

A Novel Approach to Ensuring a Leak Free Injection Port.

Michael A. Goss and Brad Rightnour

Restek Corporation, 110 Benner Circle,
Bellefonte, PA 16823



Abstract

In Split/Splitless injection ports, it can be difficult to make and maintain a good seal with a conventional metal inlet disk. The metal to metal seal dictates that you apply considerable torque to the reducing nut, but this does not always ensure a leak-tight seal. Over-tightening of the reducing nut can cause the critical seal on the bottom of the injector to become damaged or flattened. Over the course of oven temperature cycling, metal seals are prone to leaks, which ultimately can degrade the capillary column and cause other analytical difficulties.

Introduction

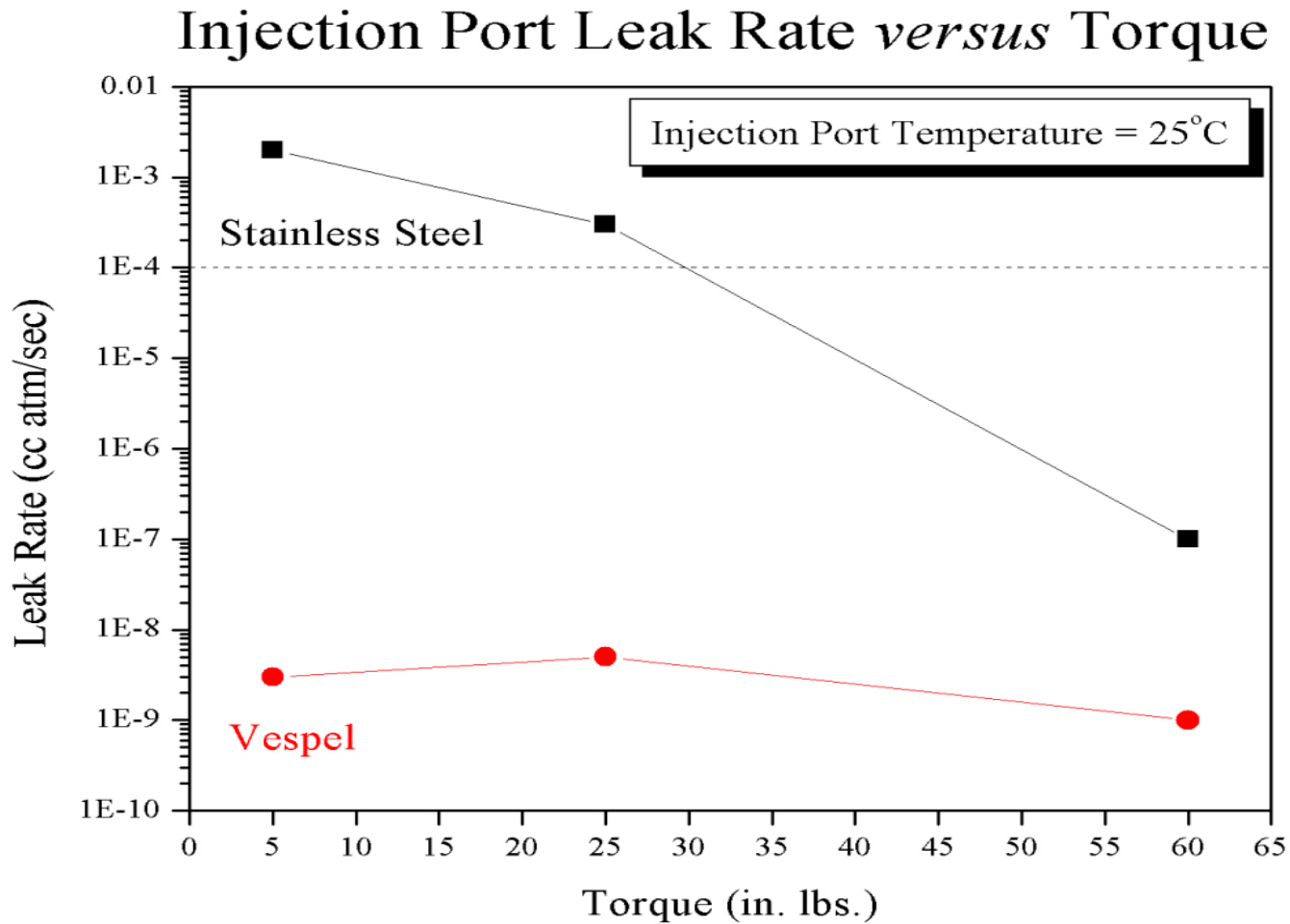
A new type of inlet seal has been developed that will greatly improve injection port performance by maintaining a reliable seal after repeated temperature cycles, without retightening the reducing nut.

This new inlet seal features two soft rings, one embedded in its top surface and the other embedded in its bottom surface. These rings eliminate the need for a washer, ensure very little torque is needed to make a leak-tight seal, and will not harm the critical seal at the bottom of the injector. The Vespel[®] rings are outside the sample flow path for worry free chromatography.

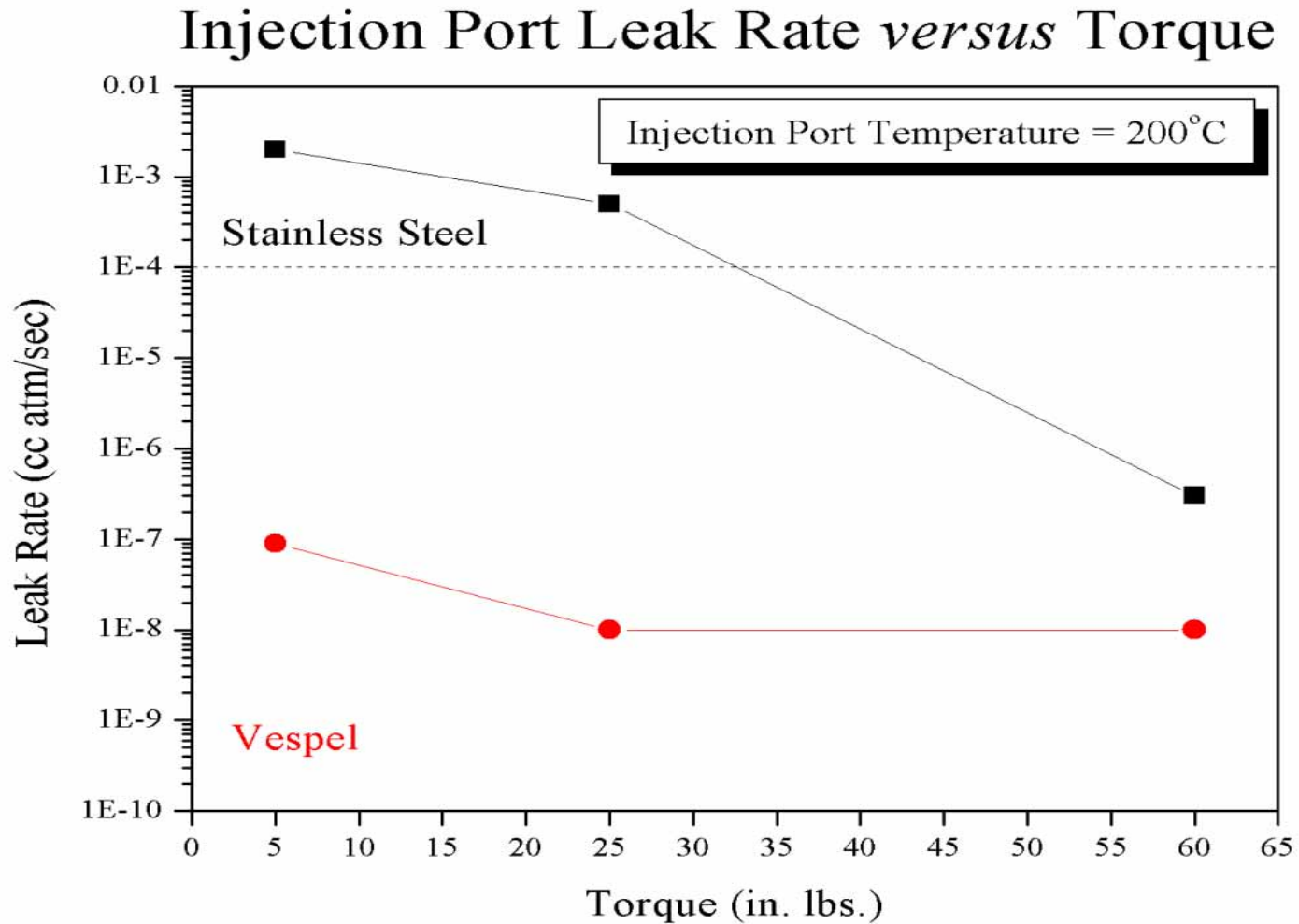
Experiment #1

The standard metal inlet seals are troublesome to get a leak-free seal even under ideal conditions. Several different inlet seals were tested using a high sensitivity leak detector with a Helium leak rate range of 1×10^{-1} to 2×10^{-10} (atm cc/sec.). The inlet seals were tested at various torques to determine the affect of tightening on the new inlet seal and a conventional standard metal inlet seal. Several seals of each type were tightened at 5, 25, and 60 pounds and the leak rates were measured at each setting. The following data shows the differences between the two types of inlet seals.

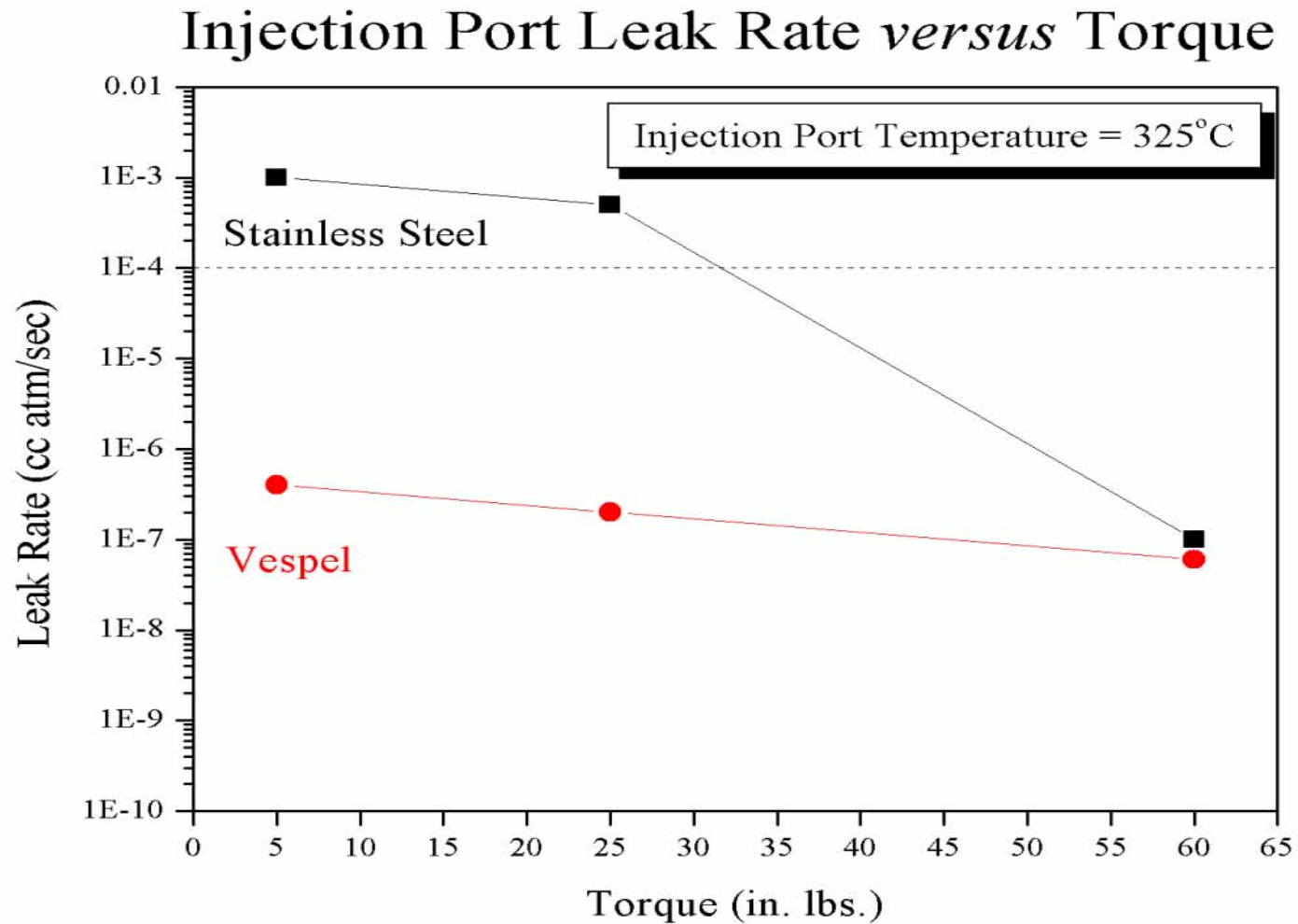
Dual Vespel[®] Ring Inlet Seal at Various Torques at 25° C.



Dual Vespel[®] Ring Inlet Seal at Various Torques at 200° C.



Dual Vespel[®] Ring Inlet Seal at Various Torques at 325° C.



Summary of Experiment #1

The Dual Vespel[®] Ring Inlet Seal has a much lower overall leak rate than standard all-metal inlet seals at torques of 5 to 60 pounds with varying injection port temperatures.

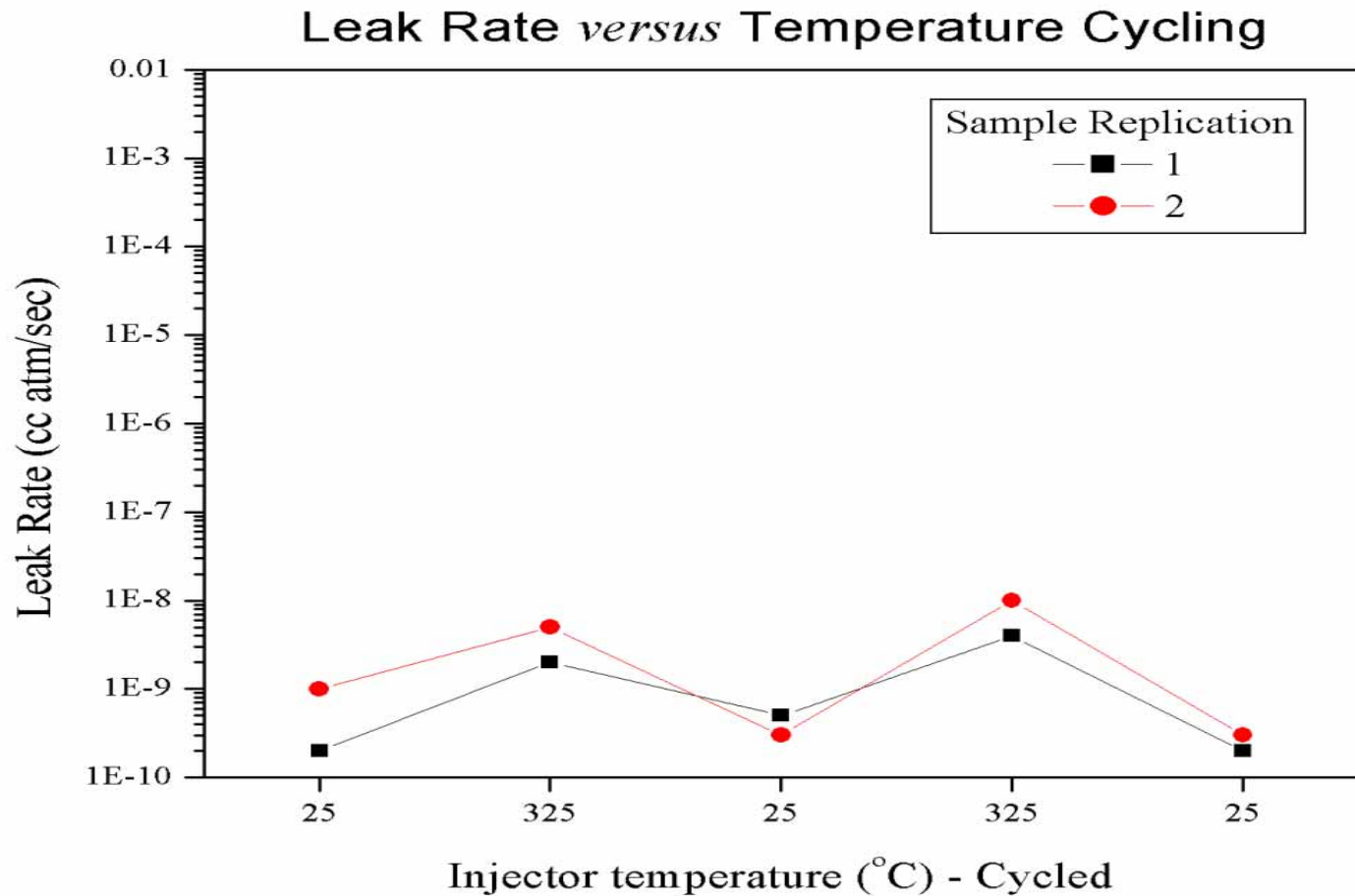
This lower leak rate results in extended column lifetime by reducing Oxygen permeation into the carrier gas and reduces detector noise, especially with sensitive detectors such as ECDs and MSDs. The soft seal will not harm the critical seal at the bottom of the injector, increasing the lifetime of the injection port.

Experiment #2

In order to ensure that the Vespel[®] material embedded in the surface of the inlet seal would not deform and cause leaks at various oven temperature cycles, the following tests were performed:

The reducing nut with the new inlet seal was tightened to a torque of 25 pounds. The injection port temperature was heated to 325° C., then cooled to 25° C. several times. The leak rate was then measured at temperatures of 25° C. and 325° C. The test was performed twice to verify the results. The following data was collected.

Dual Vespel[®] Ring Inlet Seal Leak Rate After Various Oven Temperature Cycles (Torque set at 25 pounds)



Summary of Experiment #2

The test was performed with two different inlet seals on different injection ports.

The test results show that the Duel Vespel[®] Ring Inlet Seal greatly improves injection port performance by retaining its sealing capability, even after repeated temperature cycles, at a very low torque (25 pounds), without re-tightening the reducing nut.

Dual Vespel® Ring Inlet Seal



Summary

The Duel Vespel[®] Ring Inlet Seal out performed the standard metal inlet seal in all of the testing.

The Duel Vespel[®] Ring Inlet Seal will not harm the critical seal at the bottom of the injection port, which will increase the lifetime of the injection port.

The Duel Vespel[®] Ring Inlet Seals will reduce operator variability by sealing at a wide range of torques, and will not be affected by temperature changes in the oven or the injection port. The Vespel[®] rings are outside the sample flow path for worry free chromatography.