Phone (302)533-5646
Fax (302)737-4547

Column Care

GC column is the central piece for a successful analytical separation using GC instrumentation. A prolonged life and application success of column are often achieved by proper column care and use. Column care includes how to install it, maintenance including storage and regeneration of performance, in some cases.

Column Installation

Pre-Installation

A key element in care is understanding GC instrument basics and its usage history. Since most GC analysis problems are not caused by the column itself, knowledge of the instrument or instrumentation is emphasized. Some listed items that one should know are:

- GC condition: brand new or well used
- GC location change
- GC idling time
- Recent maintenance of GC system including gas line
- Abnormal GC column degradation
- Previous GC column performance record/logbook
- Carrier gas quality

This hints to instrument system performance for future use. Particularly, if previous GC columns exhibited shortened life due to high bleed, ghost peak, peaking tailing, no signal or high baseline signal, it more likely points to problems such as gas leakage, dirty inlet, gas flow or blockage of the detector jet.

If the GC exhibits some of the above symptoms, fix the problem following GC manufacturer's recommendations. Some of common recommendations are:

- Use high purity gases including carrier gas and makeup gas
- Change seals, septum and liner
- Change column ferrules
- Clear jet(s)
- Condition GC inlet and detector at high temperature for hours

Installation

After checking out these practices, the GC instrument is ready for the installation of the GC column. It is vital to understand and know the history of the GC instrumentation since it is usually the cause of poor performance. Once the GC instrumentation parameters are reviewed, proper installation of the GC column is the next step. The recommended step-by-step GC column installation is as follows:

1. Carefully uncoil the column one half coil on both ends
2. Loosely hang the column inside the oven
3. Cut the column on each end 3-5cm with a column cutter as evenly and neatly as possible. Do not hand break column end tip without a cutter
4. Thoroughly examine the cut. Re-cut if the cut across appears uneven.
5. Use ferrules and column nuts that are in good condition
6. Install the column nuts and ferrules to both the column ends
7. Attach column end to the inlet or detector. Follow the instrument manufacturer's specification on column end tip length required, typically 2-3cm for split/split inlet or 1-2mm gap length for the FID jet. Ensure the column end does not touch the metal wall multiple times, as repetitive touching may damage the column tips which impacts sample introduction.
8. Finger-tighten the column nut with another hand holding the column end position for proper insertion, then use the proper size wrench to completely tighten the nuts. Do not over tighten as it may smash the column. Make sure the column tip insert length is within the range of the GC manufacturer's recommendation or specification.
9. Stepwise setup the column pressure to establish column flow
10. Repeat connection to the detector
11. Securely hang the column inside GC oven. Do not over uncoil GC column at each end as it may become broken after thermal cycling
12. Adjust the column flow to the manufacturer's specification. A general flow setting recommendation is as follows:

Column ID (mm)	Column flow range (ml/min)
0.53	5—8
0.32	1.0—2.5
0.25	0.8—1.5
0.2	0.4—1.0

13. Check for any leak signs, avoid bubble testing
14. Set the proper inlet and detector temperatures
15. Set the oven temperature around 50-100°C
16. Turn on the detector
17. Check for a stabilized signal level. If the signal is too low or too high, it may indicate a leaky column connection. Re-install the column and re-slice as needed. Note: Be careful of hot surfaces.

18. Condition columns at its upper temperature limit - (10-20°C) for minimum 30 min. Overnight conditioning is preferred.
19. Check for detector signal. If it is too high, there may be a leaking problem, dirty inlet/detector, or bad column. Find the source of leak, re-install the column, clean the inlet/detector or change another column.
20. Adjust column flow to analysis conditions

Proper column installation will produce consistent and successful analysis results. Taking careful steps in installing will enable the user to become familiar, as well as become effective in troubleshooting steps. However, column storage is also important and must not be overlooked to ensure storage conditions are not a factor in poor testing performance.

Column Storage

Idling inside GC oven

Maintain proper oven temperature, typically 100-150°C under column gas flow.

Outside GC oven

Seal both ends of the column with septa, store the column in the original box. Avoid moisture, particulate/dust or chemical vapor invasion. Minimize any long term vibration from storage environment.

Column Regeneration

Recommended GC Column Conditioning

Condition or bake column at isothermal temperature close to the column upper temperature limit with 2x column working flow for 2 to 24 hours. This regeneration can be effective for sample contamination. Do not exceed the temperature limit because it may cause accelerated degradation of the column if oxygen is present in the carrier gas stream. The surfaces may become too active for many polar compounds.

Multiple Solvent Injections

Injecting solvent multiple times at 50-100°C oven temperature may regenerate column performance. Avoid setting oven temperature below the solvent boiling point as solvent condensation will wash out the stationary phase. The column surface may become less inert after multiple injections.

Trim Column Ends

Trim both ends of column by 0.1-1m is an easy and effective way to regenerate column performance. Trim longer lengths at the detector end than at inlet end. Adjust instrument conditions accordingly to maintain retention time locking. Condition the column for 30-120 mins after trimming. Be careful not to trim an excessive amount.

Solvent Rinse

Offline solvent rinse is an old fashion to regenerate column performance. As column becomes commodity product, we do not recommend this technique. Instead, we recommend solvent injection as an alternative.

In extreme cases, use high grade solvents compatible to stationary phase, such as non-aqueous solvent for wax column or hexane/toluene solvents for polysiloxane column. Columns should only be rinsed with 2 to 3 the column volume solvent at low pressure. Avoid rapid pressure changes to minimize potential column breakage. Gas purge dry column for 30-60 min after rinse and follow up with proper column conditioning.

Recommended MS Grade Column Conditioning

MS grade columns are specially made by manufacturer to minimize column bleed at its maximum isothermal temperature. Because of the time consuming MSD operation, column manufacturers usually do not test MS column by GC-MSD. Column bleeds are often measured by GC-FID. As MSD is operated at ultra-high vacuum pressures, new MS columns are more likely to exhibit high column bleed initially after installation. This requires that newly installed MS column be carefully conditioned prior to use.

1. Before installing new MS columns, check for the latest working condition of MSD, such as auto tune, background, noise, vacuum, ion source cleanliness, helium line trap, etc. For more details, refer to the MSD manufacturer's instruction.
2. Properly pump-down the MSD, power off the instrument as needed before column installation. If the instrument is equipped with a non-vent connector, maintain adequate helium flow at all times. Maintain MSD cleanliness as much as possible.
3. Install the MS column with ferrules and fittings that are in good condition. Cut column MSD end about 3 to 5cm to have a clean 10 to 15 cm section of the column. Insert the column to the GC-MSD transfer line to the manufacturer specified length.
4. Setup proper column gas flow
5. Set the oven temperature to 50-60°C
6. Power on the instrument and pump up the MSD

7. Wait for complete MSD pump up. Check for any signs of leak by following the manufacturer's guideline.
8. Setup the proper MSD parameters in accordance with the manufacturer's specification.
   Steps 9 to 12 can be substituted with MSD manufacturer's procedure.
9. Once MSD vacuum is established, increase the oven temperature to 100°C. Maintain this temperature for minimum 30min.
10. Manual Baking-out: (for 5MS column only) Program the GC oven temperature for column baking: 60°C (30min) 10°C/min to 280°C (10min) 10°C/min to 300°C (15min), 10°C/min to 325°C (30min). If it is an offline MSD, cut off one coil of the column at detector end and connect this end of the column to the MSD.
11. Manual Baking-out: (for other MS columns) Program the GC oven temperature: 60°C (30min) 10°C/min to the column upper temperature limit-20°C (30min), 10°C/min to the column upper temperature limit (30min).
12. Do not run the background check, noise check, autotune or other MSD methods during this manual baking-out period.
13. After completion of the manual baking out, run autotune to see check spectra readings: 18 (water), 28(nitrogen), 32 (oxygen), 79, 207 and 281 are high. If the air peak is high, check for gas leak signs.
14. If the spectrum 207 is less than 5% of the tune spectra (such as PFTBA), e.g. 219, run a background check. Otherwise, repeat column baking-out as described in step 10.
15. If a longer backing time is not possible or if column exhibits high bleed, repeat solvent injection. Solvents such as hexane, toluene, and dichloromethane can be used. Injection amount ranges from 2 to 5ul. Injection times should be 3-6mins. Time intervals between two solvent injections should be 5—10min. Oven temperature should be set around the column upper temperature limit.
16. If the high column bleed still persists, manually decrease the temperature of the transfer line from 320/280°C to 250°C to see if the column bleed decreases accordingly. If the column bleed decreases, use the 250°C transfer line temperature temporarily.
17. After solvent injection and the column bleed is still persistently high, the column may be defective or damaged by oxidation.
18. Occasionally check for gas line leaks from the gas supply to the injector, including septum leaks. Use new ferrules or ferrules in fairly good condition. Do not over tighten. Follow the instrument manufacturer's troubleshooting /maintenance guidance.

MS columns do not require a long conditioning period prior to use. If high column bleed is persistent, it is recommended to change to a new MS column. Extensive periods of MS column conditioning time will result in contamination of the MSD and may damage column from leak in gas path or oxygen in carrier gas stream.

Practical Tips:

- Check carrier gas, supply pressure, trap, and gas valve
- Always run the carrier gas through the MS column
- Check leaks at septum or inlet. Use new septum if possible.
- Check for proper ferrule use for MSD
- Check for column breakage
- Do not over-tighten column nuts to MSD transfer line
- Make sure MSD is functioning properly
- Keep MSD ion source as clean as possible
- Run oven temperature at application maximum temperature, not the column maximum temperature to minimize column "bleed"

PLOT Column Installation and Conditioning Recommendations

1. Carefully loosen a comfortable working length of column from cage at each end. Avoid excessive vibration at column tips.
2. Cut 1 to 5cm from each column end
3. Install column to the GC inlet per GC manufacturer's suggestion
4. Do not connect the PLOT column to GC detector
5. Setup proper column flow, check for any breakage during column flow buildup. Use bubbling technique for a quick flow check.
   a. Recommended column flow of 6 - 10ml/min for 0.53mm ID column and 2-4ml/min for 0.32mm ID column. Measure flow with a flowmeter as needed
6. Recommended column flow of 6 - 10ml/min for 0.53mm ID column and 2-4ml/min for 0.32mm ID column. Measure flow with a flowmeter as needed
7. Leave the column detector end either inside oven or outside oven
8. Program a brief condition temperature profile. Lower temperatures for 2-5 mins, 150°C for 10min, higher temperatures for 10-30 mins.
9. Decrease oven temperature, recheck column flow as necessary, install the column to the GC detector while avoiding excessive vibration.
10. Light the detector and check for unusually high signal noise. If noise appears too high, cutoff 1 - 5cm of the column tip and reconnect the column detector side connection.
11. Repeat the brief column condition temperature profile to establish a stable baseline with less noise
12. Condition column as needed to activate PLOT column and decrease signal level

Troubleshooting

GC is a complicated system and basic knowledge of troubleshooting technique in the following areas is deemed useful: method, sample, instrument, column and software. These components are closely correlated and affect each others performance. When a problem is arises, there is often is no single root-cause. Hurrying to solve problems may be a temporary fix due to time constraints such as changing a column, but often leads to overlooking the real root-cause and the problem may manifest itself again. However, a planned and systematic approach to troubleshooting tends to yield a faster and more accurate way to solve an issue.

Causes of Column Degradation

There are various root causes for column performance degradation. Most root causes are related to oxygen present in the carrier gas stream (oxidation), thermal damage, and sample contamination. Check for obvious instrumentation issues as dictated above before attempting more time-consuming troubleshooting actions. The following table gives lays out general troubleshooting process and some remedies. It is important to follow proper column installation step and to maintain

decent maintenance of instrument to prevent column performance degradation.

Table 1 – Troubleshooting General Guide for Column Degradation

Degradation Symptoms	Root cause	From	Recommended Corrective Actions
-High column bleed -Column selectivity change/ shift -Retention time shift -Chopping baseline profile -Peak tailing -Blockage of detector jet and abnormal signal level	-Oxygen in carrier gas flow path -Bad column	- Low grade carrier gas used - Leak in gas line connection - Instrument leak: inlet, pressure regulator, valve - Home gas plumb connection with dead/void volume in the flow path	-Change carrier gas grade -Use traps -Properly plumbing -Fix instrument leaks -Purge column for a long period of time at low temperatures -Avoid high temperature chromatography -Change column
-Peak tailing -Unstable or noisy detector signal - Retention time shift	-Moisture present in gas stream line -Sample contamination -Thermal damage at temperature over column upper limit	-System off for long time -Low grade carrier gas -Large dirty sample introduction	-Use trap in carrier gas line -Bake instrument -Do not turn off instrument unless necessary -Good sample preparation -Trim contaminated column ends by 0.1-0.5m
-Accelerated column bleed -Significant column selectivity change -Severely peak tailing -Peak broadening	-Thermal damage	-Oven temperature exceeding the column upper temperature limit -Too high inlet/detector temperatures -Combination of oxidation, duration time and column temperature	-Almost irreversible degradation -Trim column each end by 0.5-1m -Reduce used temperatures -Switch high grade carrier gas
-Column breakage	-Human error -Instrumentation error -Bad column	-Bad column -Gas pressure pulse -Fast temperature ramping up/down - Over tightening column nut	-Slowly setup carrier gas pressure with EPC -Reduce temperature ramping -Butt-connect the broken column -Re-install column

Troubleshooting problems is a vast subject and covering every possible problem and its solution is challenging. However, we offer some basic troubleshooting techniques based on our column expertise.

1. **Define and clearly state the problem**

2. **Check for obvious mistakes and avoid overlooking simple steps**

   Document logs of events will provide internal guidance and will help maintain a good working condition for your GC system and prolonged column lifetime. The following list covers simple and easy related causes:

   - Instrument conditions: any power outrage, temperature setting of injector, detector, oven and aux, EPC/EFC setting, valve on/off, cable connection, oven heater/sensor, etc.
   - Instrument maintaining and repair log. Is it a time to maintain instrument?
   - Software: Does software function properly and correctly?
   - Sample: sample shelf time, sample drain from solvent evaporation thru the punched cap, inlet septum leak
   - Gases: Supply gas drain or insufficient pressure, valve on/off, gas line leak, log grade gas used, gas cylinder changeover, wrong gas connection, etc

3. **Troubleshooting based on Performance**

   - Baseline
     a. Wandering: inlet leaking, loose connections at inlet or detector side, carrier gas contamination, detector contamination, column contamination, inlet/detector heating zone temperature varying
     b. Drafting: downward: column not thoroughly conditioned, instrument not stabilized; upward: column bleed from oxidation, instrument contamination
     c. Spiking: dirty detector: dust inside inlet/detector/column, electrical noise
     d. Noise: dust in detector, detector gas and gas flow, column contamination or oxidation, PCB issue
     e. Offset: chip in inlet, column installation at detector side, unstable carrier gas flow (clogging or inconsistent leaking)
   - Peak
     a. No peak at all: column broken, column not properly installed, non carrier gas, detector off, syringe needle clogged, no sample introduction, valve off/ on, wrong signal channel
     b. Missing peak: column improper installation at inlet/detector, column contamination, inlet contamination, column selectivity change over time, column oxidized, solvent effect, aged sample, improper syringe/liner used
     c. Peak shape broadening: too much sample loading, very low split flow, split valve off, sample becomes concentrated over time, column is oxidized, low oven temperature, part of column outside oven, heating zone is not properly setup
     d. Peak(s) is too small: inlet septum leak, syringe needle clogging, improper column installation including loose connection, split flow change, liner contamination (for active compounds), column contamination, column inertness, vial/syringe quality issue, gold seal contamination, lower oven temperature
     e. Peak tailing: column inertness or column contamination, improper column installation(mostly at inlet), dirty inlet/detector, active butt-connector

for column-column connection, effect of solvent-stationary phase match, increased column activity from high temperature run, unknown active compounds, sample overloading, column performance decreased, column oxidized

f. Peak fronting: too much sample loading, sample is too concentrated, column performance decreased, split flow decreased from error or clogging
g. Split peak: poor sample injection technique, dual sample injections, cool inlet, improper column installation, column not neat cut, dirty liner, column selectivity change over time, oven temperature changed

- Elution and retention time
  a. Elution order change: column selectivity change over time, solvent effect, mismatch of column (stationary phase and solutes), oven temperature change, carrier gas flow change, ghost peak from contaminations or carry over contamination, wrong peak identifications
  b. Retention time shifting: leak in inlet, carrier gas flow shifting/change, oven temperature shifting, column contamination, solvent effect, column performance decreased (phase stripping by solvent, phase oxidizing)
- Separation
  a. Resolution decreased: improper column installation, column selectivity change overtime, column contamination, oven temperature change, too much sample introduction, carrier gas flow change, column performance decreased

4. Practical Tips

a. Good column installation techniques and periodic checks for proper installation should be practiced
b. Identify problem, state and analyze problem logically and systematically
c. Check and fix obvious errors or mistakes
d. Look for available expertise to fix protblem efficiently and effectively
e. Perform corrective and preventative maintenance on instrumentation
f. Develop your troubleshooting skills from educational sources and from knowledgeable peers
g. Quick fixes: re-install column, cut column both ends off, baking column at high temperatures, solvent injection and changing to a different solvent

5. Column Performance Degradation

Columns from a manufacturer should have good performance initially. Under careful control and minimal error conditions, column should perform well for a long time. But in reality, many columns lose their performance earlier than their typical lifetime. Column performance loss is usually caused by:

- Oxidation from leaking, impurity in carrier gas
- Improper instrumentation conditions
  * temperature is higher than column upper temperature limit
  * too fast temperature program run for fast analysis
  * installation
  * improper precondition and post run
  * too much sample loading
- Column contamination
  * sample containing high boiling point compounds
  * reaction of active compounds to stationary phase
  * carry-over contamination from instrument and sample
  * accumulating moisture effect
- Incompatible /mismatched solvent to column stationary phase

Symptoms of column performance degradation:

- Loss or decrease retention
- Loss of sample loading capacity
- Peak tailing
- Peak size reduced
- Peak broadening
- Peak missing
- Resolution decreased

Take care of the column, because it is critical tool that determines the analysis success.