An Innovative Approach to Low Mass, Zero Dead Volume Connection of Fused Silica Columns

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Abstract

A common problem when joining fused silica columns together is obtaining a secure and leak tight seal. There are many different types of connectors available on the market today that allow the user to repair a broken column, or connect a transfer line or guard column to an analytical column.

Metal type connectors are often used and offer a secure connection, but it is often difficult to obtain a good, zero dead volume union. Press-Tight® connectors offer an inert connection, but these can disconnect when subjected high temperatures, pressures and turbulence in the GC oven.

There is a new type of connector that offers both a secure connection at high temperatures and pressures with zero dead volume. This new connection features a small union that will not disconnect after repeated heating and cooling cycles. This poster will illustrate the effectiveness and advantages of the new connector. We will show that the new connector gives the analyst a reliable seal, zero dead volume in the flow path, and has a lower thermal mass than standard metal type connectors.

Introduction

The Alumaseal™ capillary column connector is the next generation of fused silica column connectors that bridges the gap from the erratic dependability of glass Press-Tight® connectors, to a connector requiring no adhesives, simple to install, and will not be vulnerable to leaking when subjected to various temperature ranges.

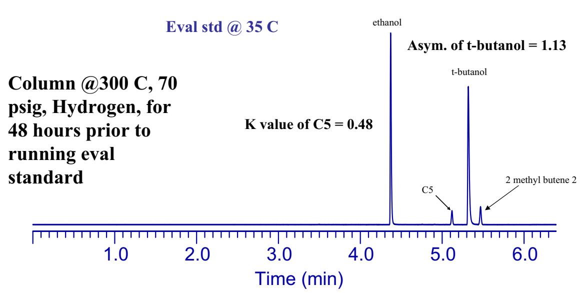
Experiments

Several experiments were performed on a Flame Ionization Detector (FID) to determine if the Alumaseal™ connector would withstand high and low temperatures without leaking. An experiment was also performed on an Electron Capture Detector (ECD) to determine the inertness of the connector.

Experiment #1

ASTM method D6730-01 (Determination of Individual Components in Spark Ignition Engine Fuels) requires that the asymmetry value of t-butanol falls within 1.0 to 5.0. and the K value of C5 must fall within .45 to .50. A 100 meter PONA column with a 2.5 meter tuning column, connected with the Alumaseal™ connector, was used for the analysis. If the connector would leak, the asymmetry value would be elevated and the K value would fall outside the optimum window. The column was held at 300 degrees C, at 70 psig of hydrogen, for 48 hours and brought back to 35 degrees C. The 30 component PONA evaluation mix was run to measure column/connector performance to the method. The asymmetry and K values were well within the specified ranges which indicated that no leaks were evident. *See* Figure 1.

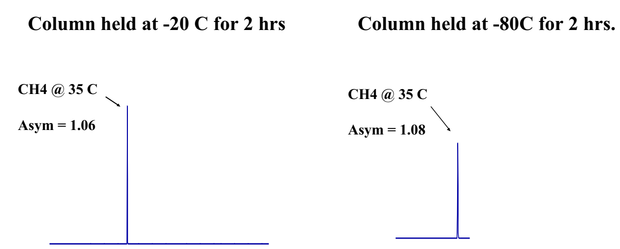
Figure 1
Front end slice of the PONA evaluation mix.
ASTM D6730-01



Experiment #1 *(cont'd)*

The PONA column and Alumaseal™ connector were then subjected to cryogenic temperatures to test the integrity of the connector. The column and Alumaseal™ connector were held at -20 degrees C. and -80 degrees C. for 2 hours respectively. The column was brought back to 35 degrees C and methane was injected to observe the asymmetry values to determine if leaks were present after being subjected to cryogenic temperatures. *See* Figure 2.

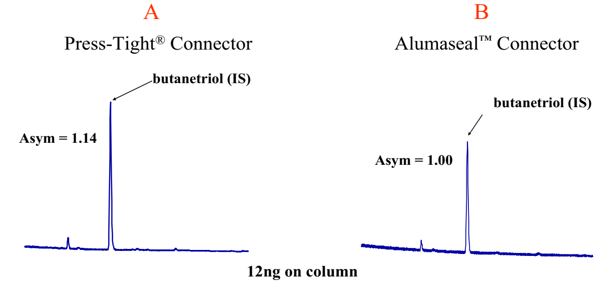
Figure 2
Alumaseal™ connector subjected to cryogenic temperatures.



Experiment #2

A 10 meter Rtx Biodiesel column (cat #10291) was connected to 2 meters of 0.53mm ID Hydroguard tubing using a standard Press-Tight® connector. A sample of B100 spiked with internal standards, as per ASTM method 6584-00e1 (Determination of Free and Total Glycerin in B-100 Biodiesel Methyl Esters), was injected on the column. The butanetriol (internal standard) typically will tail if any active sites or dead volume is present in the Press-Tight® connector. The peak asymmetry was calculated for butanetriol. *See* Figure 3A. The Press-Tight® was then removed and the Alumaseal™ connector was installed. The experiment was repeated and the peak asymmetry of butanetriol was again calculated. *See* Figure 3B. The data indicated that the Alumaseal™ connector did not contribute any negative activity to the internal standard butanetriol.

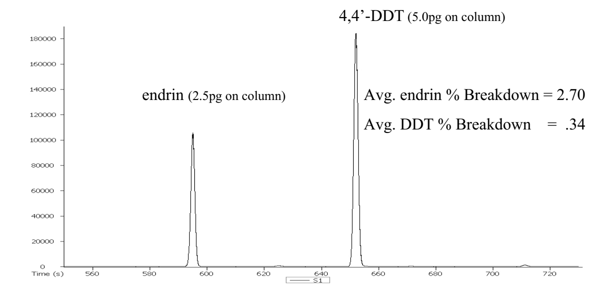
Figure 3
Peak asymmetry performance of Press-Tight® vs. Alumaseal™ connector on Rtx Biodiesel (cat #10291)
(10m x 0.32mm ID, 0.10 um column connected to 2m x 0.53mm ID Hydroguard tubing)



Experiment #3

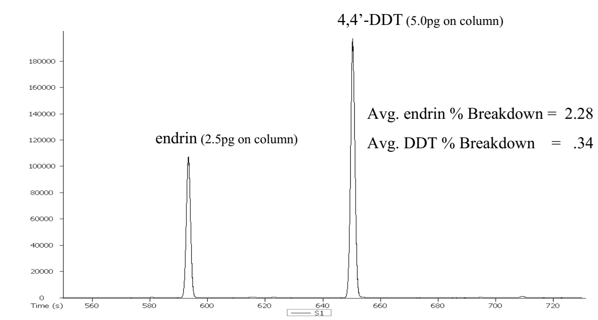
In this experiment an Rxi 5ms column was installed in an Agilent 6890 GC equipped with a micro Electron Capture Detector (ECD). Several injections of the Organochlorine Pesticide System Evaluation Mix (cat #32417) were run and the average breakdown of endrin and 4,4'-DDT was calculated. *See* Figure 4.

Figure 4
Breakdown before connector installation.



The Rxi 5ms column was then cut approximately 2.5 meters from the injector and the Alumaseal™ connector was installed. Several runs of the pesticide mix were run again under the same conditions and endrin and 4,4'-DDT breakdown was calculated. The Alumaseal™ connector did not contribute to any additional breakdown of endrin and 4,4'-DDT. *See* Figure 5.

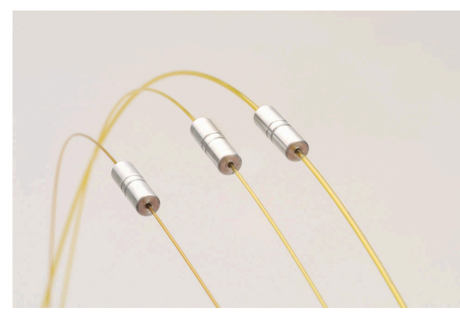
Figure 5
Breakdown after connector installation.



Conclusions

The experiments performed have shown that the Alumaseal™ connector is a viable alternative to glass Press tights® and bulky metal unions. The Alumaseal™ connector performed well even when subjected to high temperatures of 380°C and sub ambient temperatures of -80°C. The Alumaseal™ connector is the most favorable choice for connecting fused silica columns and offers a connection that is leak-tight, inert, and has no dead volume.

Alumaseal™ connector



General Applications