

Agilent creates a more perfect union for capillary column connections with the Ultimate Union.

By Lindy Miller, Agilent Technologies

Have you stopped using retention gaps or guard columns to save your capillary GC columns?

Are you tired of the arts and crafts required to get a permanent, leak-free seal when using fragile press-fit connectors?

Frustrated by leaks with metal unions once you start your temperature programmed sequences?

Though their benefits are well known, not many chromatographers use retention gaps or guard columns. Chromatographers walk away from the promise of longer column life and sharper peaks because it is difficult to make and maintain a reliable, leak-free seal.

A variety of column connectors or unions have been available, each with their shortcomings:

- **Metal fittings** have dead volume – void areas unswept by carrier gas. Sample components can migrate into this space, causing peak broadening.
- **Glass press-fit unions** are economical but make a very fragile seal that is prone to leak while in use. The sealing process is also difficult and time consuming, with a recommended half-hour curing time.
- **Combination unions** offer a mechanical device to hold the press-fit together in an attempt to make the seal less fragile, but they are only a compromise that does not shorten the time it takes to create a reliable press-fit seal.

Now you can get a fast, reliable and robust column connection with Agilent's new Ultimate Union.

Applying new technology to an age-old problem, Agilent has solved the chromatography challenge of column connections. Designed for use with Agilent's new patented Micro Fluidic diffusion-bonded plated technology, Agilent's Ultimate Union lets you make leak-free, inert column connections quickly and easily... again and again.

Take a look at Figures 1 and 2 and you'll see the important difference between the Ultimate Union and other metal fitting designs. The very small space beyond the column end in the Ultimate Union is easily swept by carrier gas to prevent peak tailing.

The Ultimate Union (Figure 2) is made of lightweight stainless steel, which when combined with SilTite metal ferrules, gives an extremely low dead volume fitting. Unlike other metal fitting designs, there is no need to retighten the ferrules after temperature cycles when using the Ultimate Union. The seal remains intact and leak free. We've also

solved the problem of the ferrule sticking in the fitting by adding a simple ferrule release hole on the side of the Ultimate Union. Just insert a paper clip into the ferrule release hole on the side and press firmly to release the ferrule.

Once you have made a column connection with the Ultimate Union, slip the connector into its clip which easily attaches to openings in the oven wall.

The Non-deactivated Ultimate Union (part number G3185-61581) is an economical choice for most applications. A deactivated version (part number G3185-61580) is also offered for samples with highly active compounds. Both unions are compatible with 100 µm up to 530 µm fused silica tubing. SilTite ferrules for use with the Ultimate Union based on the column diameters you are using are sold separately.

Learn more about the Ultimate Union. Visit www.agilent.com/chem/ultimateunion or contact your Agilent Representative.

SS Fitting Comparison

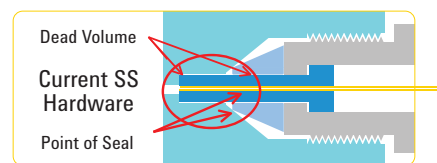


Figure 1

Exposure to polyimide and unpurged annular spaces are greatly reduced

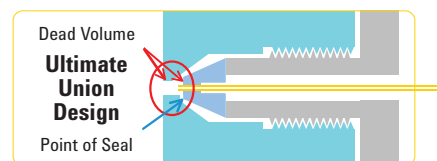


Figure 2



Ask the Experts

Looking for maintenance and troubleshooting advice?

Agilent's experts are backed by 40 years of practical experience and they want to share it with you. So check here first for the fast tips and tricks that can help you get the results you need.

When I change liners in the 6890 injection port, the liner often breaks, forcing me to clean the inlet. How can I prevent my inlet liners from breaking?

By Lindy Miller, Product Manager

It isn't clear from your question if the liners are breaking while being installed or removed, but either way, we have a solution.

I assume that you are using Agilent inlet liners manufactured to the proper tolerances and dimensions for the 6890 inlets, so this problem is generally caused by one of two things:

1. The liner is improperly installed, or
2. The Liner O-rings are sticking in the injection port

The best way to avoid the problem of breaking liners is to upgrade to the Flip-Top Inlet system. The 6890 users who have already upgraded find that it not only helps them to save time when changing liners, but also seals the weldment without the worry of cross-threading, overtightening and breaking the liner.



Agilent Flip Top Inlet Sealing System

Until you can upgrade your inlets to a Flip Top system, here are some tips to help you properly install a liner in a 6890 S/S inlet:

1. Place a new O-ring on the liner about 1 to 2 cm from the top of the liner. (IMPORTANT: Never try to re-use an old liner O-ring.) Carefully insert the liner with O-ring into the inlet (Figure 1).
2. Lay a column cutting wafer (p/n 5181-8836) across the top of the liner and gently push the liner down into the inlet until the wafer is flush with the top of the inlet. IMPORTANT: Tapered liners are pushed down until they hit the seal at the bottom of the inlet.
3. Screw on the weldment top being careful not to push the liner down further. Tighten the weldment to firm finger-tightness, and then use the special inlet wrench (p/n 19251-00100) to snug the weldment and form a leak-free seal.
4. Do NOT over tighten the fitting to avoid breaking the liner.

You can eliminate steps 3 and 4 – along with burnt finger tips – by installing a Flip-Top Inlet System.

If liners break when you remove them, we recommend that you try our new Non-Stick Inlet O-rings. Agilent now plasma treats liner O-rings using the same process as our Non-Stick Inlet Septa. This prevents the O-ring from sticking to the glass liner and the metal, so that it is easy to remove the liner without breaking it.

Avoid the problems caused by broken liners with Agilent's Flip-Top Inlet Sealing System and Non-Stick Inlet O-rings. **Learn more at www.agilent.com/chem/fliptop and www.agilent.com/chem/o-rings.**



Figure 1

Strategies for getting the best from your NPD

By John Wisniewski & Kevin Cooksy, Agilent Technologies



Any way you look at it, obtaining the best possible performance from a Nitrogen Phosphorus Detector (NPD) is a challenge, and bead life,

sensitivity and peak tailing are the main reasons why. By focusing on these troublesome areas, Agilent engineers produced a better bead and identified a better procedure for igniting it. They also improved the system to avoid bead breakage, and identified factors that may cause tailing of very polar compounds.

Increase the life of your bead.

6890 NPD ceramic beads are consumable parts that cannot be re-coated. To obtain the longest life from a bead, it is imperative that the gas system is clean and the bead is not used at overly high offsets. With a proper gas supply, the bead will ignite and achieve reasonable stability within a couple of days. When the bead is not being used, turn off the hydrogen reagent gas. It's the best way to prolong bead life.

Check for cleanliness.

Whenever you work with an analytical instrument, it is critical to confirm that the system is free of contamination from the outset. This is doubly true when you are using an NPD. Before operating an NPD, observe the offset at operating temperature with all gases on except hydrogen. A clean system normally registers between 0.3 and 0.9 pA. Anything above 1.0 pA will lead to decreases in response. Contaminated ceramic insulators on the collector are the most common cause of a high pA reading. In many cases, only the upper ceramic insulator and seal are contaminated because they are directly in the sample path. When changing the ceramic insulators and seals, the detector should be cooled down to 150°C or less to minimize the chance of stripping the three set screws. All gas supplies should have moisture and organic traps installed. We also recommend that all gases including the detector hydrogen, air and make-up gas have traps installed.

Get the gas flows and operating temperature right.

We advise setting flow rates as follows:

- Hydrogen: 3.0 to 3.3 ml/min.
- Air: 55-65 ml/min.
- Nitrogen make-up gas: 5.0 to 10.0 ml/min.

You may also use helium as a make-up gas, but only at flow rates under 5.0 ml/min. That's because the cooling effect on the bead creates the need to use higher bead voltages to obtain the desired target offset.

The NPD performs best at temperatures between 320°C and 335°C because the upper ceramic seal stays cleaner at higher temperatures. There is also an added benefit to working this way. You'll need lower ignition voltages than when the detector is operated at cooler temperatures.



Gas type

Recommended flow

Carrier gas

(helium, hydrogen, nitrogen)

Capillary, choose optimum flow based on column dimensions.

Detector gases

Hydrogen

3.0 mL/min (maximum flow is 5 mL/min)

Air

60 mL/min

Capillary makeup
(helium, nitrogen)

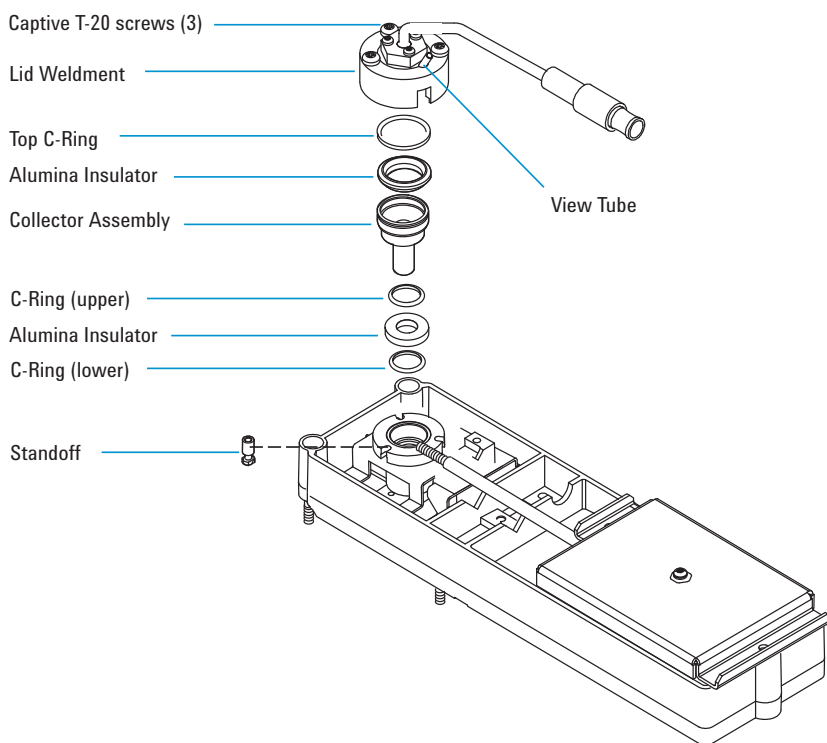
Nitrogen: 5 to 10 mL/min
Helium: less than 5 mL/min

Ensure peak symmetry.

Analysis of polar drugs exhibits asymmetric peak shapes – especially tailing – for several reasons. The capillary column is often a factor and should be eliminated as a potential cause before turning to the more complex NPD. Similarly, it is extremely important to ensure that the glass inlet liners are clean. Agilent's split liner (5183-4647) provides the best precision and also works well with EPC inlets.

For splitless injections, the deactivated single taper liner with glass wool (5062-3587) is generally an excellent choice. Install the liner with the inlet in split mode and a high split ratio (400-600:1) to purge any polysiloxane polymers that exist on a new liner. The inlet should always be cooled to below 100°C before changing liners to avoid damage to the polysiloxane deactivation coatings.

Follow these suggestions, and you're certain to see great improvements in the running of your Nitrogen Phosphorous Detector.



**Take the
GC ?
Challenge**

Answer 10 questions and prove to yourself – and your colleagues – that you have what it takes to keep your GC in tip top form. If you're among the best of the best, you could be eligible to win one of our great prizes. **Don't miss out!** Go to www.gcchallenge.com/chem/gcquiz7 today.