GC Troubleshooting*

Environmental Applications: Selected GC Column Phases and Features

Applications	US EPA Method	HP Phase (increasing polarity)	Composition
Headspace—Volatiles	504	HP-1	Dimethyl polysiloxane
General purpose trihalomethanes	501.1, 501.2, 501.3	HP-5	5% Phenyl
Semivolatiles 1618—Organophosphorus Pesticides	525, 625, 8270	HP-5 MS	Low-bleed 5% Phenyl
Phenols—Base neutrals, Semivolatiles, Polar compounds	625, 1625, 8270	HP-5 TA	Low-bleed 5% silphenylene Highly deactivated
Purgeable halocarbons, Volatiles, Chlorofluorocarbons	501.3, 502.2, 503.1, 524.2, 601, 602, 8010, 8015, 8020	HP-624	6% Cyanopropyl-phenyl 94% Dimethyl siloxane copolymer
Volatile organics, Purgeables	502.2, 524.2, 601, 602, 8024, 8260	HP-VOC	Intermediate polarity
Pesticides, Alcohols, PCBs, and Herbicides	505, 508, 608, 617, 8080, 8081	HP-1301	6% Cyanopropyl-phenyl 94% Dimethyl siloxane copolymer
Aroclors, Amines, Pesticides, Herbicides, N-P-Pesticides	507, 614, 619, 622, 8140, 8141, 8081	HP-35	35% Diphenyl 65% Dimethyl siloxane copolymer
Chlorinated Pesticides, Phenols, Semivolatiles	608, 625, 1625, 8270	HP-608	Intermediate polarity
Chlorphenoxy acetic acids Organo chlorine pesticides	515, 608, 615, 8081, 8150, 8151	HP-1701	14% Cyanopropyl-phenyl 86% Dimethyl siloxane copolymer
Polar compounds, Phenols, Organo chlorine pesticides	552, 552.1, 604, 605, 607, 614, 619, 622, 8140	HP-50+	50% Diphenyl 50% Dimethyl siloxane copolymer

GC/MS Troubleshooting*

Symptom	Possible Cause	Solution
Does not autotune to DFTPP/BFB	Tune targets incorrect Software/hardware problem	Manual tune, correct target ratios Try alternate tune
Cannot manual tune to DFTPP/BFB	Software/hardware problem	Try alternate tune
Low response to all compounds	Split time incorrect EM voltage incorrect EM aging Septum leaking Injection needle partially plugged	Check split times Compare with autotune EM voltage Increase EM voltage or replace EM Replace septum Replace needle
Low response to some compounds	Dirty injection port Dirty front end on column Temperatures set incorrectly	Replace injection liner Remove 1 ft from front of column Compare temperature zones to previous work
Poor replicate injection response	Poor injection, manual injection Carrier gas low	Check with autosampler Check carrier gas pressure
Poor replicate sample response	Poor sample preparation	Check with replicate injection of same sample
No response	Instrument not on Vacuum pump tailed Filament burned out Column broken Column plugged	Turn power on Check vacuum Go to alternate filament or replace filament Check column Inject air or other gas Look for signal

**Reference: "GC Inlets: An Introduction," Matthew S. Klee, Ph. D., Agilent Technologies 1990, Part Number 5958-9468.*

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GC Troubleshooting*

Capillary Column Analysis

Split Inlet

Symptom	Possible Cause	Solution
Low peak areas, lost peaks, generation of new peaks	Inlet too hot	Reduce inlet temperature 50°C, reevaluate
	Dirty inlet	Clean/replace liner
	Contact with metal	Use glass columns, liners
	Compounds too labile	Derivatize the sample Use cool on-column injection
	Active packing	Remove packing
	Active liner	Change liner type Deactivate liner
	Residence time too long	Increase split flow Increase column flow
Low areas for late eluters	Solvent BP too low	Use higher-boiling solvent
Needle discrimination	Inlet temperature too low	Increase inlet temperature to 50°C, reevaluate
	Needle dwell time too long	Use fast autoinjector
Inlet discrimination	Inlet temperature too high	Decrease to 50°C
	Inlet dwell time too short	Reduce split flow
	No glass wool or in wrong place	Center in the liner
	Split flow too high	Decrease split flow
	Injection volume too big	Decrease injection volume
Wide peaks	Split flow too low	Increase split flow
	Adsorption in Inlet	Change the liner Remove packing Increase inlet temperature
	Column overloaded	Increase the split flow
Area irreproducibility	Fluctuation in split ratio	Check the flow controllers Check for leaks (septum, liner, column)
	Sample flashback	Reduce the sample size Reduce the inlet temperature Use a large liner
	Variable injection volume	Check the injection technique Use on autoinjector
	Decomposition	Remove liner packing Decrease inlet temperature
Retention time irreproducibility	Overload	Increase split ratio Inject less
	Column degradation	Cut 0.5 m off inlet end Replace column

Splitless Inlet

Lost peaks	Inlet too hot	Reduce temperature 50°C and try again
Skewed peaks, Artifact peaks	Active packing	Remove/minimize packing (degradation)
	Active liner	Change liner Deactivate liner
	Liner too small	Use larger-volume liner
	Long residence time	Increase column flow rate
Wide peaks	No solvent effect	Reduce oven temperature Use a higher-boiling solvent
	No stationary phase focusing	Reduce initial column temperature
Split peaks	Solvent/column not compatible	Use a different solvent Use a retention gap
Area Irreproducibility	Flashback	Reduce injection volume Use higher-boiling solvent Use larger liner
	Purge time or flow variability	Check purge on/off times
Retention time irreproducibility	Inaccurate purge delay	Check and correct
	Incompatible solvent	Use retention gap

Cool On-Column

Peak loss, artifact peaks	Active retention gap or column	Change or deactivate the retention gap (degradation); Clean or replace the column
Wide peaks	Insufficient focusing	Lower the column temperature
	No solvent effect	Reduce the oven temperature Use higher-boiling solvent
	Column overloaded	Dilute the sample Inject less Use a thicker film column
Split peaks	Solvent and column not compatible	Use a different solvent Use a retention gap
	Solvent and major-component interaction	Dilute the sample Use another inlet
Area irreproducibility	Sample too big	Reduce injection volume

Capillary Column Analysis

PTV

Symptom	Possible Cause	Solution
Lost peaks, artifact peaks (degradation)	Active packing	Remove the packing
	Active liner	Change the liner type or deactivate liner
	Liner too small	Use a larger liner Ramp temperature slower
	Residence time large	Increase column flow rate
Wide peaks	No solvent effect	Reduce oven temperature Use higher-boiling solvent
	No stationary-phase focusing	Reduce the initial column temperature
	Slow sample transfer from inlet	Increase inlet temperature ramp
Split peaks	Solvent/column not compatible	Use a different solvent Use a retention gap Try solvent elimination mode if early peaks are not important
Area irreproducibility	Sample too big Purge time or flow variability	Reduce injection volume Check instrument and correct

Auxiliary Sampling Devices

Headspace

Symptom	Possible Cause	Solution
Sample degradation	Transfer lines too hot	Reduce temperature
	Vial temperature too hot	Reduce bath temperature
Baseline perturbations	Valve resetting during run	Increase "inject time"
	System contamination	Bake out valve and transfer line Clean/replace sampling needle
Peak tailing, broad peaks	System flow too slow	Increase headspace flow Decrease GC flow Increase split flow
	System voids	Check connections Reduce liner volume (id) Reduce volume of connecting tubing
	Insufficient focusing	Use columns with lower ß Lower initial column temperature
Peak areas too small	Equilibration time too short	Increase time
	Vial temperature too low	Increase 20°C, reevaluate
	Vent time too short or too long	Adjust
	Vial cap leak	Use new sample Reseal vial
	Leaking inlet septum	Replace/tighten septum
	Leaking connections	Inspect and reseal connections
	Split flow too high	Reduce split/GC flow
Sample contamination	Sample exposed too long before sealing	Seal immediately Minimize transfer times
	Ambient air contaminants	Purge vial with argon before sealing
	Sample carryover	Clean sampling needle Sample vial too full
	Leaching from GC septum	Choose different septum type

Purge and Trap

Baseline perturbation	System contamination	Clean transfer lines
	High water background	Use a different trap Use a water removal device Purge samples for a shorter time
Peak tailing, broad peaks	Desorption flow too slow	Increase purge flow Decrease GC flow to inlet
	Slow desorption	Reduce amount or type of adsorbent Use a different adsorbent Increase ramp rate
	System voids	Check connections Reduce inlet liner volume Reduce volume of connecting tubing
	Interferences from water	Use a different trap Use a water removal device Purge samples for a shorter time
	Transfer line temperature low	Increase line temperature
Peak areas too small	Sampling time too short	Increase purge time
	Adsorbent not working	Replace adsorbent tube
	Leaking connections	Inspect and reseal connections
Sample contamination	Sample exposed too long before sealing	Seal vail immediately Minimize transfer times
	Sample carryover	Clean sampling lines Replace trap

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