GC Troubleshooting Guide

Your guide to solving common problems and staying productive

Agilent

Checking the Basics

A surprising number of problems involve fairly simple and often overlooked components of the GC system or analysis. Many of these items are transparent in the daily operation of the GC and are often taken for granted ("set it and forget it"). The areas and items to check include:

- Gases, pressures, carrier gas average linear velocity, and flow rates (detector, split vent, septum purge)
- Temperatures (column, injector, detector, and transfer lines)
- System parameters (purge activation times, detector attenuation and range, mass ranges etc)
- Gas lines and traps (cleanliness, leaks and expiration)
- Injector consumables (septa liners, O-rings, and ferrules)
- Sample integrity (concentration, degradation, solvent, and storage)
- Syringes (handling technique, leaks, needle sharpness, and cleanliness)
- Data system settings and connections

Condensation Test

Use this test whenever injection or carrier gas contamination problems are suspected (e.g. ghost peaks or drastic tailing).

1. Leave the GC between 40 to 50 °C for 8 or more hours.
2. Run a blank analysis (i.e. start the GC run with no injection) using the normal temperature conditions and instrument settings.
3. Collect the chromatogram for the blank run.
4. Immediately repeat the blank run when the first one is completed. Do not allow more than 5 minutes to elapse before starting the second blank run.
5. Collect the chromatogram for the second blank run and compare it to the first chromatogram.
6. If the first chromatogram contains a larger amount of peaks and baseline instability, the incoming carrier gas line or the carrier gas is contaminated.
7. If both chromatograms contain few peaks or little baseline drift, the carrier gas and incoming carrier gas lines are relatively clean.

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Ghost Peaks or Carryover

Possible Cause	Solution	Comments
Contaminated syringe	Wash or replace syringe	Use solvent blanks to check for carryover
Septum bleed	Use high-temperature, low-bleed septa; condition septa before use	Use septa rated for 10–20 °C above maximum operating temperature; replace septa frequently
Sample residue	Bake out column at high temperature	Follow column manufacturer's recommendations for conditioning
Contamination in gas lines or traps	Check gas purity and replace traps if needed; install a new gas filter	Check moisture and hydrocarbon traps
Sample residue in injection port (e.g. liner, seal, or packing)	Clean or replace liner, seal, or packing; bake out inlet at high temperature	Use deactivated liners

Excessive Baseline Noise

Possible Cause	Solution	Comments
Septum bleed	Use high-quality, low-bleed septa; condition septa before use	Use septa rated for 10–20 °C above maximum operating temperature
Column bleed	Bake out column; trim column	Follow column manufacturer's recommendations for conditioning
Contaminated detector	Clean detector	Follow detector manufacturer's recommendations
Leak in system	Locate and fix leak	Check for leaks after any change in system
Contaminated carrier gas	Check gas purity; replace gas if needed; install or change gas filters	Use high-purity gas and gas filters
Contaminated detector gases	Check gas purity; replace gas if needed; install or change gas filters	Use high-purity gas and gas filters
Detector not optimized	Optimize detector	Refer to detector manual
Column not conditioned	Condition column	Follow column manufacturer's recommendations for conditioning

Baseline Instability or Disturbances

Possible Cause	Solution	Comments
Septum bleed	Clean; Trim column	Use high-quality, low-bleed septa; condition septa before use
Column bleed	Bake out column	Follow column manufacturer's recommendations for conditioning
Contaminated detector	Clean detector	Follow detector manufacturer's recommendations
Contaminated carrier gas	Check gas purity; replace gas if needed; install or change gas filters	Use high-purity gas and gas filters
Contaminated detector gases	Check gas purity; replace gas if needed; install or change gas filters	Use high-purity gas and gas filters
Detector not optimized	Optimize detector	Refer to detector manual
Leak in system	Locate and fix leak	Check for leaks after any change in system

Fronting Peaks

Possible Cause	Solution	Comments
Column overload	Reduce amount of sample on column (dilute sample, increase split ratio, decrease injection volume, or use higher capacity column)	Use higher split ratio or dilute sample
Incompatible solvent/stationary phase	Change solvent or stationary phase	Use solvent appropriate for stationary phase
Injection technique	Change injection technique	Use proper injection technique
Column not conditioned	Condition column	Follow column manufacturer's recommendations for conditioning
Poor column installation	Reinstall column	Follow column manufacturer's recommendations for installation

Tailing Peaks

Possible Cause	Solution	Comments
Active sites in inlet system	Clean or replace liner; replace seal or packing; trim column	Use deactivated liners
Column overload	Reduce amount of sample on column (dilute sample, increase split ratio, decrease injection volume, or use higher capacity column)	Use higher split ratio or dilute sample
Column contamination	Condition column; trim column	Follow column manufacturer's recommendations for conditioning
Incompatible stationary phase	Use different column	Use column appropriate for application
Improper column installation	Reinstall column	Follow column manufacturer's recommendations for installation
Poor column cut	Trim column	Use proper technique when cutting column
Dead volume in system	Check for leaks; reinstall column	Check for leaks after any change in system

Split Peaks

Possible Cause	Solution	Comments
Improper injection	Change injection technique	Use proper injection technique
Poor column installation	Reinstall column	Follow column manufacturer's recommendations for installation
Poor column cut	Trim column	Use proper technique when cutting column
Incompatible solvent/stationary phase	Change solvent or stationary phase	Use solvent appropriate for stationary phase
Column not conditioned	Condition column	Follow column manufacturer's recommendations for conditioning

Retention Time Shift

Possible Cause	Solution	Comments
Change in column temperature	Check temperature settings	Check temperature settings
Change in carrier gas flow rate	Check flow rate; replace gas if needed	Check flow rate; replace gas if needed
Change in column dimensions	Use different column	Use column appropriate for application
Leak in system	Locate and fix leak	Check for leaks after any change in system
Change in carrier gas composition	Check gas purity; replace gas if needed	Use high-purity gas
Change in column stationary phase	Use different column	Use column appropriate for application

Change in Peak Size

Possible Cause	Solution	Comments
Change in detector response	Check gas flow rates; check detector settings	Check gas flow rates; check detector settings
Change in sample concentration	Check sample preparation procedure	Use proper technique
Change in injection volume	Check syringe and injection technique	Use proper technique
Change in injection technique	Check injection technique	Use proper technique
Change in column temperature	Check temperature settings	Check temperature settings
Change in carrier gas flow rate	Check flow rate; replace gas if needed	Check flow rate; replace gas if needed
Change in column dimensions	Use different column	Use column appropriate for application
Leak in system	Locate and fix leak	Check for leaks after any change in system
Change in carrier gas composition	Check gas purity; replace gas if needed	Use high-purity gas
Change in column stationary phase	Use different column	Use column appropriate for application

Loss of Resolution

Possible Cause	Solution	Comments
Change in column temperature	Check temperature settings	Check temperature settings
Change in carrier gas flow rate	Check flow rate; replace gas if needed	Check flow rate; replace gas if needed
Change in column dimensions	Use different column	Use column appropriate for application
Leak in system	Locate and fix leak	Check for leaks after any change in system
Change in carrier gas composition	Check gas purity; replace gas if needed	Use high-purity gas
Change in column stationary phase	Use different column	Use column appropriate for application
Poor column installation	Reinstall column	Follow column manufacturer's recommendations for installation
Poor column cut	Trim column	Use proper technique when cutting column
Dead volume in system	Check for leaks; reinstall column	Check for leaks after any change in system
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1. Understanding the eBook Instruction Guide Agilent
    - The Rise of Digital Reading Instruction Guide Agilent
    - Advantages of eBooks Over Traditional Books
2. Identifying Instruction Guide Agilent
    - Exploring Different Genres
    - Considering Fiction vs. Non-Fiction
    - Determining Your Reading Goals
3. Choosing the Right eBook Platform
    - Popular eBook Platforms
    - Features to Look for in an Instruction Guide Agilent
    - User-Friendly Interface
4. Exploring eBook Recommendations from Instruction Guide Agilent
    - Personalized Recommendations
    - Instruction Guide Agilent User Reviews and Ratings
    - Instruction Guide Agilent and Bestseller Lists
5. Accessing Instruction Guide Agilent Free and Paid eBooks
    - Instruction Guide Agilent Public Domain eBooks
    - Instruction Guide Agilent eBook Subscription Services

- Instruction Guide Agilent Budget-Friendly Options

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   - Instruction Guide Agilent Compatibility with Devices
   - Instruction Guide Agilent Enhanced eBook Features
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   - Adjustable Fonts and Text Sizes of Instruction Guide Agilent
   - Highlighting and Note-Taking Instruction Guide Agilent
   - Interactive Elements Instruction Guide Agilent
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   - Joining Online Reading Communities
   - Participating in Virtual Book Clubs
   - Following Authors and Publishers Instruction Guide Agilent
9. Balancing eBooks and Physical Books Instruction Guide Agilent
   - Benefits of a Digital Library
   - Creating a Diverse Reading Collection Instruction Guide Agilent
10. Overcoming Reading Challenges
    - Dealing with Digital Eye Strain
    - Minimizing Distractions
    - Managing Screen Time
11. Cultivating a Reading Routine Instruction Guide Agilent
    - Setting Reading Goals Instruction Guide Agilent
    - Carving Out Dedicated Reading Time
12. Sourcing Reliable Information of Instruction Guide Agilent
    - Fact-Checking eBook Content of Instruction Guide Agilent
    - Distinguishing Credible Sources
13. Promoting Lifelong Learning
    - Utilizing eBooks for Skill Development
    - Exploring Educational eBooks
14. Embracing eBook Trends

- Integration of Multimedia Elements
- Interactive and Gamified eBooks

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