D3606 Application Column

Before starting:

1. It is critical that the columns are oriented correctly. Verify the correct orientation prior to installation using the small metal tag marked “inlet” located at the inlet end of each column. The inlet of Column 1 (backflush column) must be connected to the injector. The inlet of Column 2 (analytical column) must be connected to the switching valve.

2. To obtain optimal oxygenate peak symmetry, a deactivated glass liner must be used in the injector. Also, use a deactivated transfer line between the injector and Column 1. This is critical for accurate quantitation of benzene and toluene.

   Restek recommends:
   - A 2 mm ID deactivated splitless glass liner, such as cat.# 23313 for an Agilent split/splitless injection port.
   - A 0.76 mm ID deactivated transfer line, such as cat.# 29226. Short lengths should be used to minimize peak broadening.

3. If using an auxiliary column oven, verify that the temperature of your GC oven is 135 °C by using an accurate thermometer or digital thermocouple thermometer. Do not simply rely on the instrument temperature display.

Maximum column temperature: 150 °C
(Exceeding this temperature will cause irreversible damage.)

Installation (Figure 1):

1. Connect the inlet of Column 1 (backflush column) to the appropriate port of the switching valve using a transfer line and metal, Vespel/graphite, or graphite ferrules. Connect the exit end of Column 1 to the appropriate port of the switching valve using a transfer line and the manufacturer-recommended ferrule. Tighten according to the switching valve manufacturer’s specifications.

2. Connect the inlet of Column 2 (analytical column) to the appropriate port of the switching valve using a transfer line and the manufacturer-recommended ferrules. Tighten to manufacturer specifications.

3. Connect the exit end of Column 2 to the detector.

4. Turn on the carrier gas and slowly increase the head pressure until the flow rate of Column 2 (analytical column) is at least 20 mL/min. Typically, the head pressure required for helium is approximately 65 psi at 135 °C. Adjust the head pressure to method specifications for other gas types.

5. Purge at ambient temperature to remove any air that is trapped in the columns and/or GC system. While purging, use an electronic leak detector to check all column and valve fittings for leaks. In addition, check all external fittings downstream of the carrier gas purifiers up to and including the connection to the GC. Oxygen and/or moisture in the system will drastically shorten the lifetime of the column set.

6. After a 10-minute purge, elevate the oven temperature to 150 °C. Allow the column to condition for approximately 30 minutes.

7. After the brief conditioning period, lower the oven temperature to 135 °C and verify that the carrier gas flow rate is still 20 mL/min using a flowmeter. Head pressure will be approximately 65 psi when using helium.

8. The column set is now ready for use. Review the included Quality Assurance test chromatogram for the specific column set that you received. Using this chromatogram as a guideline, set the backflush time, inject a sample, and compare the results to the supplied Quality Assurance test chromatogram. If needed, optimize the backflush time by carefully following the instructions in ASTM D3606 for your particular instrument (approximately 2.4 minutes).

Simple tips to help your D3606 column set last longer:

1. Ultra-high purity carrier gas (helium or nitrogen) is essential. Not only should the highest quality gas be purchased from your supplier, but high-quality indicating gas traps should be located as close to your GC as possible. It is imperative that you prevent moisture and oxygen from entering the columns and our Super Clean gas filter baseplate traps (cat.# 22019) are ideal for this purpose.

2. Properly set the backflush time. It is important to prevent C9 and heavier components from entering Column #2.

3. Never exceed the maximum temperature of 150°C. Damage to this column set will be irreversible.