A New Split/Splitless Injection Port Eliminates Sealing Problems and Offers Improved Inertness

Brad Rightnour, Mike Goss, and Jonathan Kaufmann

Restek Corporation, 110 Benner Circle, Bellefonte, PA 16823

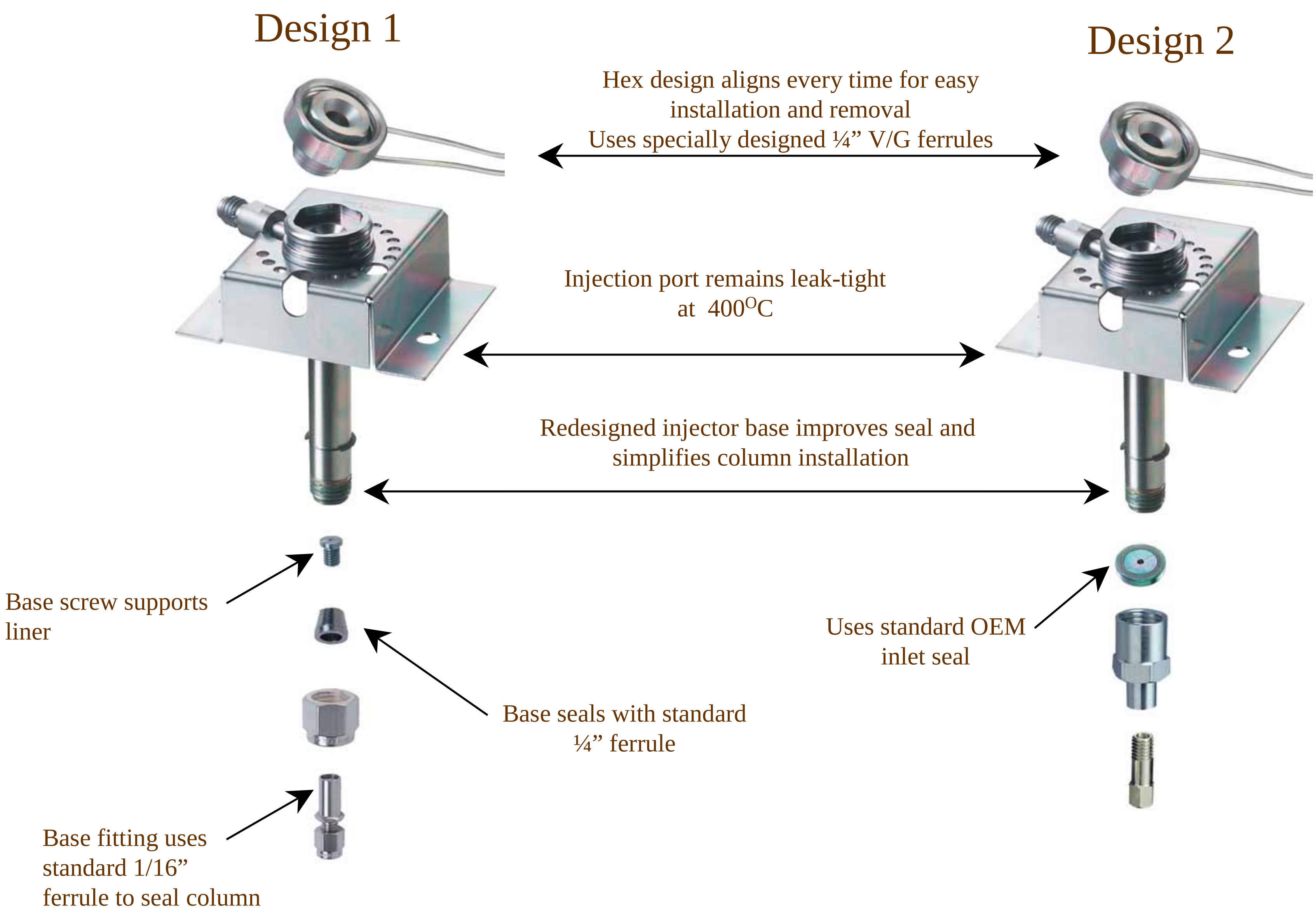
Abstract

Common problems associated with split/splitless injection ports include analyte breakdown or adsorption to the hot metal surfaces of the inlet body and difficulty in obtaining a reliable seal at the liner and base of the injection port. Further, split/splitless injection ports have been costly to maintain; traditionally, users have had to use special and often expensive parts to maintain these ports.

Introduction

A new split/splitless injection port eliminates the trouble areas associated with this type of injector, improving inertness and ensuring reliable sealing. An inert coating on the metal surfaces reduces sample breakdown when the surfaces are heated, ensuring more accurate data and allowing greater productivity. The new injection port eliminates critical sealing problems associated with metal-to-metal seals, and requires only inexpensive standard components for maintenance. In combination, these features make the new injection port easier, more reliable, and more economical to operate and maintain.

New Split/Splitless Injection Port



Experimental

Several criteria were selected to determine the inertness and reproducibility of the new injection port design and ensure that proper vaporization and flow characteristics were maintained. The first experiment was to investigate if injection port discrimination existed in the new injection port.

To demonstrate the absence of aliphatic mass discrimination the MA EPH method[1] reference mix was used to determine if the C_{28} to C_{20} response ratio meet the required >0.85.

The test was conducted on an Agilent 5890 Series II GC with an FID. The calculated results (Figure 1) show that the response ratio for C_{28} to C_{20} is 1.0. The response ratio meets the required standard of >0.85.

1. Method for the Determination of Extractable Petroleum Hydrocarbons: Commonwealth of Massachusetts, May 2004, Revision 1.1

Figure 1

C_{28}:C_{20} response ratio indicates no mass discrimination in the new port.

Injection: splitless

GC: Agilent 5890 Series II

Inj. temp.: 320°C

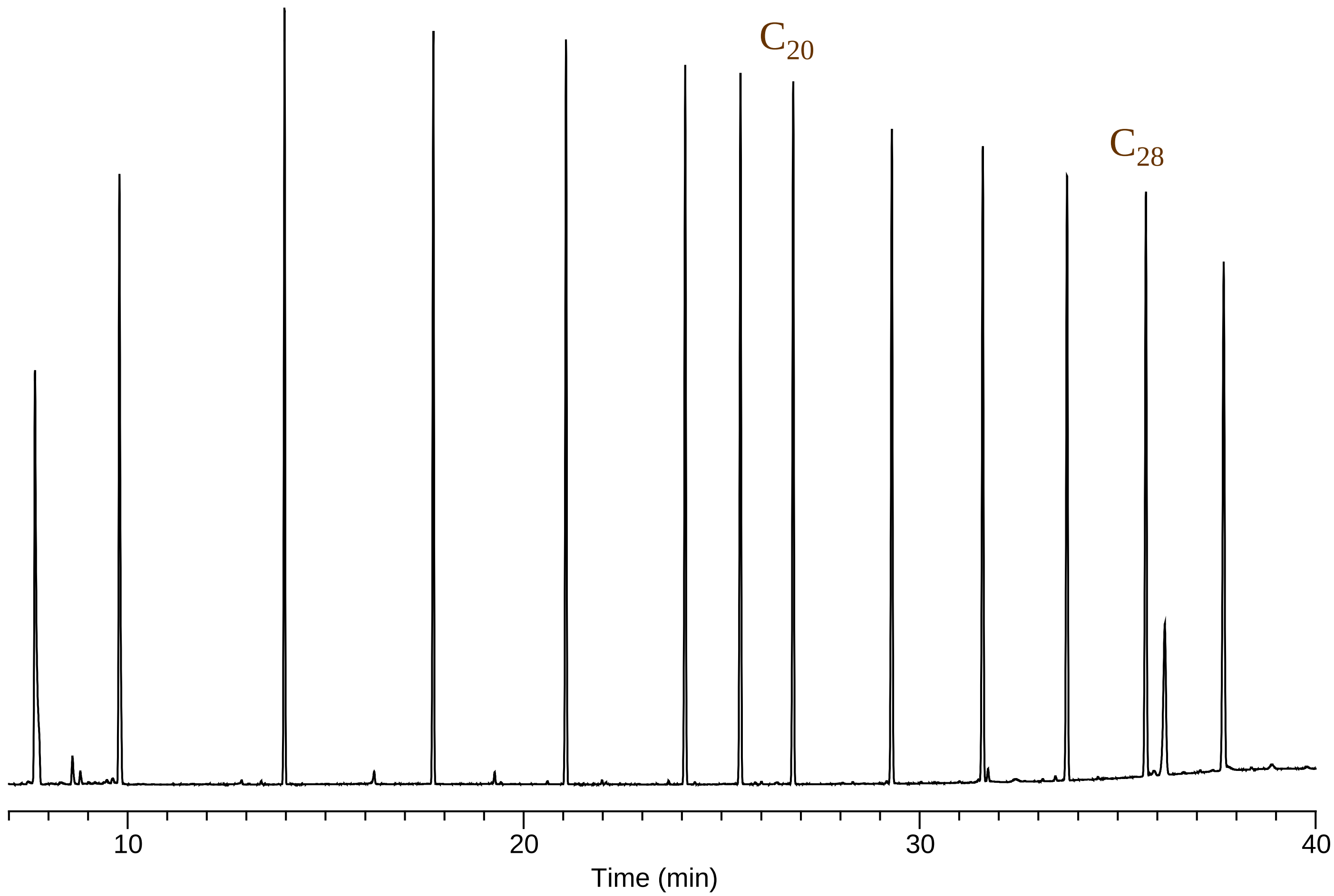
Carrier gas: helium, constant pressure

Linear velocity: 20 cm/sec.

Oven temp.: 40°C (hold 1.5 min) to 320°C @ 15°C/min (hold 5 min).

Det: FID @340°C

Sample: MA EPH reference mix (cat#31459)



XTI®-5 30m, 0.25mm ID, 0.25µm (cat# 12223)

The second experiment was to determine the reproducibility of peak area ratios between injection ports and whether active sites or dead volume existed inside the new injection port. A mixture of hydrocarbons and phenols was injected; the peak area ratio was calculated using 2,4-dinitrophenol and C14 (Table 1).

An XTI® injection mix with an on-column concentration of 7-17ng also was used. Responses were excellent for all probes, including the active compounds 2,4-dinitrophenol, 1,2-hexanediol, and benzoic acid (Figure 2).

Table 1

Equivalent peak area values show there is no adsorption and no dead volume in the new injection port.

Conventional Injector	**New Sure Fit™ Injector**
2,4-dinitrophenol/C14	2,4-dinitrophenol/C14
Peak Area ratio %	Peak Area ratio %
0.69	0.74
0.66	0.70
0.70	0.66
0.71	0.88
0.65	0.70
Mean = 0.68	**Mean = 0.74**

Figure 2

Excellent responses for active probes.

Injection: split

GC: Agilent 5890 Series II

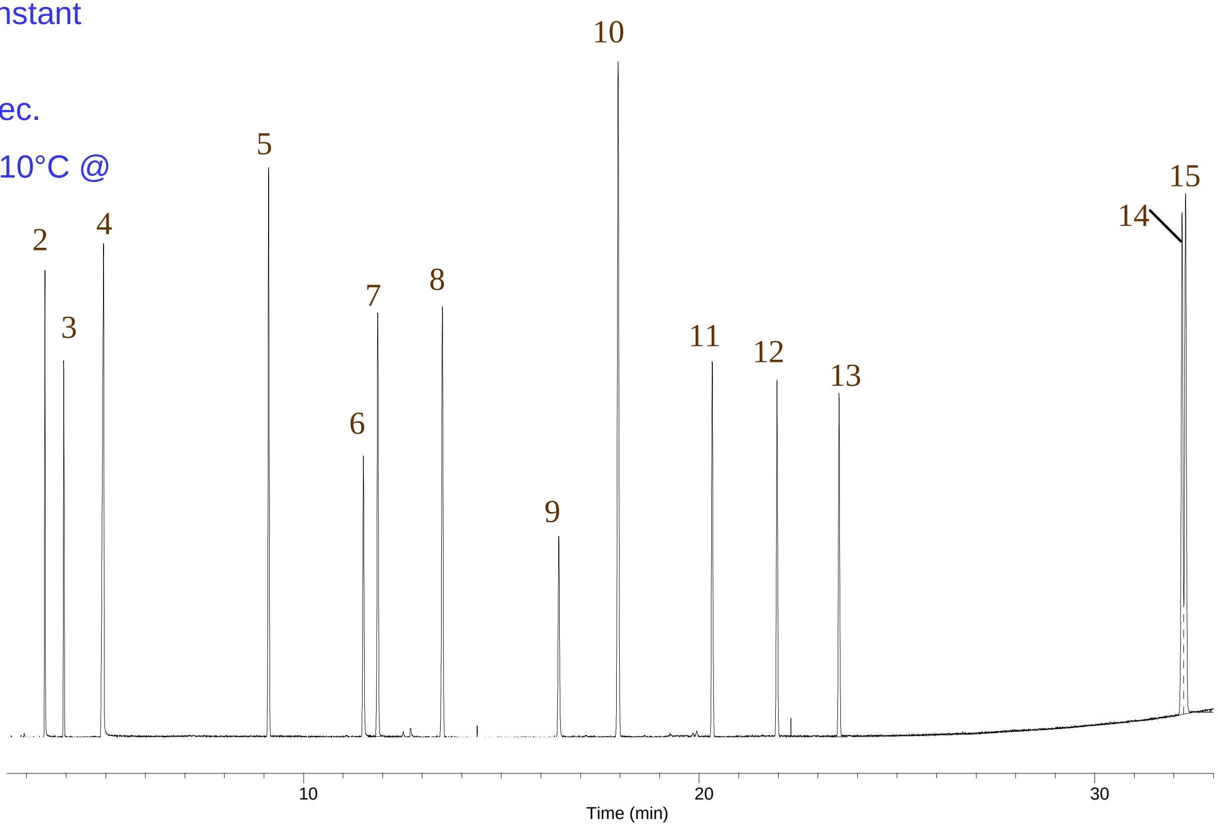
Inj. temp.: 325°C

Carrier gas: helium, constant pressure

Linear velocity: 20 cm/sec.

Oven temp.: 100°C to 310°C @ 6°C/min.

Det: FID @325°C



XTI®-5 30m, 0.25mm ID, .25µm (cat# 12223)

2. 1,2-hexanediol
3. Nitro-di-n-Propylamine
4. Benzoic Acid
5. C-14
6. 2,4-Dinitrophenol
7. Nitrophenol
8. Nitroaniline
9. Pentachlorophenol
10. Carbazole
11. C-20
12. C-21
13. C-22
14. Benzo b Fluoranthene
15. Benzo k Fluoranthene

Conclusion

The new Sure Fit™ injection port incorporates an innovative hex design which makes aligning the split/splitless weldment and shell weldment easy and makes installation and removal of liners trouble free. One Sure Fit™ shell weldment design allows the use of an inexpensive replacement to the standard OEM inlet seals, and can be easily cleaned or replaced when severely contaminated. An alternative Sure Fit™ shell weldment with a specially designed fitting uses a standard ¼” ferrule to seal the base of the weldment.

The new Sure Fit™ injection port uses a specially designed Vespel®/graphite dual taper ferrule to seal the liner inside the weldment. The new ferrule design eliminates the need for multiple graphite or fluorocarbon o-rings for different GC liners and conditions to minimize chance of leaks.

The Sure Fit™ injector is Siltek® treated to provide optimal inertness and resistance to temperature and ph extremes inside the GC system.