

Reduction of Mass Spectrometer  
Downtime During Column Change  
Using A Critical Orifice  
Connection Device



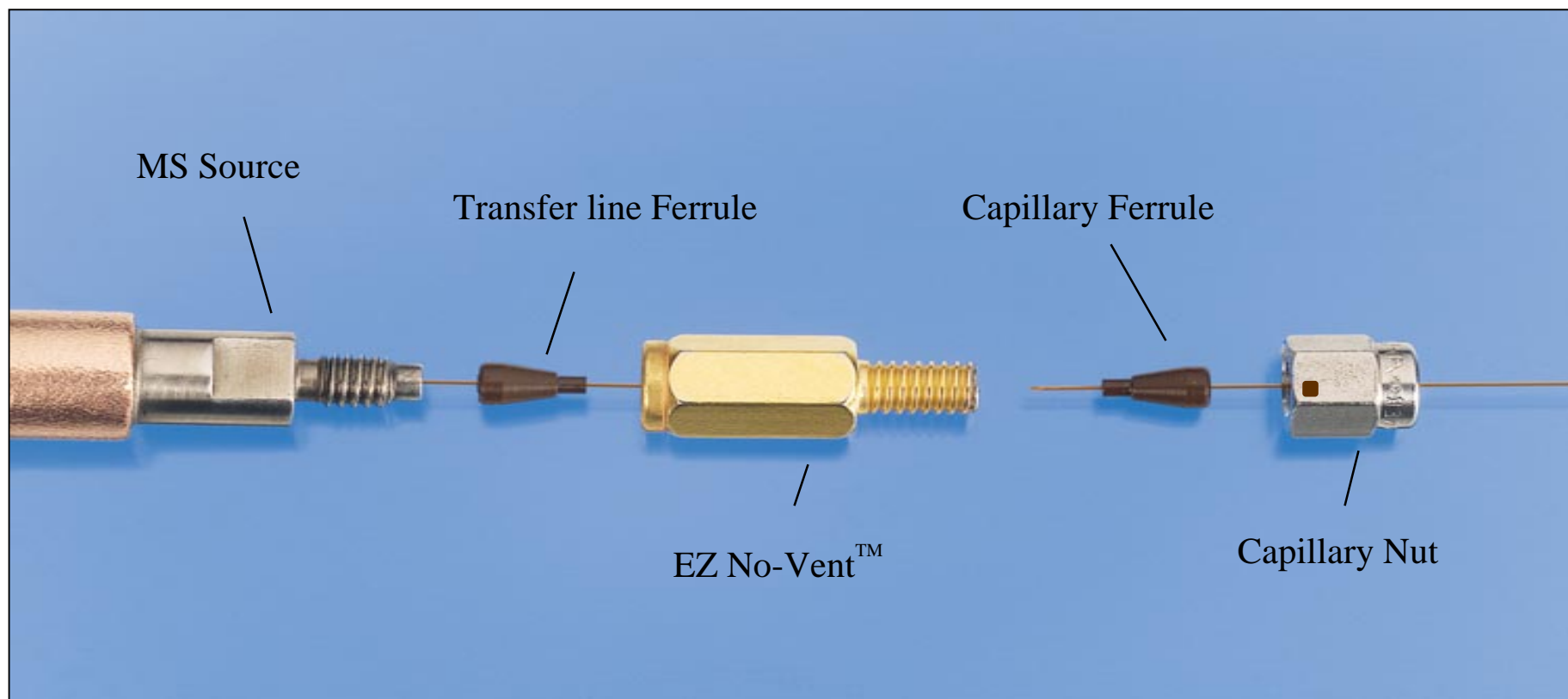
# Abstract

A common problem with the use of mass spectrometers is the amount of time needed to change a capillary column. Prior to removing the column it is necessary to vent the MS system. After installation of a new column the MS must be pumped down to a high level of vacuum prior to use. Overall this process can take as long as 5 hours, during which time the instrument is non-productive. A new mass spectrometer connector allows analyst to easily change the capillary column without the need to vent the instrument.

# Introduction

Changing a column in a GC/MS is a lengthy process and can take valuable time away from productivity. The new EZ No-Vent™ connector was designed to allow the analyst to change columns in the MS without venting. The EZ No-Vent™ connector utilizes a critical orifice to minimize the introduction of oxygen, and thus eliminates the need to use a purge gas. The EZ No-Vent™ connector easily attaches to the MS source with a standard ferrule, and without the need for special tools or plumbing. It also does not require secondary flow or pressure controllers, connection to the GC electronics, or additional gas lines.

# EZ No-Vent™ Connector



# Experiment #1

The EZ No-Vent™ connector was tested using highly volatile gases that are very susceptible to tailing from the presence of dead volume in the sample pathway. The chromatographic system initially was configured using a purge-and-trap concentrator connected to the capillary column by a split injection port. The column was directly inserted into the MS interface (Figure 1). The same configuration was then evaluated with the EZ No-Vent™ connector at the MS interface. Any dead volume in this fitting would result in significant tailing and broader peaks. As shown the peak shape was excellent using this new fitting (Figure 2).

# Figure 1:

## Before Using EZ No-Vent™ Connector

1. dichlorodifluoromethane
2. 1,2-dichlorotetrafluoroethene (Freon® 114)
3. chloromethane
4. vinyl chloride
5. bromomethane
6. chloroethane
7. trichlorofluoromethane

Inj.:Purge & Trap

GC: Agilent 6890

Inj. temp.: 300°C

Carrier gas: helium, constant flow

Flow rate: 1.0mL/min.

Oven temp.: 60°C isothermal

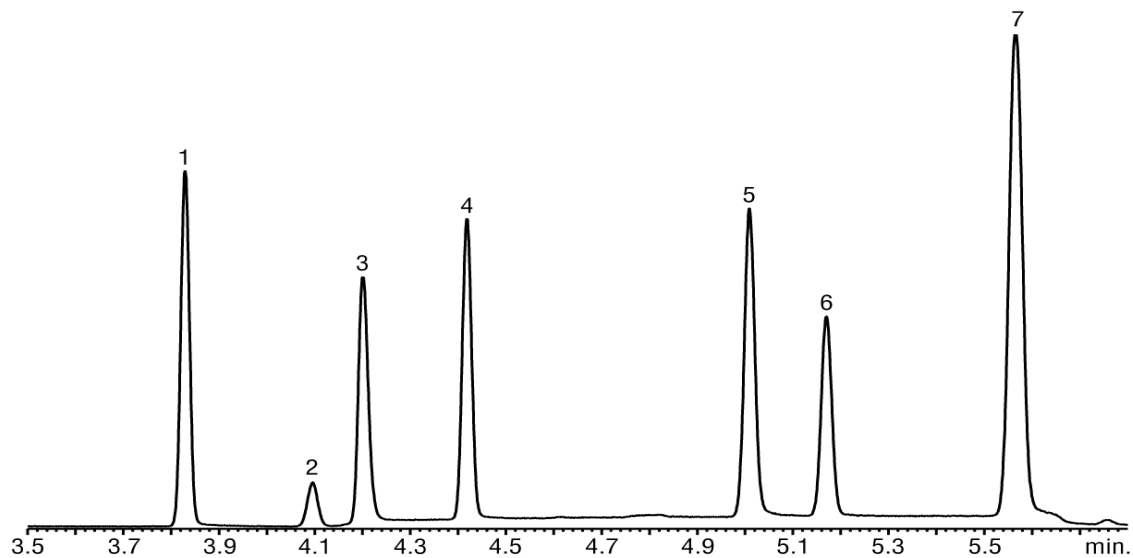
Det:Agilent 5973 GC/MS

Transfer line temp.: 280°C

Scan range: 35-550 amu

Tune: BFB

Ionization: EI



Rtx®-624 60m, 0.25mm ID, 1.4µm (cat# 10969), column connected directly to MS source.

Volatile Gas Mix 502.2 Calibration Mix#1 (gases) cat# 30042

# Figure 2:

## Column Connected to EZ No-Vent™ Connector

1. dichlorodifluoromethane
2. 1,2-dichlorotetrafluoroethene (Freon® 114)
3. chloromethane
4. vinyl chloride
5. bromomethane
6. chloroethane
7. trichlorofluoromethane

Inj.:Purge & Trap

GC: Agilent 6890

Inj. temp.: 300°C

Carrier gas: helium, constant flow

Flow rate: 1.0mL/min.

Oven temp.: 60°C isothermal

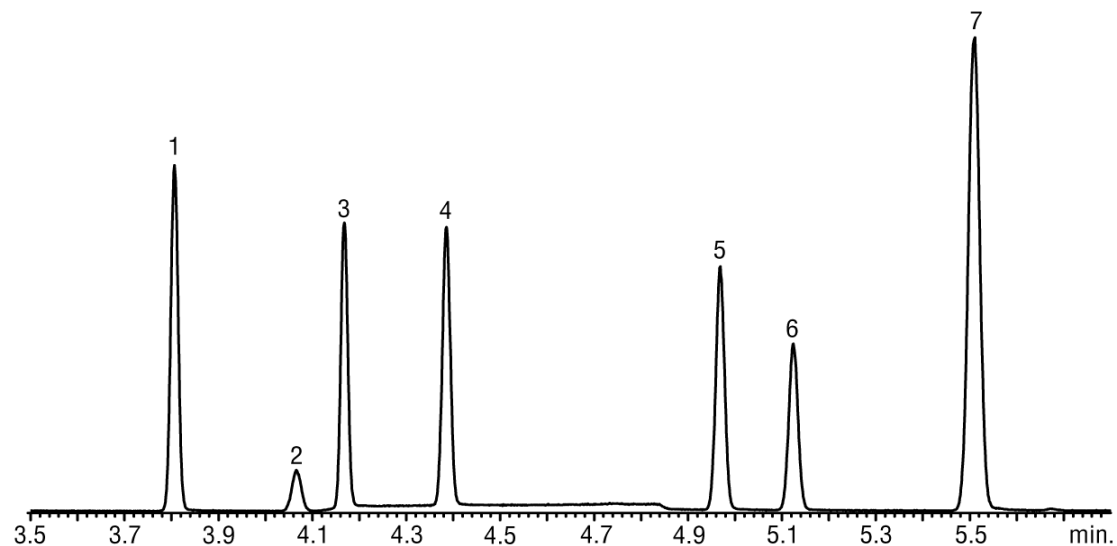
Det:Agilent 5973 GC/MS

Transfer line temp.: 280°C

Scan range: 35-550 amu

Tune: BFB

Ionization: EI



Rtx®-624 60m, 0.25mm ID, 1.4µm (cat# 10969) column connected to EZ No-Vent™ connector.

Volatile Gas Mix 502.2 Calibration Mix#1 (gases) cat# 30042

# Experiment #2

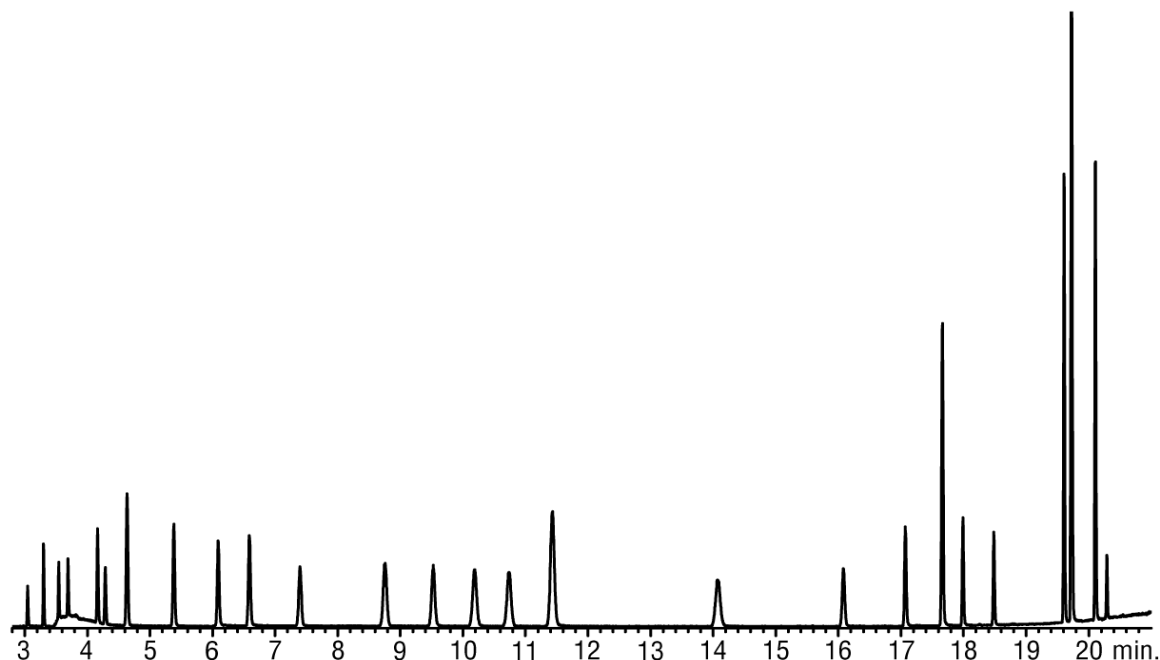
The EZ No-Vent™ connector allows columns to be changed without venting the MS, which can save a day of downtime. An application specific test was used to evaluate the ability of the MS to stabilize after a column change and without venting the MS. Again, purge-and-trap was used with halogenated volatiles. The first chromatogram (Figure 3) was run using an Rtx-624 phase and was acquired at 08:12 AM. A different column was installed and chromatogram acquired at 09:28 AM, with excellent peak shape and response (Figure 4) . Between runs the tune was verified and passed bromofluorobenzene (BFB) criteria. EZ No-Vent™ technology allows several column changes in a single day, without harm to the MS.



# Figure 3:

## 1<sup>st</sup> Column, Equilibrated System

Inj.:Purge & Trap  
GC: Agilent 6890  
Inj. temp.: 300°C  
Carrier gas: helium, constant flow  
Flow rate: 1.0mL/min.  
Oven temp.: 60°C (hold 15 min)  
to 220°C @ 30°C/min (hold 1 min)  
Det: Agilent 5973 GC/MS  
Transfer line temp.: 280°C  
Scan range: 35-550 amu  
Tune: BFB  
Ionization: EI



Rtx®-624 60m, 0.25mm ID, 1.4µm (cat# 10969),

EZ No-Vent™ connector, equilibrated system.

Halogenated Volatiles @ 200 ppb in 5mL/RO water.

502.2 Calibration Mix#1 (gases) cat# 30042

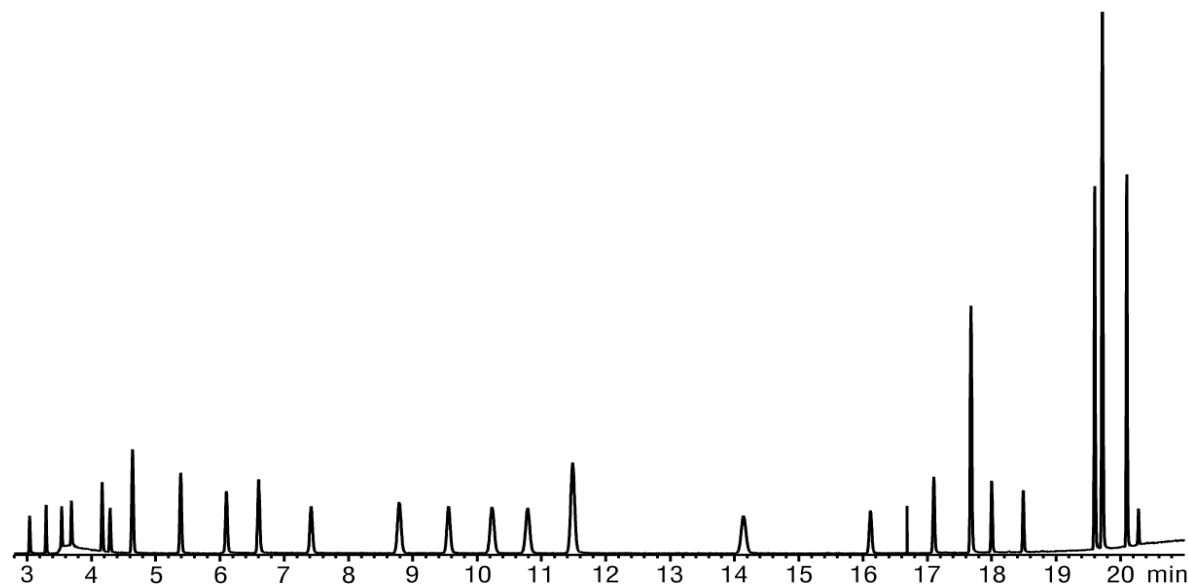
502.2 Calibration Mix#2 cat# 30043

BTEX standard cat# 30213

# Figure 4:

Acquired 76 minutes after Installing 2<sup>nd</sup> Column, without venting

Inj.:Purge & Trap  
GC: Agilent 6890  
Inj. temp.: 300°C  
Carrier gas: helium, constant flow  
Flow rate: 1.0mL/min.  
Oven temp.: 60°C (hold 15 min)  
to 220°C @ 30°C/min (hold 1 min)  
Det: Agilent 5973 GC/MS  
Transfer line temp.: 280°C  
Scan range: 35-550 amu  
Tune: BFB  
Ionization: EI



Rtx®-624 60m, 0.25mm ID, 1.4µm (cat# 10969) EZ No- Vent™ connector,  
acquired 76 min. after installing new column.

Halogenated Volatiles @ 200 ppb in 5mL/RO water.

502.2 Calibration Mix#1 (gases)                      cat# 30042

502.2 Calibration Mix#2                                cat# 30043

BTEX standard    cat# 30213

# Conclusion

The EZ No-Vent™ connector allows the analyst to easily change capillary columns in the MS without the time consuming need to vent the system. The EZ No-Vent™ connector allows the analyst to acquire a maximum number of runs, and make more efficient use of time, for higher productivity. The EZ No-Vent™ connector is easy to install and does not require additional plumbing or special tools. A critical orifice minimizes the introduction of oxygen into the MS during column changes, which eliminates the need for additional purge gases. The EZ No-Vent™ connector is gold-plated for maximum inertness and worry-free operation.