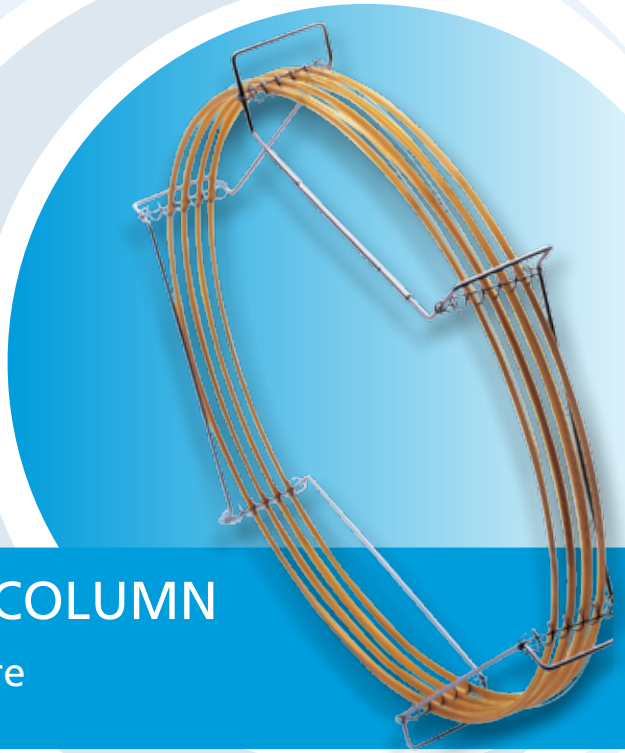
GAS CHROMATOGRAPHY



GC CAPILLARY COLUMN

Installation procedure

SGE Analytical Science

Congratulations on your purchase of an SGE GC Capillary Column.

SGE's GC Capillary Columns

In developing our range of GC capillary columns, SGE has focused on achieving advanced levels of robustness and reproducibility while continuing to deliver premium performance. The benefit for you is minimum downtime, maximum productivity and complete confidence that the column will continue to deliver accurate analytical results.

Why SGE?

SGE has a long history developing and producing GC Capillary Columns, with SGE's founder Ernest Dawes first being involved making glass capillary columns in 1959. SGE is the only independent manufacturer of GC capillary columns with a fused silica capillary tubing production facility. This gives us full control over every step of GC capillary column manufacture, from the fused silica production through to testing of the final product. As a result this column has been manufactured to exacting specifications at every stage.

This booklet provides a simple stepwise approach to installing your new column into the GC, and also presents you with some useful information, hints and tips to help you always get the best performance and lifetime from your column.

HELPFUL HINTS

Before Column Installation

Many chromatographic problems have their origin in leaking septa or a 'dirty' inlet liner. Before installing a new column, replacement of these two items is recommended as good practice. SGE has a complete range of liners and septa to suit all instruments.

To make your inlet liner choice easier visit www.sge.com/linerselectiontool.

1 Check gas filters, carrier gas supply

If your carrier gas supply employs oxygen and water filters, check the indicators on these filters to ensure they are not exhausted and replace if necessary, to protect your detector.

2 Place correct nut and ferrule on the column and cut column end

Select the correct ferrule for your column and application. Graphite ferrules are recommended for most applications. For the GC-MS interface, the SilTite™ metal ferrule from SGE will provide a leak-free connection that never needs re-tightening.

Visit www.sge.com for more information on ferrule selection.

SilTite™ Metal Ferrules

- Have the same thermal expansion coefficient as SilTite™ nuts thus eliminating leaks.
- No more re-tightening after installation.
- Specifically designed reusable SilTite™ nut for use with SilTite™ ferrules.

Once the nut and ferrule have been placed on the column, a 10 cm section of the column needs to be removed. Hold the column between your index finger and thumb and scribe across the surface of the column with the SGE cutting tool. This will leave a scratch in the polyimide surface. Apply a slight pressure to either side of the scribe mark. The column will snap and should leave a clean square end. If a clean break is not achieved, repeat the scribing process making sure that you are further into the column than the initial scribe.

3 Install column into the injector

As all GC injection systems vary, consult your GC installation manual for the correct distance a column should be inserted into the injector. Once inserted, finger tighten the nut. Using a wrench, tighten by about another 1/2 turn. You should not be able to pull the column out of the ferrule. If movement is felt, tighten until secure. If this cannot be achieved check that the correct ferrule has been used.

If the column is installed too far into the inlet, the distance for good mixing of the sample is reduced and some discrimination can be observed. It is also possible that if the column is inserted too far into the injector, the needle of the syringe will go beyond the end of the column at the time of injection. In this circumstance, very poor sensitivity or no peaks at all will be observed.

Using SilTite™ FingerTite connections enables column repositioning to be a thing of the past. Set your ferrule depth before installing the GC column. For more information visit www.sge.com/siltitefingertite

4 Turn on carrier gas and check flow

Turn on the carrier gas and adjust the column pressure to the desired value. If pressure for the column has not been pre-determined, adjust pressure temporarily to that recommended as listed in Table 1. Cut the column end according to the procedure described in Step 2. Check column flow by dipping the column end into a small vial containing a solvent (e.g. pentane). A stream of bubbles should be observed. If not, check for possible leaks in the injector or for any sign of damage to the column.

Table 1 – Approximate column head pressure (gauge) in psi (kPa) for optimum linear velocity*

Column	*Column Length (m)*							
(mm)	*10*	*12*	*15*	*25*	*30*	*50*	*60*	*120*
0.1	-	44 (300)	57 (390)	-	-	-	-	-
0.15	15 (105)	18 (130)	-	41 (280)	50 (340)	87 (600)	-	-
0.22	-	8 (60)	-	17 (120)	22 (150)	37 (260)	46 (320)	-
0.25	-	-	8 (55)	-	16 (110)	-	35 (240)	74 (510)
0.32	-	4 (27)	5 (32)	8 (55)	10 (70)	17 (115)	20 (140)	-
0.53	-	1.4 (10)	2 (12)	3 (20)	4 (24)	6 (40)	7 (50)	-

**Calculated for helium at 100 °C and with an atmospheric detector. Approximate pressures for both hydrogen and helium.*

HELPFUL HINTS

Using the wrong size syringe will compromise your results.

To maximize accuracy and reproducibility, we recommend that the minimum volume injected from a syringe is 20 % of full scale.

SGE manufactures a comprehensive range of manual, autosampler and instrument syringes. We have the syringe to suit your application, from microvolume 0.5 µL to jumbo 2 L sizes.

HELPFUL HINTS

SGE Improves your GC Connectivity. *SGE understands the importance of connections in your GC. Our new connections range of ferrules, µ-unions and SilFlow™ splitters all provide inertness, low thermal mass and can be finger tightened. For more information visit www.sge.com/silflow*

5 Install column into detector

Place nut and ferrule on the column. Cut the column end again to ensure no ferrule material is deposited in the column end. As all instruments require the column end to be located at different positions in the detector, consult your GC installation manual. Determine if the signal is relatively stable and is not subject to sharp movements. This would indicate a problem with the column position or foreign material in the detector. The baseline should stabilize in a uniform manner. If not, remove column, inspect column end and, if necessary, the detector assembly. Consult your GC manual for this step.

6 Check for leaks

Once the column is installed and a preliminary gas flow applied, check for leaks. For non GC-MS applications (e.g. FID, ECD, NPD being used) you only need to check the injector system. Use an electronic leak detector if possible. To check for leaks at a GC-MS interface, use a stream of argon. If a leak is present, an argon signal will be detected on the MS system.

7 Set carrier gas flow

To set the carrier gas flow at the optimum velocity, a non-retained compound should be injected. Set the column temperature to 50 °C and inject the appropriate compound (Table 2) to determine the column gas velocity. For columns with a film thickness > 3.0 µm, an oven temperature of 100 °C is recommended.

Table 2 – Non-retained compound to use for capillary column carrier gas velocity determination

Detector	Compound
FID	Methane, Butane
MS, TCD	Argon, Methane, Butane, Air
ECD	Methylene Chloride[1]
NPD	Acetonitrile[2]
PID	Ethylene, Acetylene

1. Only use the headspace of methylene chloride, do not inject neat solvent.
2. A column temperature of 100 - 130 °C is required as below this temperature range acetonitrile can be retained by the column.

Table 3 lists the time it will take a non-retained compound to elute for a column set at the optimum velocity for either hydrogen or helium carrier gas.

Table 3 – Time (seconds) needed for a non-retained compound to elute at optimum gas velocity

Column Length (m)	**Helium** (25 cm/sec)	**Hydrogen** (40 cm/sec)
12	50	30
15	60	37
25	100	60
30	120	73
50	200	120
60	240	146

8 Column conditioning

SGE columns have been pre-conditioned to guarantee that a stable baseline can be achieved quickly. However for optimum performance SGE recommends that a column always be conditioned prior to first use on your instrument.

As a precaution if using a Mass Spectrometer, or other highly sensitive detector, the column should be removed from the detector during conditioning. Never exceed the maximum cycling temperature specified for the column for more than 10 to 15 minutes. The maximum continuous operating temperature of the column can be found on the test report supplied with the column. It is also the lower of the two temperatures stated on our website or literature. For example in the case of a column with a temperature range of –40 ˚C to 360/370 ˚C, 360 ˚C is the maximum continuous operating temperature, –40 ˚C is the minimum temperature, and 370 ˚C is the maximum cycling temperature.

Install the column into the Gas Chromatograph injector (following the manufacturer's instructions), then set the carrier gas flow (see table on page 6). Ensure that there is gas flowing from the end of the column before installing into the detector. To condition the column, program the Gas Chromatograph to ramp at 10 ˚C/min to the maximum continuous operating temperature of the column. Once the heating ramp has started monitor the detector signal, it should initially increase and then gradually decrease until a flat baseline is observed. Continue to condition the column for 60 minutes at the maximum continuous operating temperature, if the baseline is not flat (still dropping) a further 30 to 60 minutes may be required.

HELPFUL HINTS

Do not use a Septum above its maximum temperature, as baking and decompostition will result. Replace regulary to avoid leaks which will lead to poor chromatography and reproducibility.

HT Septa

- *BTO grade silicone*
- *Maximum injector temperature 400 °C*

Table 4 – Table of recommended gas flows for column conditioning

Column ID	Flow Helium (at max temperature)
< 0.2 mm	1.0 mL/min
0.2 - 0.32 mm	2.0 mL/min
> 0.32 mm	5.0 mL/min

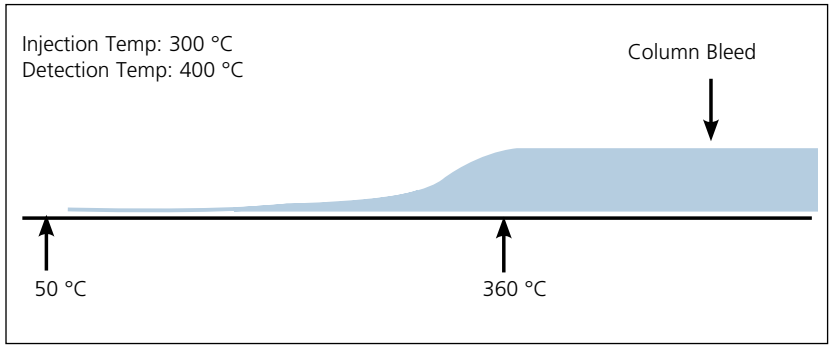
A common bleed profile can be seen in Figure 1. In general, thick film (> 1.0 µm) and polar phases do require additional conditioning time to produce a stable baseline. If a stable, flat baseline is not achieved even after additional conditioning, the system may have an air leak or another form of contamination. Cool the column down to room temperature and re-check the fittings for leaks or possible sources of contamination. If the problem is not resolved, contact your local SGE office for assistance.

9 Column storage

When a column is not in use, SGE recommends that column ends are sealed. Sealing column ends eliminates the possibility of contaminants and any other foreign material entering the column and causing long term damage.

Please note the injector and detector ends of your column so that on reinstallation the column is properly oriented.

Figure 1 – Column Bleed Profile



10 Regeneration of Columns

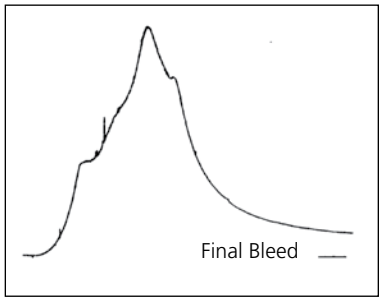
Deterioration of column performance with use is very much application dependent. However, it is important to note that SGE columns have been specifically designed to resist permanent damage, and usually a simple 1 or 2 step recovery process is all that is needed to restore the column to its original performance. Typical symptoms of deterioration in column performance include unstable signal, large baseline rises on temperature programming, or peak tailing especially for active components. If you observe these symptoms, and have verified the column to be the cause, follow the process below.

A. ***Trimming the column.*** The first few centimeters of a capillary column are the most prone to damage from problems such as deposition of non-volatile material from the sample, the effects of large amounts of solvent being deposited on the column, or even just mechanical damage from the syringe needle being inserted into the column during on-column injection. If poor peak shape is observed either in the form of tailing or simply broad peaks, and the problem is thought to be related to the capillary column, the simplest solution is often to remove approximately 50 cm from the front of the column. Follow the guidelines given earlier (Step 2) when cutting the column and fitting the ferrule. Remember: pieces of fused silica, polyimide, metal coating or ferrule material in the column will also cause tailing and broad peaks.

B. ***Conditioning.*** If trimming the column does not resolve the problem, the column can usually be returned to its original condition by conditioning. Generally the column should be programmed at about 10 ˚C/min to its maximum continuous operating temperature. The maximum continuous operating temperature can be found on the column test report supplied with the column. It is also the lower of the two temperatures stated on our website or literature. For example in the case of a column with a temperature range of –40 ˚C to 360/370 ˚C, 360 ˚C is the maximum continuous operating temperature, –40 ˚C is the minimum temperature, and 370 ˚C is the maximum cycling temperature. The detector noise should increase to a maximum and then gradually decrease until a flat baseline is observed (Figure 2). The length of time at maximum temperature required to achieve this will depend on the degree of contamination, and may vary anywhere from 1 hour to 24 hours in an extreme example. Never exceed the maximum cycling temperature specified for the column for more than 10 to 15 minutes. As a precautionary measure the column should be disconnected from the detector during conditioning as some samples may deposit materials on the detector.

Figure 2 – Column Conditioning

Column is programmed to its maximum operating temperature and held at that temperature

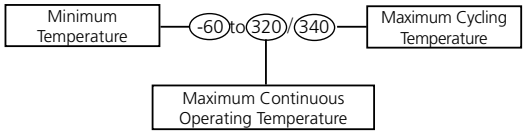


HELPFUL HINTS

A brief conditioning program is recommended as a good routine practice whenever a column is installed or reinstalled after storage, even if only to check the baseline and confirm the system is operating correctly, and is likely to take no more than 60 minutes.

Table 5 – Characteristics of SGE GC stationary phases.

For each SGE GC column phases temperature limits are represented three ways:



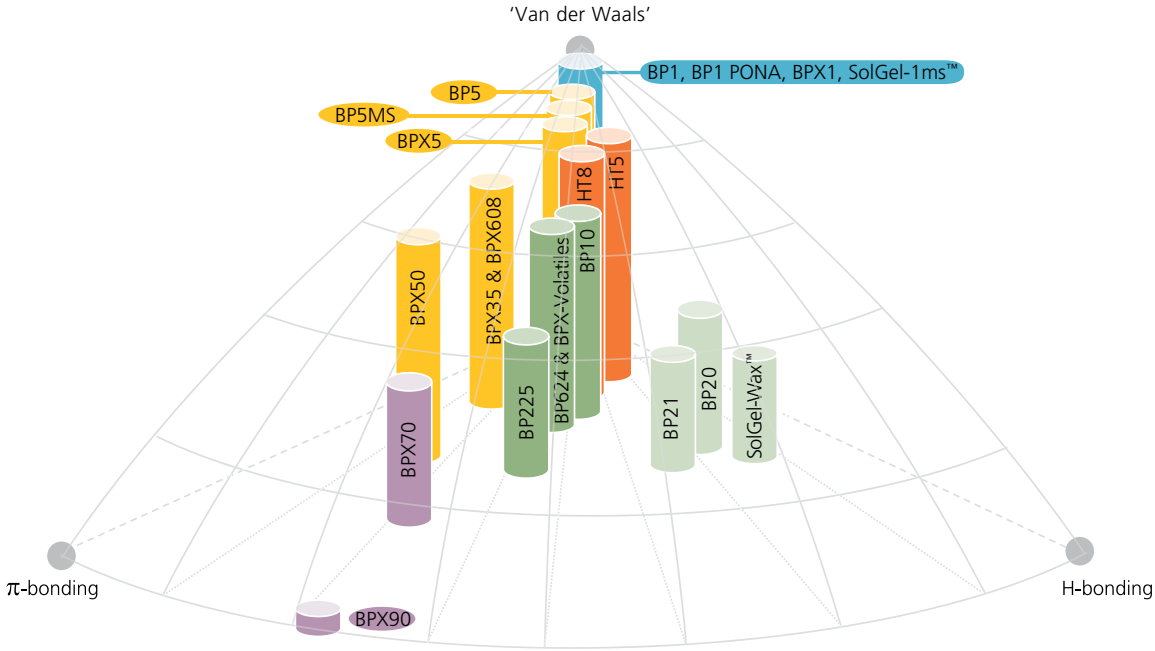
Phase	Description	Phase Attribute	Polarity	Operation Temperatures
BP1	100 % Dimethyl polysiloxane	General purpose column Low bleed	Non-polar	0.1 - 1.0 µm -60 °C to 340/360 °C 1.5 - 3.0 µm -60 °C to 300/320 °C 4.0 - 5.0 µm -60 °C to 280/300 °C
BPX1	100 % dimensionally stabilized methylpolysiloxane	Ideal for simulated distillations e.g. ASTM D2887 in the petroleum industry	Non-polar	Aluminum Clad 0.1 µm film -30 °C to 430/430 °C Polyimide Clad 0.1 - 0.9 µm film -30 °C to 400/400°C 2.65 µm film -30 °C to 370/370 °C
BP5MS	5 % Optimized silphenylene	Low bleed MS grade column for general purpose	Non-polar	-40 °C to 330/350 °C
BPX5	5 % Phenyl polysilphenylene-siloxane	Ideal for GC-MS, ECD Ultra low bleed	Non-polar	0.1 - 1.2 µm - 40 °C to 360/370 °C 1.5 - 3.0 µm - 40 °C to 350/360 °C
BP5	5 % Phenyl 95 % Dimethyl polysiloxane	General purpose column Low bleed	Non-polar	0.25 - 1.5 µm - 60 °C to 340/350 °C > 1.5 µm - 60 °C to 280/300 °C
HT5	5 % Phenyl polysiloxane-carborane	High temperature Well suited to the petroleum C5 - C120 anaylsis	Non-polar	Polyimide Clad 0.1 - 0.5 µm 10 °C to 380/400 °C Aluminum Clad 0.1 - 0.5 µm 10 °C to 460/480 °C
HT8	8 % Phenyl polysiloxane-carborane	Ideal for PCB analysis Low bleed	Non-polar	0.1 - 1.0 µm -20 °C to 360/370 °C
BP10 (1701)	14 % Cyanopropylphenyl	Used in environmental analysis Low bleed	Moderately polar	-20 °C to 280/300 °C
BPX35	35 % Phenyl polysilphenylene-siloxane	Ultra low bleed Ideal for pesticides/herbicides	Moderately polar	0.1 - 1.0 µm 10 °C to 330/360 °C
BPX50	50 % Phenyl polysilphenylene-siloxane	Ultra low bleed Ideal for pharmaceuticals	Moderately polar	0.1 - 1.0 µm 80 °C to 360/370 °C
BP225	50 % Cyanopropylphenyl	Suitable for sugars analysis	Moderately polar	0.1 - 1.0 µm 40 °C to 230/250 °C
BP20 (WAX)	Polyethylene glycol	Suitable for use in GC-MS systems alcohols/free acids	Polar	0.1 - < 1.0 µm 20 °C to 260/280 °C ≥ 1.0 µm 20 °C to 240/260 °C
BP21 (FFAP)	Polyethylene glycol (TPA treated)	Bonded FFAP alcohols/free acids	Polar	0.1 - 1.0 µm 35 °C to 240/250 °C
BPX70*	70 % Cyanopropyl polysilphenylene-siloxane	Optimized for FAME Low bleed	Highly polar	0.1 - 1.0 µm 50 °C to 250/260 °C
BPX90*	90 % Cyanopropyl polysilphenylene-siloxane	Ideal for pesticides, perfumery, aromatics	Extremely polar	80 °C to 280 °C
BPX608	35 % Phenyl polysilphenylene-siloxane	Optimized for separation of EPA 608 Organics Chlorinated Pesticides	Moderately polar	0 °C to 360/370 °C
BP624	Cyanopropylphenyl Dimethyl polysiloxane	Optimized for analysis of EPA drinking water target components	Slightly polar	1.4 µm - 3.0 µm - 0 °C to 230/240 °C
BPX-Volatiles	Cyanopropylphenyl polysilphenylene-siloxane	Optimized for analysis of EPA drinking water target components	Slightly polar	1.0 µm - 3.0 µm - 0 °C to 290/300 °C
BP1-PONA	100 % Dimethyl polysiloxane	Optimized for the analysis of gasoline hydrocarbons	Non-polar	-60 °C to 340/360 °C
SolGel-1ms™*	100 % Dimethyl polysiloxane in a Sol-Gel matrix	High temperature inert GC-MS column	Non-polar	0 °C to 340/360 °C
SolGel-WAX™*	Polyethylene glycol in a Sol-Gel matrix	High temperature inert column Ideal for GC-MS	Polar	30 °C to 260/280 °C
CYDEX-B	Permethylated ß-cyclodextrin in OV1701	Chiral separations	---	30 °C to 220/240 °C

POLARITY SCALE

SGE has redefined the polarity scale!

Different GC capillary phases can separate mixtures using different mechanisms. Our Comprehensive Polarity Scale ranks columns by their separation mechanisms, giving you more information to help choose the phase that's right for your sample.

3D Phase Polarity Scale



- Dimethyl Polysiloxane
- Phenyl Polysilphenylene Siloxane
- Polycarborane Siloxane
- Cyanopropylphenyl Siloxane
- Cyanopropyl Polysilphenylene Siloxane
- Polyethylene Glycol

⚠ CAUTION

Damage to the protective polymer or aluminum coating of the column must be avoided as this will almost certainly result in fracture of the column.

Column ends should be properly sealed when not in use.

Product Warranty

SGE warrants the enclosed capillary column against defective materials, breakage and faulty workmanship for a period of forty five (45) days from the date of shipment. SGE also warrants the column to meet the performance obtained under the conditions given in the enclosed report. This warranty implies free replacement of the column upon receipt of proper proof of the defect.

For assistance at any time regarding column use or selection, please contact your local SGE office, distributor or visit www.sge.com

AUSTRALIA & PACIFIC REGION
SGE Analytical Science Pty Ltd
7 Argent Place, Ringwood
Victoria 3134 Australia
Toll Free: 1800 800 167
Tel: +61 (0) 3 9837 4200
Fax: +61 (0) 3 9874 5672
Email: support@sge.com

JAPAN
SGE Japan Inc
RK CUBE 3F 6-85, Ohta-machi
Naka-Ku, Yokohama-shi
Kanagawa 231-0011 Japan
Tel: +81 45 222 2885
Fax: +81 45 222 2887
Email: japan@sge.com

EUROPE
SGE Europe Ltd
European Head Office
1 Potters Lane
Kiln Farm Milton Keynes
MK11 3LA United Kingdom
Toll Free: 00800 2790 8999
Toll Free Fax: 00800 2626 2609
Tel: +44 1908 568 844
Fax: +44 1908 566 790
Tel France: +33 1 69 29 80 90
Fax France: +33 1 69 29 09 25
Tel Germany: +49 (0) 6155 / 60746 0
Fax Germany: +49 (0) 6155 / 60746 50
Email: europe@sge.com

UNITED STATES OF AMERICA
SGE Incorporated
2007 Kramer Lane
Austin Texas 78758 USA
Toll Free: (800) 945 6154
Tel: +1 512 837 7190
Fax: +1 512 836 9159
Email: usa@sge.com

SGE Analytical Science

www.sge.com