GC Packed Column Information

Preconditioning Instructions

To precondition a GC packed column, install the column to the injection port only. Do not attach the column to the detector port. Initiate a flow of carrier gas that would be normal for the column. (i.e. 25cc/min for 1/8" metal or 2mm i.d. glass; 50cc/min for 1/4" metal or 4mm i.d. glass). The carrier gas should be free of moisture, oils and oxygen. We highly recommend the use of the Alltech® Gas Purifier and Oxy-Trap™ for this purpose.

The preconditioning should proceed at room temperature for 30 minutes. You may then program (either automatically or manually) to the desired temperature. The upper temperature should be 25°C lower than the upper temperature limit listed on the column tag. Alternatively, for longer column life, condition at 25°C above the maximum operating temperature used in your analysis as long as this doesn't exceed the upper temperature limit minus 25°C. For example, AT-1000 has an upper temperature limit of 250°C. The maximum recommended temperature for preconditioning is 225°C. If however, your analysis will be conducted at 115°C, precondition at 140°C and your column will last longer.

The preconditioning should continue for 4-48 hours at the desired temperature. Overnight conditioning is usually sufficient, but for some phases and sensitive electron capture work, the full 48 hours may be necessary. Cool the column completely with carrier gas flowing. At this time the column can be connected to the detector for use.

If a column has been out of use for a while it should be reconditioned briefly. This time, it can be connected to the detector. Purge with pure carrier gas for 30 minutes, then program up to the desired temperature (carrier gas still flowing) and hold for 30 minutes to 2 hours before starting analysis.

Exception - Molecular Sieve columns should be preconditioned for 4-16 hours at 300°C. If you have any further questions regarding column handling, please call our Technical Support Department at 1-800-255-8324.

Troubleshooting Common Problems

Unresolved Peaks on a Column Which Worked Well in the Past

Possible Cause	Possible Solution
1. Wrong column temperature.	**1.** Check and adjust temperature.
2. Wrong carrier gas flowrate.	**2.** Check and adjust flowrate.
3. Sample size too large.	**3.** Reduce sample size.
4. Poor injection technique (too slow).	**4** Use a rapid, smooth injection technique.
5. Column contaminated or deteriorated.	**5.** Recondition column, repack first 6" of column, or replace column.

Tailing Peaks

Possible Cause	Possible Solution
1. Active sample adsorbing on injection port, transfer lines, column, or glass wool.	**1.** Use more inert system: Deactivated surfaces.
2. Column or injection port temperature too low.	**2.** Increase temperature (do not exceed maximum temperature for column). Injection port should be 25°C greater than the highest boiling point in sample.
3. Two compounds coeluting.	**3.** Increase sensitivity and reduce sample size. Lower temperature 10-20°C to resolve peaks.
4. Needle hitting packing in column inlet.	**4.** Remove some packing.
5. Column deteriorating.	**5.** Replace column.
6. Column contaminated.	**6.** Recondition.

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Troubleshooting Common Problems Continued

Irregular or Unstable Baseline	
Possible Cause	**Possible Solution**
1. Column bleed or contamination.	**1.** Recondition column.
2. Contaminated detector or injection port.	**2.** Clean detector and/or injection port.
3. Carrier gas leak.	**3.** Change septum and check for column leaks.
4. Inconsistent carrier gas regulation.	**4.** Check carrier gas supply for sufficient pressure. Replace tank if ≤500psig pressure.
5. Gas impurities or contaminated gas line.	**5.** Change gas tank, use gas purifiers, and clean metal tubing.
6. Gas flows not within minimum / maximum limits of the instrumentation (including hydrogen and air on FID), or poorly regulated flow.	**6.** Measure flows and verify against manual specifications.
7. Defective detector.	**7.** Troubleshoot detector.
8. Septum bleed.	**8.** Condition septum or replace septum.

Ghost Peaks	
Possible Cause	**Possible Solution**
1. Column adsorption and subsequent desorption of sample	**1.** Try more inert packing material.
2. Contaminated syringe	**2.** Try new syringe with clean solvent. If ghost peaks disappear, clean syringes more thoroughly.
3. Adsorption in transfer line	**3.** Use glass-lined stainless steel for transfer lines.
4. Poor injection technique	**4.** Use rapid, smooth injection technique.

Retention Times Longer (or Shorter) on Same Column	
Possible Cause	**Possible Solution**
1. Column temperature too low or too high.	**1.** Check and adjust temperature.
2. Carrier gas flowrate too low or too high.	**2.** Measure flowrate with a properly calibrated source of measurement atcolumn exit and adjust.
3. Septum or column leak.	**3.** Check and correct if needed.
4. Column contaminated or deteriorated.	**4.** Recondition column or replace column.
5. Sample overload.	**5.** Reduce sample size.
6. Recorder defect.	**6.** Check Recorder.

Fronting Peaks	
Possible Cause	**Possible Solution**
1. Column overload.	**1.** Decrease sample size.
2. Two components coeluting.	**2.** Decrease sample size, decrease temperature, and increase sensitivity.
3. Sample condensation.	**3.** Check injection port and column temperatures. Increase if necessary.
4. Sample decomposition.	**4.** Use inert system and deactivated packing.

Broad Solvent Peaks	
Possible Cause	**Possible Solution**
1. Dead volume in injection port.	**1.** Use smaller volume injection liner and/or on-column injection.
2. Using extremely dilute samples.	**2.** Adjust sample or decrease oven temperature to condense solvent then program up.
3. Poor injection technique.	**3.** Use rapid, smooth injection techniques.
4. Injection port temperature too low.	**4**. Increase injection port temperature.
5. Sample solvent interacts with the detector.	**5.** Change sample solvent.
6. Sample solvent retained by column.	**6.** Change sample solvent.

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