leak Detection on **PLASTIC OPHTHALMIC CONTAINERS**

CHALLENGE

A major global pharmaceutical entity that manufactures and distributes a wide variety of Ophthalmic products requested the services of Packaging Technologies & Inspection/Leak Detection Associates (PTI/LDA). The specific request was to utilize our helium leak package testing methods to ascertain the seal quality of their container system. The primary package consisted of a three (3) piece container – the 10mL PE bottle, a press fit dispensing nozzle, and the threaded cap closure. Each component interface could be a source of leakage: the nozzle plug seal to the bottle neck ID/land seal and the closure seal to the nozzle open ended tip. The current leak testing method consisted of a vacuum bubble test, however the client was well aware that this test method would not be sufficient in order to mitigate the potential for product contamination via microbial ingress.

HELIUM LEAK TESTING EVALUATION

A series of leak testing projects began by first obtaining baseline helium leak rate values of their current container components. Individual leak test samples were identified by the bottle blow mold cavity number as well as the nozzle tip injection mold cavity number. This initial screening provided insight as to whether specific component parts/combinations would lead to high helium leak rates. The sample test preparation consisted of:

- Pressing the nozzle tip into the bottle neck.
- Applying the closure to a specified torque (4.0 – 4.5 lbf-in.)
- Creating a small hole in the bottle base (to allow helium gas to fill the bottle interior during the test cycle).



Sample Test Container

The helium leak testing procedure consisted of:

- Placing the Closure/Neck of the bottle package into a custom vacuum chamber fixture, inverted. This fixture would allow only the exterior area below the shoulder to be subjected to the vacuum pressure of the helium leak detector (HLD).
- Once a vacuum cycle reached fine test mode, the test sample was allowed to stabilize for approximately 15 seconds.
- With a stable background leak rate, helium gas flowed to the interior of the bottle through a syringe/flowmeter device. The flow remained constant for approximately 10 seconds.
- Final leak rate data was collected 30 seconds after reaching fine test mode on the HLD.



Bottle Vacuum test fixture with closure/neck under vacuum pressure

RESULTS

Initial helium leak rate data from the bottle samples noted two key issues:

1. High helium leak rates – sample groups showed upwards of 50% leak failures as defined by the modified Kirsch helium leak rate criteria ($1.0 \text{ E-}06 \text{ mbar-L/sec}$).
2. A specific nozzle tip cavity number exhibited high helium leak rates in almost all sample groups. Upon further part inspection, it was determined that the injection mold gate mark was creating a leak pathway.

Additional leak test studies were conducted after the following were instituted:

1. The closure application torque was increased to a range of 5.0 – 5.5 lbf-in. for all test sample preparations.
2. The one nozzle tip cavity (from above) underwent tool re-finishing to mitigate the leak pathway being created by the mold design.

The following two tables highlight:

- Table 1 – Representative helium leak rates collected prior to changing the closure application torque and performing the nozzle tip cavity re-finishing.
- Table 2 - Representative helium leak rates collected after changing the closure application torque and performing the tip cavity re-finishing.

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HARD VACUUM TEST MODE – Table 1

Bottle: 10mL, White Bottle, Tip; Gray Closure

Closure Application Torque: 4.0 – 4.5 lbf-in.

All Nozzle tips as provided (no re-finishing)

Sample Number	Bottle Cavity	Tip Cavity	Helium Leak Rate from Test Fixture ^{1,2}	Helium Leak Detected (Y/N)?
1	64	A14	1.8 E-05	Y
2	68	A12	7.4 E-05	Y
3	56	A18	1.9 E-05	Y
4	53	A25	1.7 E-08	N
5	72	A15	1.1 E-05	Y
6	54	A1	1.4 E-05	Y
7	51	A23	5.2 E-09	N
8	74	A19	4.1 E-09	N
9	58	A9	5.6 E-09	N
10	54	A17	5.6 E-06	Y
11	54	A11	1.0 E-05	Y
12	69	A21	5.3 E-09	N
13	72	A13	5.4 E-05	Y
14	61	A10	5.2 E-09	N
15	70	A6	1.0 E-08	N
16	70	A4	9.0 E-07	N
17	65	A3	4.8 E-09	N
18	60	A7	2.1 E-03	Y
19	67	A2	2.1 E-05	Y
20	52	A22	5.8 E-09	N
21	59	A16	8.8 E-06	Y
22	70	A8	5.3 E-05	Y
23	62	A5	4.4 E-05	Y
24	72	A24	4.7 E-05	Y

¹Note: All leak rate values are in mbar-L/sec.

²Reading taken post helium flow to bottle interior (at 30 sec. timeline)

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HARD VACUUM TEST MODE – Table 2

Bottle: 10mL, White Bottle, Tip; Gray Closure

Closure Application Torque: 5.0 – 5.5 lbf-in.

Specific Nozzle tip tooling issue was re-finished.

Sample Number	Bottle Cavity	Tip Cavity	Helium Leak Rate from Test Fixture ^{1,2}	Helium Leak Detected (Y/N)?
1	50	A1	1.9 E-09	N
2	73	A2	1.8 E-09	N
3	65	A3	1.4 E-09	N
4	52	A4	1.8 E-09	N
5	53	A5	2.1 E-09	N
6	58	A6	2.4 E-09	N
7	69	A7	1.9 E-09	N
8	51	A8	1.4 E-09	N
9	62	A9	2.2 E-09	N
10	68	A10	1.4 E-09	N
11	56	A11	1.9 E-09	N
12	66	A12	1.8 E-09	N
13	72	A13	1.5 E-09	N
14	63	A14	1.3 E-09	N
15	55	A15	2.4 E-09	N
16	71	A16	2.6 E-08	N
17	64	A17	1.8 E-09	N
18	57	A18	1.4 E-09	N
19	60	A19	2.2 E-09	N
20	59	A21	1.9 E-09	N
21	54	A22	1.4 E-09	N
22	74	A23	1.5 E-09	N
23	70	A24	2.1 E-09	N
24	61	A25	1.6 E-09	N

¹Note: All leak rate values are in mbar-L/sec.

²Reading taken post helium flow to bottle interior

PLASTIC CONTAINER HELIUM LEAK TESTING CONCLUSIONS

- Application torque of the closure to the threaded bottle played a huge part in the outcome of helium leakage. By just applying one additional lbf-in of torque, the bottles went from a leak failure rate of over 50% to 0%. After torque relaxation, significant leak pathways were created at the closure/tip interfaces.
- After careful inspection of molded parts, it was found that the gate mark area of one specific nozzle tip cavity presented enough deformation to create a helium leak pathway. Once that gate area was re-finished, helium leakage from the particular nozzle tip was eliminated.
- Due to the significant increase in sensitivity, the helium leak test method for this package application proved far superior to the bubble test (ASTM D4991) that had been the standard leak test protocol. The question of product sterility could now be assured by utilizing this enhanced test methodology.

ADDITIONAL CONCLUSIONS

- Helium is a highly sensitive leak test technology, capable of detecting extremely small leaks, which is not possible with other leak testing methods.
- Using a high vacuum technique, the leak test thresholds can be set to very low sensitivity levels allowing unique comparisons between package components as demonstrated in this case study.
- Helium leak detection is a highly sensitive solution for many applications ranging from package design, tooling qualification, production line setup and on-going product quality monitoring. It is also effective in accommodating a variety of package types including cold form blister cards, foil pouches, parenteral vials, syringes, pre-filled syringes and unique medical devices.
- Compared to conventional vacuum bubble and dye penetration test methods, packages can be quantitatively tested using helium as the tracer gas that ensures higher levels of accuracy. Such an approach allows a comparison between multiple packaging materials and forms, production line settings and stability storage conditions, supporting the entire product lifecycle.



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