





TROUBLESHOOTING TOOLS

- Flow meter
- New syringe
- Methane or other non-retained compound
- New septa, ferrules and injector liners

- Leak detector
- Column performance test sample
- Reference column
- Instrument manuals

PRE-INSTALLATION CHECKLIST

- Replace oxygen, moisture and hydrocarbon traps as necessary.
- Check gas cylinder pressures to ensure that an adequate supply of carrier, make-up and fuel gases are available. Carrier gases should be of the highest purity.
 Note: It is critical that oxygen and water be removed from the carrier gas by the appropriate use of filters and adsorbents.
- Ensure that the injection port is clean and free of sample residues, septum, or capillary debris.
- Check and replace as necessary critical injector components such as seals, liners, and septa.
- Check and replace detector seals as necessary.
- Carefully inspect your column for damage or breakage.

CRITICAL COLUMN INSTALLATION STEPS

INJECTOR INSTALLATION

Note: GC columns do not have a specific directional flow when received from the manufacturer. Upon initial use of your new Zebron[™] column, Phenomenex recommends the practice of dedicating one specific end of the column for injector installation only. This is particularly important when dealing with active/caustic or contaminating compounds. If these compounds are routinely injected onto the column, degradation of the phase will occur—leading to higher bleed. A typical first step to remedying (removing) this bleed would be to trim 10 cm from the front (injector) end of the column and keep trimming this inlet end of the column as necessary. Trying to remedy any bleed issues by trimming the column may not work if both ends have been interchangeably installed into the inlet.

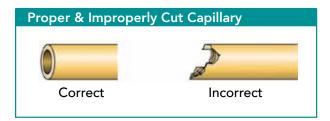
- 1. Place a capillary nut and ferrule on the injector end of the GC column, allowing a section of column to protrude. Trim one to two centimeters from the protruding end to remove ferrule contamination that may have entered the column. Inspect the cut with a magnifier to ensure that a smooth, clean, square-cut edge has been made recut if necessary. See figure on p. 3.
- 2. Carefully hang the column in the GC oven, being cautious not to scratch or damage the polyimide coating on the capillary tubing. Rotate the column to avoid sharp bends of the capillary column and any contact of the column with oven surfaces.
- 3. Insert the column into the injector exactly the correct distance specified in the instrument manual. Tighten the ferrule nut finger-tight then ½ turn with a wrench. If the column can still be moved, tighten another ¼ turn until the column is secure.
- 4. Adjust the carrier gas to obtain the flow rate listed on the test chromatogram.

CRITICAL COLUMN INSTALLATION STEPS (cont'd)

DETECTOR INSTALLATION

Note: For users with sensitive detectors such as MS and ECD, column conditioning steps should be performed before installing the column to prevent contamination and frequent maintenance of the detector.

- 1. Place the column nut and ferrule past the end of the column and cut a centimeter or two off the end of the column. Be sure that the ferrule is the right size and pointing in the correct direction. Inspect the cut with a magnifier and ensure that the cut is square and smooth. See figure below. Recut if needed.
- Insert the outlet end of the column into the detector exactly the distance prescribed in the instrument manual. Distances will vary between detectors. Tighten the ferrule nut finger-tight then ½ turn with a wrench. If the column can still be moved, tighten another ¼ turn until the column is secure.
- 3. Inspect the column connections for leaks using an electronic leak detector. Leaks at the inlet end may introduce oxygen to the column that will result in increased column bleed and damage to the column phase.



COLUMN CONDITIONING

 Allow sufficient time for the carrier gas to flow through the column to purge any oxygen that may be in the system. Raise the temperature of the column to the maximum isothermal operating temperature that is listed on the individual Zebron[™] GC Column Test Report. Maintain this temperature until a constant baseline is achieved. Conditioning times will depend on the phase identity and thickness, with thicker films taking longer to stabilize. In order to minimize the downtime of the instrument, columns can be conditioned overnight at the maximum isothermal temperature.

INSTALLATION TESTING

- 1. Inject a detectable unretained sample, such as methane for an FID, to determine dead volume time and linear gas velocity at the desired column temperature. Adjust gas pressure for optimal flow depending on carrier gas selection.
- 2. The non-retained peak must have ideal peak shape or installation is faulty and needs to be redone.

CHECKING FOR LEAKS

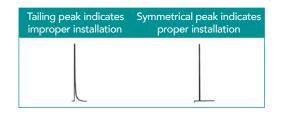
Use a thermoconductivity detector to check for leaks. It is highly sensitive to H_2 , He, and N_2 and will not contaminate the instrument or column. Liquid leak indicators are not recommended for capillary columns. There is the risk of drawing the liquid into the column or fittings and contaminating the system.

Note: If Vespel[®] ferrules are being used, leakage can occur after the initial heating phase due to ferrule deformation. Be sure that the fitting is re-tightened after this initial heating phase, then carefully check all corrections for leaks.

UNRETAINED VOLUME PEAK SHAPE TESTING

If the peak is broad and/or tailing, check the following:

- Improper column positioning/insertion into inlet
 or detector
- Gross contamination of the splitter sleeve
- Chipped or cracked splitter sleeve
- Improper sweeping of sample at column end by makeup gas
- Damaged or crushed column end



NON-RETAINED PEAK TIMES AND MARKERS

Methane with FID/TCD:

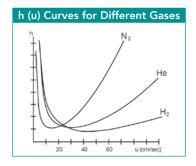
Calculate linear velocity by injecting 25-100 μ L of 1% methane in N₂ gas blend. Measure the retention time of the methane peak and calculate the following: **Linear Velocity (u) = L/t**_a

Detector Type	Marker Compound	
ECD	Methylene chloride ^{2, 3} , Dichlorodifluoromethane	
FID	Methane, Butane ¹	
NPD	Acetonitrile ^{2, 4}	
PID ELCD	Vinyl chloride	
TCD, MS	Methane, Butane ¹ , air	
 From a disposable ligh Place 1-2 drops in an a the headspace of the v Use a column tempera 	utosampler vial and tightly cap. Shake and inject 1-2 µL from rial. Do not inject any liquid.	

4. Use a column temperature above 95 °C.

RECOMMENDED NON-RETAINED RETENTION TIMES

Length (m)	H ₂ (sec)	He (sec)	N ₂ (sec)
15	38	75	150
30	75	150	300
60	150	300	600



COLUMN DIMENSION SELECTION GUIDE

	Length (L)	ID (r)	Phase Thickness (d _f)
RESOLUTION (R)	$R \sim \sqrt{L}$ To double resolution, quadruple the length	$R \sim \frac{1}{r}$ Resolution decreases with increased diameter	$R \sim \frac{1}{\sqrt{d_f}}$ Resolution decreases with increased thickness
SAMPLE CAPACITY (SC)	$SC \sim \sqrt{L}$ Longer columns have slightly better capacity	SC ~ r ² Capacity is exponential as diameter increases	SC ~ d _f Sample capacity increases with thicker films
RETENTION TIME (t _r)	t _r ∼ L Longer columns require longer analysis times	t _r ∼ r Smaller diameter columns allow faster analysis times	$t_r \sim d_f$ Thicker films require longer analysis times



COLUMN PHASE SELECTION GUIDE

Pola	rity Scale	Zebron [™] Phase	Highlight	
<u> </u>	5	ZB-1	Non-polar phase suited for true boiling point separations	
ola	5	ZB-1ms	Extremely low bleed column for non-polar compounds	
ے ا	5	ZB-1HT Inferno™	High temperature stability (430 °C) for non-polar compounds	
Non-Polar	5	ZB-1XT SimDist	Metal column with increased accuracy for simulated distillation analysis, high temperature stability (450 °C)	
	8	ZB-SemiVolatiles	Specifically designed and tested for supreme inertness to active acids and amines; our column of choice for SVOCs, PBDEs, and environmental PAHs	
	8	ZB-5	Versatile low polarity column for general lab use	
-	8	ZB-5ms	Arylene phase for enhanced resolution of PAHs and multi-ring aromatic compounds	
	8	ZB-5MSi	Very inert for good peak shape of active compounds, such as acids and bases	
	8	ZB-5MS <i>PLUS</i> ™	The next generation of inertness for food testing, specialty chemical, and toxicology analyses	
	8	ZB-5HT Inferno	High temperature stability (430 °C) to separate high molecular weight compounds and eliminate carry overs	
9 ZB-XLB		ZB-XLB	Low polarity si-arylene column with bleed and sensitivity levels designed for MS detectors	
-	9	ZB-XLB-HT Inferno	High temperature analysis of mid-polar compounds	
	13	ZB-624	Optimized for separating volatile organic compounds (VOCs)	
	18	ZB-35	Intermediate polarity column for high molecular weight analysis	
	18	ZB-35HT Inferno	High temperature analysis of mid-polar compounds	
	19	ZB-1701	Optimized phase providing an alternate selectivity to phenyl phases with similar polarity	
	19	ZB-1701P	Specially tested to ensure good response for DDT and endrin	
	24	ZB-50	High polarity column capable of high - temperature bake-out to remove contaminants	
	52	ZB-WAX <i>PLUS</i> ™	100 % aqueous stable with high retention of alcohols and other chlorinated solvents	
Polar	57	ZB-WAX	Bonded, solvent rinsable phase excellent for separating polar complex mixtures	
ď	58	ZB-FFAP	Provides better peak shape for underivatized acids	
é		ZB-CLPesticides-1 & -2	Run multiple chlorinated pesticide classes by GC/ECD on one column set	
Exclusive		ZB-MultiResidue [™] -1 & -2	Novel phase designed for pesticides, herbicides, and insecticides for all detection methods	
:clr		ZB-Bioethanol	Fast analysis of bioethanol fuel	
ш		ZB-Drug-1	Rapid and improved separations for drugs of abuse	
		ZB-BAC-1 & -2	More accurate blood alcohol analysis, especially for post-mortem samples	
	* 6			

* See individual phases for detailed specifications and limitations.

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Composition	Polarity	Temperature Range* (Isothermal/TPGC)	MS Certified
100 % Dimethylpolysiloxane	Nonpolar	-60 to 360/370 °C	1
100 % Dimethylpolysiloxane	Nonpolar	-60 to 360/370 °C	1
100 % Dimethylpolysiloxane	Nonpolar	-60 to 400/430 °C	1
100 % Dimethylpolysiloxane	Nonpolar	-60 to 450 °C	\checkmark
5 % Phenyl-Arylene 95 % Dimethylpolysiloxane	Nonpolar	-60 to 325/350 °C	1
5 % Phenyl 95 % Dimethylpolysiloxane	Nonpolar	-60 to 360/370 °C	\checkmark
5 % Phenyl-Arylene 95 % Dimethylpolysiloxane	Nonpolar	-60 to 325/350 °C	1
5 % Phenyl 95 % Dimethylpolysiloxane	Nonpolar	-60 to 360/370 °C	1
5 % Phenyl-Arylene 95 % Dimethylpolysiloxane	Nonpolar	-60 to 325/350w °C	1
5 % Phenyl-Arylene 95 % Dimethylpolysiloxane	Nonpolar	-60 to 325/350 °C	
Proprietary	Intermediate	30 to 340/360 °C	1
Proprietary	Intermediate	30 to 400 °C	1
6 % Cyanopropylphenyl 94 % Dimethylpolysiloxane	Intermediate	-20 to 260 °C	
35 % Phenyl 65 % Dimethylpolysiloxane	Intermediate	40 to 340/360 °C	✓
35 % Phenyl 65 % Dimethylpolysiloxane	Intermediate	40 to 400 °C	\checkmark
14 % Cyanopropylphenyl 86 % Dimethylpolysiloxane	Polar	-20 to 280/300 °C	
14 % Cyanopropylphenyl 86 % Dimethylpolysiloxane	Polar	-20 to 280/300 °C	
50 % Phenyl 50 % Dimethylpolysiloxane	Polar	40 to 320/340 °C	\checkmark
Polyethylene Glycol	Polar	20 to 250/260 °C	
Polyethylene Glycol	Polar	40 to 250/260 °C	1
Nitroterephthalic Acid Modified Polyethylene Glycol	Polar	40 to 250/260 °C	
Proprietary	Intermediate	40 to 320/340 °C	
Proprietary	Intermediate	-60 to 320/340 °C	1
Proprietary		-60 to 340/360 °C	1
Proprietary		40 to 320/340 °C	1
Proprietary		-20 to 260/280 °C	1

* See individual phases for detailed specifications and limitations.

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