

APPLICATIONS

Effective Leak-Free and Zero Dead-Volume SecurityLINK™ UHPLC Connections at High Pressures

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Background

When using a UHPLC/HPLC, sub-2 μm particle or small ID column, additional dead-volume in a LC system can cause poor peak shape. The use of a zero dead-volume connection ensures no resulting bad chromatography due to the connection. When running at the higher backpressures of UHPLC columns, it is vital that the zero dead-volume connection can be installed quickly and trusted not to leak during the analysis.

Introduction

The minimization of the dead or dwell volume in a UHPLC system is an important consideration. Additional dead-volume in a system can lead to excess diffusion contributing to poor peak shape which in turn can cause failing results during method transfers or when assessing system suitability. This is especially true for both UHPLC and HPLC where chromatography problems can be seen when sensitive columns with lower internal diameters or core-shell/sub-2 μm particles are used.

While sub-2 μm particles and lower internal diameter columns can offer additional resolution and sensitivity to many LC methods, they do come with the side effect of increased backpressures. This increase can potentially cause connections/fittings to pop out or begin leaking, which can cause incorrect capture of data or even a loss of data due to leak sensors shutting off systems. Unfortunately, this can happen at any time, including after the sequence has already been set to run overnight. It is vital that an analyst can trust that once installed, their zero dead-volume connection will not leak.

This technical note investigates the effectiveness of the SecurityLINK fittings to maintain leak-free connections between UHPLC systems and columns.

Figure 1.
Zero Dead-Volume Connection

SecurityLINK UHPLC Fitting System

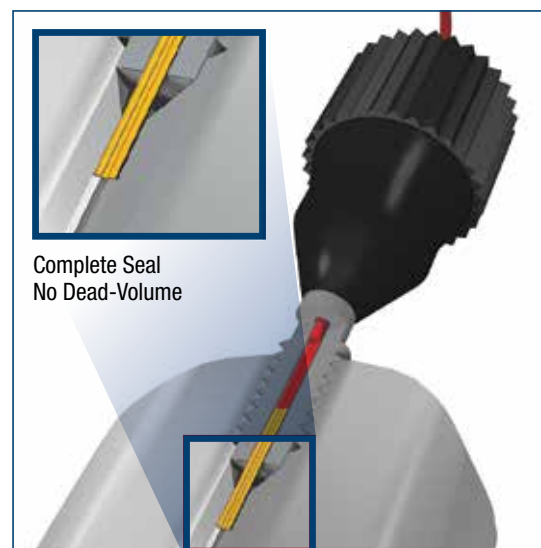
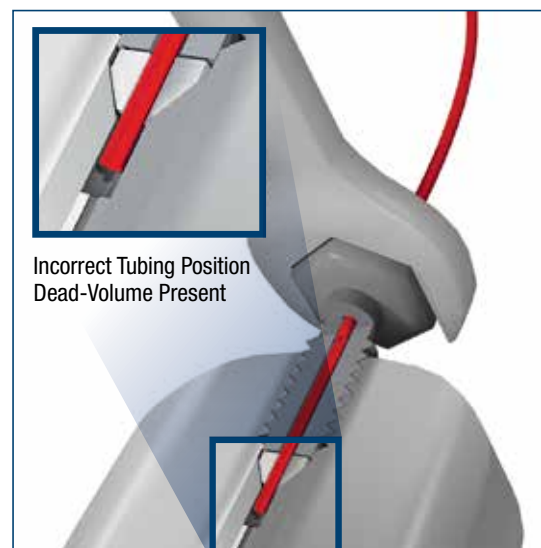


Figure 2.
Incorrect/Poorly Made Connection

Conventional Fittings (Nut and Ferrule)



Experiment Conditions

Three different PEEKsil™ SecurityLINK™ 100µm x 250mm connections (AJ1-2441) were installed for this experiment on a Waters® ACQUITY® i-Class UPLC® system. The tubing was connected directly from the autosampler port to a Kinetex® 1.7µm Biphenyl 100 x 2.1mm column. Each SecurityLINK was finger-tightened until one audible 'click' was heard for the connection to the autosampler port and the column. No additional torque or tools were employed for the installation of each SecurityLINK past finger-tightening. For the pressure and leak study, two flow rates, 0.32 and 0.45 mL/min, were run on the connections for 3 minutes each.

Table 1.

Pressure Readings from SecurityLINK PEEKsil Connections at Two Flow Rates

SecurityLINK Number	0.320 mL/min	0.450 mL/min
1	10,065 psi (694 bar)	13,981 psi (964 bar)
2	10,152 psi (700 bar)	14,068 psi (970 bar)
3	10,239 psi (706 bar)	14,170 psi (977 bar)
Average	10,152 psi	14,073 psi

UHPLC Conditions

Column: Kinetex 1.7 µm Biphenyl

Dimensions: 100 x 2.1 mm

Part No.: 00D-4628-AN

SecurityLINK Part No.: AJ1-2441

Mobile Phase: Water/Methanol (40:60)

Flow Rate: 0.320 or 0.450 mL/min

Temperature: Ambient

Table 2.

Pressure Readings and Leak Testing on SecurityLINK PEEKsil Connection on High Pressure Pump

Pressure	Leak detected
18,000 psi	NO
20,000 psi	NO
22,000 psi	NO
25,000 psi	NO

Conditions

SecurityLINK Part No.: AJ1-2421

Mobile Phase: Methanol 100 %

Results and Discussion

No leaking was observed at any point during the experiment as evident by continuous leak detection and pressure observation during the analysis. Pressures for each of the connections at both flow rates are reported in **Table 1**. Flow rates of 0.32 and 0.45 mL/min were chosen as they represent typical flow rates for a 2.1 mm ID column and neared the 15,000 psi pressure max of the system. An average pressure of 10,152 psi was observed at the flow rate of 0.32 mL/min, while the flow rate of 0.45 mL/min gave an average pressure of 14,073 psi across the three different PEEKsil SecurityLINKs used. SecurityLINK connections are rated for use at pressures up to 19,000 psi. The pressure data was collected during this experiment using a high pressure pump to demonstrate leak-free connection at 18,000 psi, 20,000 psi, 22,000 psi, and 25,000 psi as reported in **Table 2**.

Other zero dead-volume connections, which employ silent fingertight installation, require an analyst to be stationed at the LC to ensure there is no leaking after the column has reached its pressure for a given method. This can lead either under or overtightening. The fingertight SecurityLINK connection employs a responsive feedback that 'clicks' when the connection is complete ensuring the proper amount of torque has been applied to warrant a leak-free connection. This enables the connection to be made quickly and without the use of tools.

Conclusion

These results demonstrate the ability of the SecurityLINK connections to be run at high pressures immediately after initial installation, with no resulting leaking or pressure fluctuations.

Ordering Information



PEEKsil™

PEEKsil Double-Sided 10-32 Fittings with 1/16 in. OD tubing

Part No.	ID (µm)	Length (mm)
AJ1-2111	25	100
AJ1-2121	25	150
AJ1-2141	25	250
AJ1-2151	25	300
AJ1-2171	25	500
AJ1-2191	25	750
AJ1-21A1	25	1000
AJ1-2211	50	100
AJ1-2221	50	150
AJ1-2231	50	200
AJ1-2241	50	250
AJ1-2251	50	300
AJ1-2271	50	500
AJ1-2291	50	750
AJ1-22A1	50	1000
AJ1-2321	75	150
AJ1-2341	75	250
AJ1-2371	75	500
AJ1-23A1	75	1000
AJ1-2411	100	100
AJ1-2421	100	150
AJ1-2441	100	250
AJ1-2471	100	500
AJ1-24A1	100	1000



PEEK-Lined Stainless Steel

PEEK-Lined Stainless Steel Double-Sided 10-32 Fittings with 1/16 in. OD tubing

Part No.	ID (µm)	Length (mm)
AJ1-3121	25	150
AJ1-3141	25	250
AJ1-3161	25	350
AJ1-3171	25	500
AJ1-3181	25	600
AJ1-3221	50	150
AJ1-3241	50	250
AJ1-3261	50	350
AJ1-3271	50	500
AJ1-3281	50	600
AJ1-3321	75	150
AJ1-3341	75	250
AJ1-3361	75	350
AJ1-3371	75	500
AJ1-3381	75	600
AJ1-3421	100	150
AJ1-3441	100	250
AJ1-3461	100	350
AJ1-3471	100	500
AJ1-3481	100	600

Stainless Steel



Stainless Steel Double-Sided 10-32 Fittings with 1/16 in. OD tubing

Part No.	ID (µm)	Length (mm)
AJ1-1421	100	150
AJ1-1441	100	250
AJ1-1461	100	350
AJ1-1471	100	500
AJ1-1481	100	600
AJ1-1521	125	150
AJ1-1541	125	250
AJ1-1561	125	350
AJ1-1571	125	500
AJ1-1581	125	600
AJ1-1621	254	150
AJ1-1641	254	250
AJ1-1661	254	350
AJ1-1671	254	500
AJ1-1681	254	600



PEEKsil

PEEKsil Single-Sided Fittings; 1/32 in. OD PEEKsil Tubing with one 10-32 fitting for 1/16 in. ports, and one side with no fitting.

Part No.	ID (µm)	Length (mm)
AJ1-2224	50	150
AJ1-2274	50	500
AJ1-2294	50	750
AJ1-22A4	50	1000

SecurityLINK tubing material includes a sleeve that provides: ID, length and part number information.

Phenomenex Column / Tubing ID Recommendation Chart

	Nano	Microbore	Analytical				Semi-Prep
Column ID	0.05 - 0.1 mm (50 µm-100 µm)	0.3 - 0.5 mm (300 µm-500 µm)	1 mm	2.1 mm	3 mm	4.6 mm	7.8 mm
Tubing ID	25 µm	50 µm	50 µm - 75 µm	100 µm	100 µm	100 µm	125 µm

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Reorder Tip:

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