General Service Bulletin

This General Service Bulletin is intended for reference only and not as a complete instruction manual. GOW-MAC recommends that all our customers take advantage of our 89 years experience and employ the resources of our Repair Department.

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I INTRODUCTION

GOW-MAC® offers a variety of detector filaments for thermal conductivity detectors. The purpose of this brochure is to outline the methods used for the field replacement of these elements.

Thermal conductivity detectors (TCDs) are used in on-stream applications as well as in gas chromatographs. The TCDs differ in internal volume, geometry, and speed of response. The choice of filaments is based on the application and corrosive properties of the gaseous stream. The characteristics of the GOW-MAC filaments are described in Bulletin SB-13. Since gas chromatographs may also use thermistors rather than hot wires, thermistor installation procedures are also covered in this bulletin.

GOW-MAC filaments are mounted on either two or three pin glass to metal seals. The 9225 and 333 mounts are gold plated with glass insulation. GOW-MAC also offers (a) hermetic seal, and (b) axial filament (730 mount shown in Fig. 2) as used in its model 10-952 TCD. TCDs with hermetic seal and axial filaments must be returned to GOW-MAC for filament replacement. Filaments on single-header mounts (Fig. 2 except the 730 mount) can be replaced in the field by following the instructions in the bulletin. Filaments in the majority of TCDs are secured with tube nuts which screw into the cell and make a mechanical seal between the flange on the filament holder and the TCD body (the concentric ridge shown in Fig. 1).

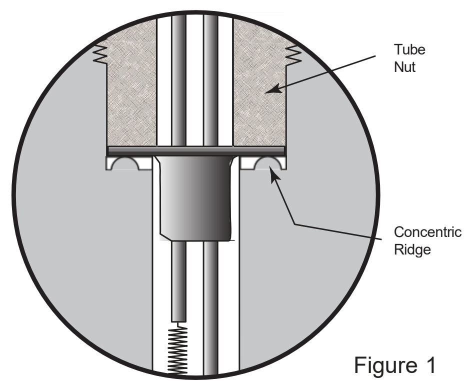


Figure 1

Filaments are sold as matched pairs and in sets of four matched filaments called "quads." Most detectors require two pairs, or four filaments. In order to make a well balanced cell, all four filaments should be closely matched under test conditions simulating installation in a TCD. Installation of a GOW-MAC quad yields an electrically superior TCD because the initial balance will be closer to a true electrical zero. Our replacement filaments come with insulated lead extension wires attached. A quad is shipped in a protective shipping block.

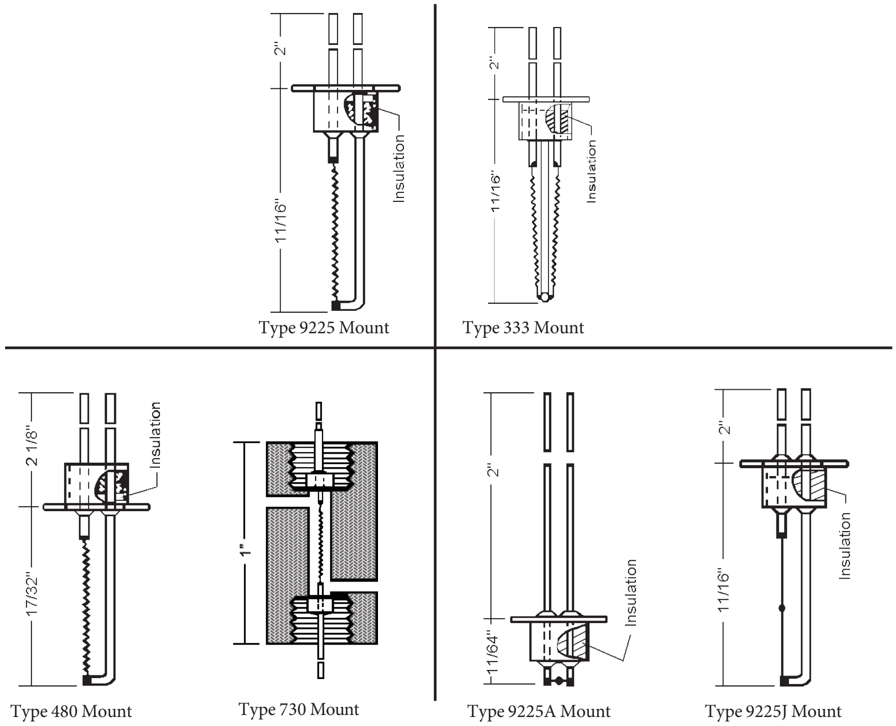
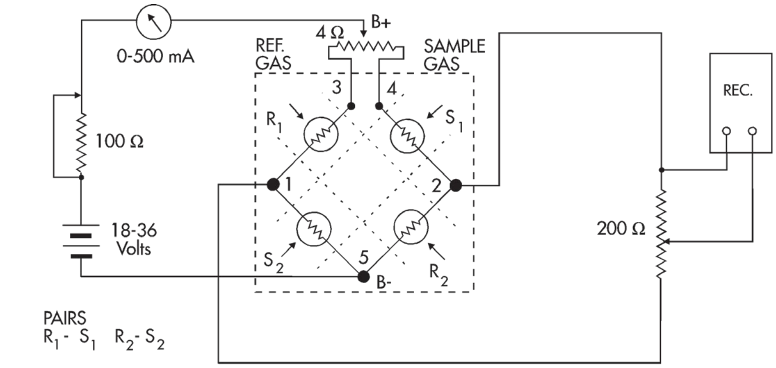


Figure 2

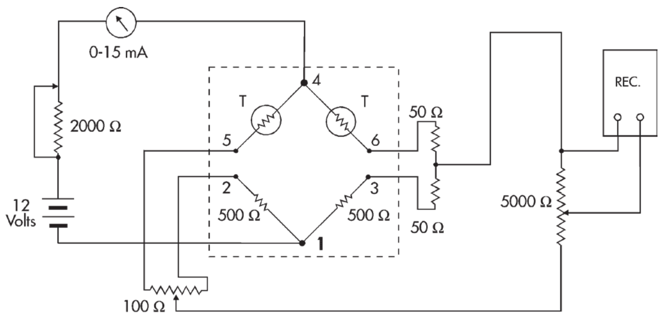
It should be noted that the terms "Sample" and "Reference" are used throughout this bulletin. Since most GOW-MAC TCDs are dual pass, these terms are used to differentiate side A and side B. In gas chromatography, they would refer to the carrier gas from column A and column B, respectively.

The electrical circuits used by various manufacturers vary but most incorporate the Wheatstone Bridge. Normally four filaments are used however some manufacturers use two-filament bridges and fixed resistors. This is more common when thermistors are the sensors. Figs. 3, 4 and 5 illustrate typical bridges with the zero adjustment between two elements in the conventional style. Fig. 6 shows the more modern approach, but note that it is still a "bridge" circuit.



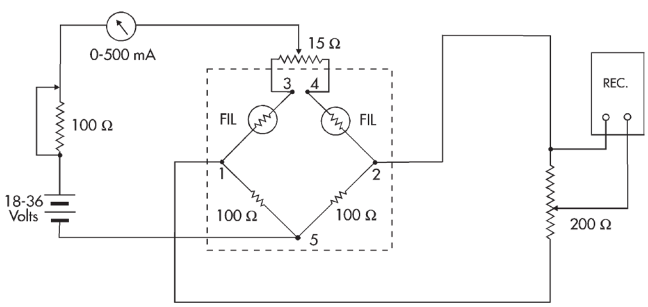
Circuitry For 4-filament Cells

Figure 3



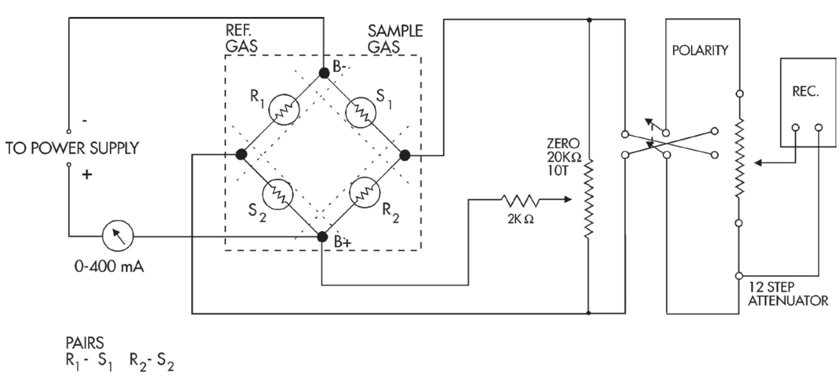
Circuitry For Thermistor Cells

Figure 4



Circuitry For 2 Filament Cells

Figure 5



Circuitry For 4-filament Flow-Through Cells

Figure 6

II REPAIR BY GOW-MAC

A. TCDs of most major manufacturers may be returned to GOW-MAC for installation of new filaments and thermistors. We will clean, repair and re-filament your thermal conductivity detector to the specifications of the original manufacturer. GOW-MAC service includes:

1. Cleaning the inside of the cell block.

2. Replacement of all filaments.

3. Replacement of electrical leads with new lead connections and high-temperature fiberglass insulation.

4. Complete pneumatic and electrical leak-testing to the original new performance specifications (See IV. A. 4).

B. GOW-MAC also offers new replacement TCDs for most of the major gas chromatographs on the market today, including many discontinued and obsolete models.

III FIELD REPAIR

A. The orientation of the filaments in the detector cell is most important. The gas passages in most detectors are usually referred to as sample and reference. In some cell designs, the sample and reference gases enter on one side, pass directly through the cell, and exit on the opposite side. In other detectors, the sample gas enters and leaves on the same side with the reference gas entering and leaving the other side. It is important to establish the gas flow passages as related to the element cavities for proper installation of the filaments or thermistors. This is easily done by noting the column connections before removal of the cell from the instrument.

B. Filaments must always be replaced in pairs. It is impractical to attempt to replace single filaments. Consult GOW-MAC Sales for recommendations for the proper filaments to use if in doubt.

C. The tools required are:

Vise
1/2" box wrench (some cells require use of an open-end wrench)
Torque wrench with 1/2 inch crowfoot attachment, capable of 15 ft-lbs

Since most TCDs used in gas chromatographs operate at elevated temperatures, soft solder in the TCD oven cannot be used. For this reason GOW-MAC filaments are supplied with extended leads welded at the factory.

D. Four Filament Cells

1. Disconnect instrument from AC power source.

2. Make a record of connections from each filament before removal from the instrument for reference when re-assembling. Disconnect the electrical connections to the filaments.

3. Remove the detector block from the instrument according to the instructions found in your instrument's operating instruction manual.

4. Support the detector in a vise. Back off tube nuts. Usually these can be reused however new nuts are supplied with quads. Also, they are available from GOW-MAC; part number 176-110. For TCDs that use nuts with a different thread, call GOW-MAC Sales for assistance.

5. Clean cavities of cell block, tubing and drilled sections with fresh acetone. If a subsequent water rinse is used, ensure all water is removed and cell is dry before installing filaments. This is most easily accomplished by baking the detector block at 150 °C for six (6) hours in an oven with a purging N_2 flow. All internal surfaces should be clean for optimum heat dissipation. Inspect cavities for deposits and particles. Inspect filament header seats (the concentric ridge in Fig. 1) for scratches across the seat that might prevent a gas-tight seal.

WARNING
ACETONE IS EXTREMELY FLAMMABLE - EXERCISE CARE WHEN USING. DO NOT EXPOSE ACETONE TO OPEN FLAMES OR SMOKING MATERIALS. DISPOSE OF WASTE PROPERLY.

6. Quads are shipped with tube nuts for use when replacing the new filaments. Loosen the tube nuts and remove the filaments (one at a time) from the quad shipping block, observing the SAMPLE and REFERENCE positions. Place the filaments in the proper detector cavity (REFERENCE AND SAMPLE according to the detector's flow configuration, Fig. 7) making sure to keep the filament helix away from gas flow inlet (place the header pin between the gas inlet and the filament).

NOTE
THE QUAD IS NOT A DETECTOR. IT IS USED ONLY AS A CONTAINER TO PROTECT THE FILAMENTS FROM DAMAGE DURING SHIPPING.

WHEN INSTALLING FILAMENTS, INSERT THEM IN THE SAMPLE AND REFERENCE SIDES OF THE DETECTOR CORRESPONDING TO THE LABELS "SAMPLE" AND "REFERENCE" ON THE QUAD SHIPPING BLOCK. POSITION FILAMENT COILS AWAY FROM GAS FLOW INLETS.

7. Tighten tube nuts to 12 ft-lbs.

CAUTION
EXCESSIVE TIGHTENING OF THE TUBE NUTS MAY CRACK THE GLASS INSULATORS. ALWAYS USE GOW-MAC TUBE NUTS AND DO NOT EXCEED THE SPECIFIED TORQUE.

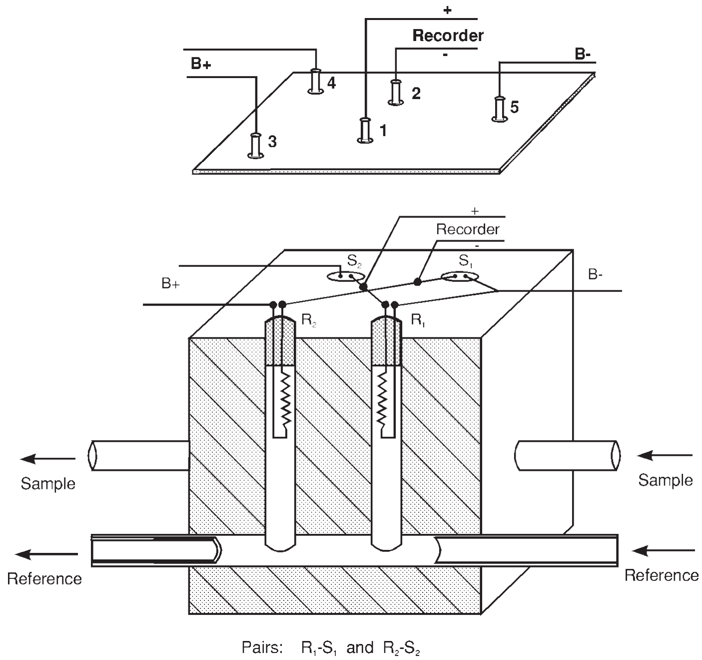


Figure 7

8. A preliminary electrical check of the detector should be made with a multimeter: (a) Measure resistance between pairs of filament leads. The measured values should be approximately the filament nominal resistance. (b) Check for shorts between filament leads (measured one at a time) and the detector body. The measured values should be infinite resistance.

9. If the detector is removed from the instrument, insulate it from the influence of ambient temperature.

10. Purge detector with carrier gas, nitrogen or air, for 5-10 minutes before turning on power.

11. Use current value from Table 1 dependent on gas used, and allow warm-up time of 15-20 minutes.

    At this time the Zero control on the instrument should be set at the center position of travel. A well balanced cell will permit maximum use of the Zero control in either direction.

12. Tighten tube nuts with torque wrench to 12 ft-lbs and leak test pneumatically at two atmospheres.

CAUTION

RELEASE PRESSURE GRADUALLY. IN FLOW-THROUGH CELLS ESPECIALLY, FILAMENTS CAN BE DESTROYED BY A RAPID CHANGE IN GAS FLOW.

13. Verify initial performance by repeating the test in steps 8-11 above.

F. TWO FILAMENT CELLS

1. Disconnect instrument from AC power source.

2. Make a record of connections from each filament before removal from the instrument for reference when re-assembling. Disconnect the electrical connections to the filaments.

3. Remove the detector block from the instrument according to the instructions found in your instrument's operating instruction manual.

4. Support the detector in a vise. Back off tube nuts. Usually these can be reused but if not, they are available from GOW-MAC; part number 176-110. For TCDs that use nuts with a different thread, call GOW-MAC Sales for assistance.

5. Clean cavities of detector block, tubing and drilled sections with fresh acetone. If a subsequent water rinse is used, ensure all water is removed and cell is dry before installing filaments. This is most easily accomplished by baking the detector block at 150 °C for six (6) hours in an oven with a purging N_2 flow. All internal surfaces should be clean for optimum heat dissipation. Inspect cavities for deposits and particles. Inspect filament header seats (the concentric ridge in Fig. 1) for scratches across the seat that might prevent a gas-tight seal.

WARNING
ACETONE IS EXTREMELY FLAMMABLE - EXERCISE CARE WHEN USING. DO NOT EXPOSE ACETONE TO OPEN FLAMES OR SMOKING MATERIALS. DISPOSE OF WASTE PROPERLY.

6. Feed the leads of replacement filaments or thermistors through the tube nuts. Note position of support assemblies (refer to Figure 2 for mount types). Type 333 W2 long support wire faces inlet. Type 9225A (thermistor) is at right angles to axis of inlet.

7. Tighten tube nuts to 12 ft-lbs.

CAUTION
EXCESSIVE TIGHTENING OF THE TUBE NUTS MAY CRACK THE GLASS INSULATORS. ALWAYS USE GOW-MAC TUBE NUTS AND DO NOT EXCEED THE SPECIFIED TORQUE.

IV CHANGING REFERENCE/CARRIER GAS

A. GOW-MAC thermal conductivity detectors for gas chromatography are tested under the following conditions:

   1. Purge at atmospheric pressure with helium at 30 mL/min (5 mL/min for micro cell).

   2. Thermally insulated TCD self-heating to steady state temperature.

3. Cell current: 250 mA in helium for all filament types except 100 mA for WX7.

4. Circuit in accordance with Figs. 3, 4, 5 or 6 should result in:

   a. Nominally balanced bridge - TCD can be zeroed.
   b. Noise level: < 10 µV peak to peak over 1 minute.
   c. Drift: < 10 µV peak to peak over 30 minutes.

5. Most TCDs are tested on helium. Changing carriers requires a corresponding change in current for best performance and to preserve the filaments. Refer to Table 1 for starting bridge currents and to Fig. 8 for maximum bridge currents given carrier gas and detector operating temperature. The following steps should be taken after leak testing all newly made gas connections including columns:

   a. Introduce new carrier gas into system at the recommended flow rate. Allow time for a complete purge.

   b. Set initial bridge current as follows:

Table 1. Recommended Starting Current Settings
Milliamperes at Ambient Temperature

Gas	Hot Wire	8K or 9K Thermistors
Argon	80 mA	6 mA
Helium	100 mA	8 mA
Nitrogen	80 mA	6 mA

   c. Turn on recorder or data reduction software.

   d. Adjust zero control until recorder reads ZERO.

   e. The power required by the W2 and W2X filaments produces increased TCD self heating and requires a longer warm-up time than needed with W/WX filaments.

A minimum operating temperature of the oven will be about 70-80 °C unless forced air ventilation permits heat dissipation.

Figure 8

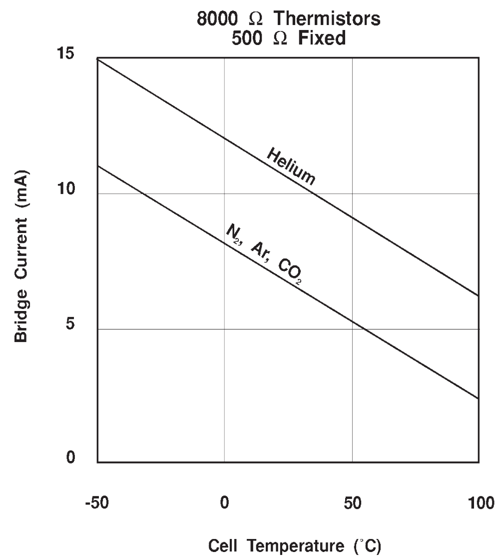
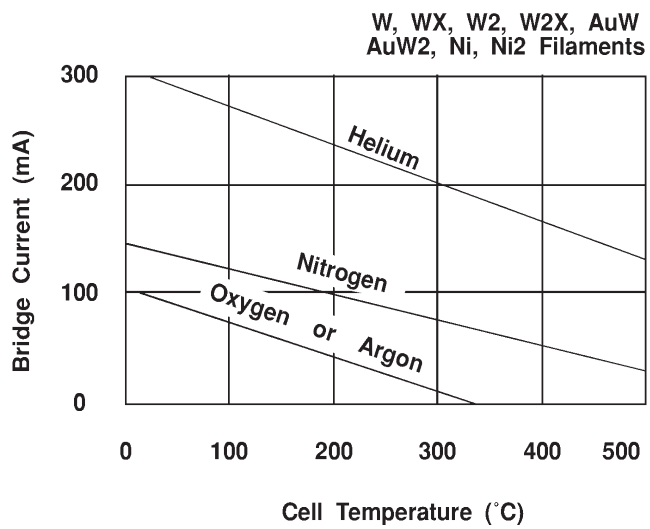


Figure 9



V MISCELLANEOUS NOTES

1. Filament life may be extended by operating at low current and low cell temperatures. The cell temperature should only be as high as needed for samples used, and current should be as low as possible consistent with sensitivity required. It is better to operate the system with lower attenuation and low bridge currents than at higher currents with higher attenuation.

2. Sensitivity increases 4 to 8 times as filament current increases by a factor of 2. However, increasing filament current excessively results in baseline instability and possible filament burnout. Care must be observed in arbitrarily changing bridge current.

3. Filaments may go out of balance over time. This occurs for several reasons. The most common is a leak in the system. Small leaks can develop at fittings. Even though the system

is under pressure, air can aspirate into the system and oxidize filaments. When this happens, one side of the bridge resistance changes and goes out of balance. This is indicated by a long-term drift in one direction. A dynamic leak check will determine if leaks are present. If a leak is proven, then the operator must seek them out. This may be difficult since the leak may develop when the instrument is hot. The GOW-MAC® Model 21-080 Gas Leak Detector was designed specifically for this type of application.

Unbalance may also occur because of samples which oxidize or corrode one side of the detector. This is demonstrated by a peak not returning to zero and establishing a new baseline above the old. Another injection will again require zero adjustment. Correction of this phenomenon is accomplished by either lowering the bridge current, detector temperature or changing to a more corrosive resistant detector element.

4. A word about SERVICE: GOW-MAC detectors are made from materials of the best grade with simplicity of design, and are thoroughly tested before shipment. In these days of service contracts, guarantees, warranties and service networks the best guarantee really is the reputation of the manufacturer.

5. If you encounter problems or have questions concerning TCDs or repairs, call GOW-MAC Sales at (610) 954-9000.

VI TROUBLE SHOOTING GUIDE

TROUBLE	PROBABLE CAUSE	CHECKS AND/OR REMEDY
1. No signal	a) Detector or power switch off. switch off.	Make sure carrier is flowing and turned on. Set bridge current to desired setting.
	b) Recorder improperly connected.	Connect recorder.
	c) Detector filaments burned out.	Replace elements. (See manual).
	d) Open circuit on detector cell.	Check for broken lead wires, loose terminal screws and broken filament(s).
2. Low cell current	a) Power Supply voltage inadequate (likely to occur with change from lower resistance element to higher resistance element).	Increase power supply capacity.
3. Recorder can't be zeroed.	a) Excessive bridge current.	Reduce bridge current (refer to operating charts).
	b) Detector contaminated.	Clean cavities and elements. Replace elements if required.
	c) Loose or corroded electrical connections.	Check connections.
	d) Detector elements oxidized bridge out of balance.	Replace all four elements.
4. Drift.	a) Change in flow sample and/or reference gas pressure.	Check flow gauges. Creeping regulator or reducing valve.
	b) Warm-up period too short.	Allow sufficient purge and temperature equilibration.
	c) Changes in ambient temperature.	Protect instrument from drafts, direct sunlight, or nearby sources of hot or cool air.
	d) Carrier gas flow leaks.	Tighten all fittings so they are leak-free.
	e) Detector contaminated.	Clean detector.
	f) Contamination in column.	Recondition column.
	g) Filament ageing.	As a temporary measure, switch sample and reference passes of the detector. Eventually replace filaments.
	h) Decompression chill by the reference gas.	Install heater or buffer volume.
	i) Cell mass (micro cells) is insufficient.	Increase heat sink mass. Heat insulate dummy resistors (in 2 element bridge).

TROUBLE	PROBABLE CAUSE	CHECKS AND/OR REMEDY
5. Short filament life.	a) Improper start-up/shutdown procedure.	First turn on gas. Wait, turn on filament current. Turn off current first - gas off last.
	b) Corrosive samples.	Check other filament materials which might have higher corrosive resistance.
6. Cycles in zero trace.	a) Oven thermostat not functioning properly.	Install unit of proper rating. Check location. (Should be near heat source).
	b) Oven heater wattage excessive.	Reduce wattage.
	c) Oven fan or circulating pump failure.	Replace.
	d) Oven insulation is inadequate.	Change material and/or cabinet design.
7. Noise in signal trace (pneumatic sources).	a) Loose or worn column connections.	Install new unions or ferrules.
	b) Septum leaks.	Replace with new septum.
	c) Leaks in sample line.	Pressure check to 2 atmospheres; components, all connections, septum and gas sampling device.
	d) Leaks in reference line.	Check: tubing, pressure regulators, and gas cylinder connections.
	e) Leaks in T/C cell.	Remove cell from instrument and check for loose tube nuts, bad seats, or a cracked header. Replace parts as required.
	f) Impurities in reference gas.	Install purifier or new cylinder.
	g) Back diffusion into	Install tail pipe (24" min.) cell.
	h) Ambient pressure changes.	Air conditioned labs may require venting to doors.
	i) Tubing, etc., not inert.	Replace.
	j) GC column contaminated.	Back flush, Remove and bake.
1. Noise in signal trace (water vapor sources).	a) Water vapor in reference or sample gas.	Install drier. (Especially important for H_2 ref. gas).
	b) Condensate in gas flow system.	Avoid traps and valleys in tubing runs. All horizontal runs should slope 5°/min.

TROUBLE	PROBABLE CAUSE	CHECKS AND/OR REMEDY
9. Noise, blips, or hash in signal trace (electrical sources).	a) Recorder defective.	Service recorder. Install voltage regulator.
	b) Line voltage variations.	Install voltage regulator.
	c) Line frequency variations.	Check the frequency.
	d) Non-shielded recorder leads.	Replace.
	e) Ungrounded recorder.	Provide common ground for all electrical components.
	f) Vibration and shock.	Cushion apparatus.
	g) Intermittent electrical connections.	Connections not clean or mechanically secure. (Clean and resolder). Replace any plug and socket connections.
10. Loss of signal sensitivity. (Reduced signal from known sample).	a) Bridge current incorrect	Check milliammeter against standard.
	b) GC output range attenuator changed.	Verify output range.
	c) Detector contaminated.	Clean detector cavities and detector elements. Replace elements if necessary.
	d) Leaks in gas train. Low filament current. Low temperature of cell block. Poor recorder connections and incorrect range setting.	Check.
	e) Carrier gas impure.	Install purifier or replace cylinder.
	f) Column impaired.	Replace.
	g) Detector wired incorrectly.	Refer to Figs. 3, 4, 5 or 6.
11. Loss of signal sensitivity. (Increases response time or lower peaks from known sample).	a) Detector contaminated.	Clean detector cavities and detector elements. Replace elements if necessary.
	b) Improper rate of flow and/or pressure of sample gas.	Check.
12. Signal peaks inverted.	a) Detector is incorrectly wired.	Check, and refer to Figs. 3, 4, 5 or 6.

TROUBLE	PROBABLE CAUSE	CHECKS AND/OR REMEDY
13. Zero adjust is over sensitive.	a) Detector filaments too hot.	Reduce bridge current.

VII RELATIVE THERMAL CONDUCTIVITY

A. ORGANIC GASES

	(RELATIVE TO AIR = 1.000)		
	80 °F 26.7 °C	100 °F 37.8 °C	200 °F 93.3 °C
Air	1.000	1.000	1.000
Acetone	0.438		0.555
Acetonitrile			0.477
Acetylene	0.815		0.946
Amylamine	0.497		
Benzene	0.398		0.525
i-Butane	0.624		0.766
n-Butane	0.611		0.743
i-Butylamine	0.536		
Carbon Tetrachloride	0.257		0.289
Chloroform	0.287		0.322
Cyclohexane	0.450		0.584
Decane		0.480	0.586
Diethylamine	0.543		
Dimethylamine	0.636		
Dipropylamine	0.456		
Ethane	0.834		1.013
Ethyl Acetate	0.411		0.506
Ethyl Alcohol	0.562		0.701
Ethylamine	0.603		
Ethyl Bromide	0.371		
Ethyl Chloride	0.470		0.562
Ethylene	0.780		0.978
Ethyl Ether	0.579		0.700
Ethyl lodide	0.258		
Freon 12	0.371		0.431
Freon 113	0.293		0.370
Heptane	0.459		0.592
Hexane	0.490		0.637
Methane	1.312		1.416
Methyl Acetate	0.450		0.545
Methyl Alcohol	0.547		0.698
Methyl Amine		0.690	
Methyl Bromide	0.272		0.326
Methyl Chloride	0.409		0.502
Methylene Chloride	0.285		0.331
Methyl lodide	0.199		0.236
Nonane		0.491	0.573
Octane		0.536	0.634
Pentane	0.581		0.705
Propane	0.700		0.837
Proplyamine		0.536	
Toluene		0.573	0.669
Triethylamine	0.483		
Trimethylamine	0.589		
Xylene		0.523	0.551

B. INORGANIC GASES

	(RELATIVE TO AIR = 1.000)		
	80 °F 26.7 °C	100 °F 37.8 °C	200 °F 93.3 °C
Air	1.000	1.000	1.000
Ammonia	0.941		1.052
Argon	0.678		0.677
Bromine Vapor		0.187	
Carbon Dioxide	0.636		0.710
Carbon Disulfide	0.305		
Carbon Monoxide	0.964		0.972
Chlorine	.340		0.365
Deuterium	5.379		5.343
Fluorine	1.067		1.107
Helium	5.734		5.497
Hydrogen	6.943		6.778
Hydrogen Bromide	0.331		0.348
Hydrogen Chloride	0.555		0.579
Hydrogen Cyanide	0.477		0.522
Hydrogen Sulfide		0.573	
Krypton		0.361	
Neon	1.886		1.812
Nitric Oxide	.991		0.998
Nitrogen	.994		0.986
Nitrogen Dioxide			2.681
Nitrous Oxide	0.664		0.759
Oxygen	1.023		1.031
Sulphur Dioxide	0.367		0.409
Water Vapor	0.692		0.769
Xenon		0.215	0.218

VIII REFERENCES

A. For further information on thermal conductivity detectors and separations the following books are recommended:

1. Dal Nogare, S., and Juvet, R.S., Jr., “Gas-Liquid Chromatography,” Interscience Pub., New York, 1962.

2. Purnell, H., “Gas Chromatography,” J. Wiley & New York, 1962.

3. David, D. J., “Gas Chromatographic Detectors,” Wiley-Interscience, New York, 1974.

4. Miller, J. M., “Separation Methods in Chemical Analysis,” Wiley-Interscience, New York, 1974.

5. McNair, H. M., and Bonelli, E. J., “Basic Gas Chromatography,” Varian Instrument Co., 1969.

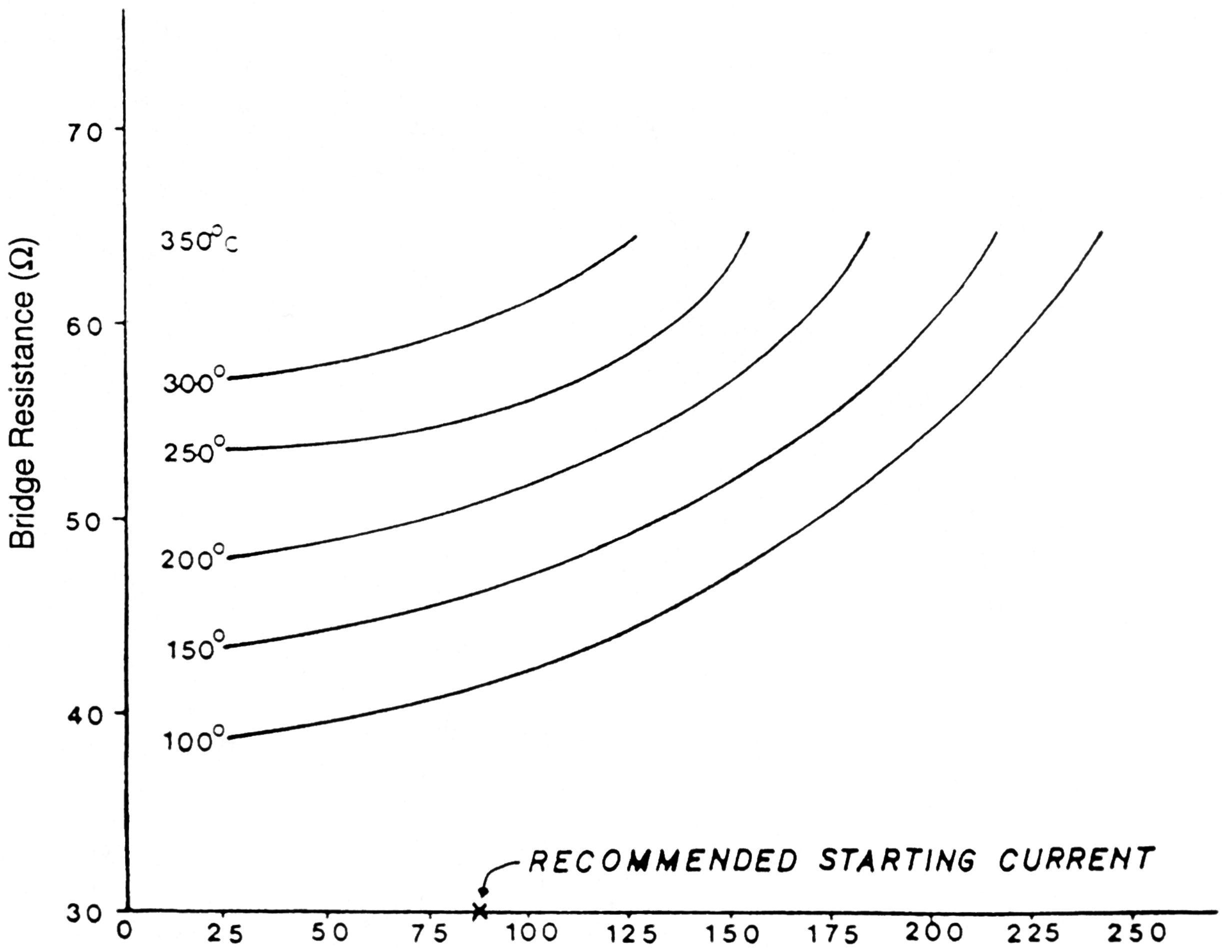
B. The following articles contain extensive bibliographies on thermal conductivity detectors:

1. Lawson, A. E., Jr., and Miller, J. M., “Thermal Conductivity Detectors in Gas Chromatography,” Journal of Gas Chromatography, August, 1966.

2. Lawson, A. E., Jr., “A Versatile, Low Cost Gas Chromatograph,” American Laboratory, May 1969.

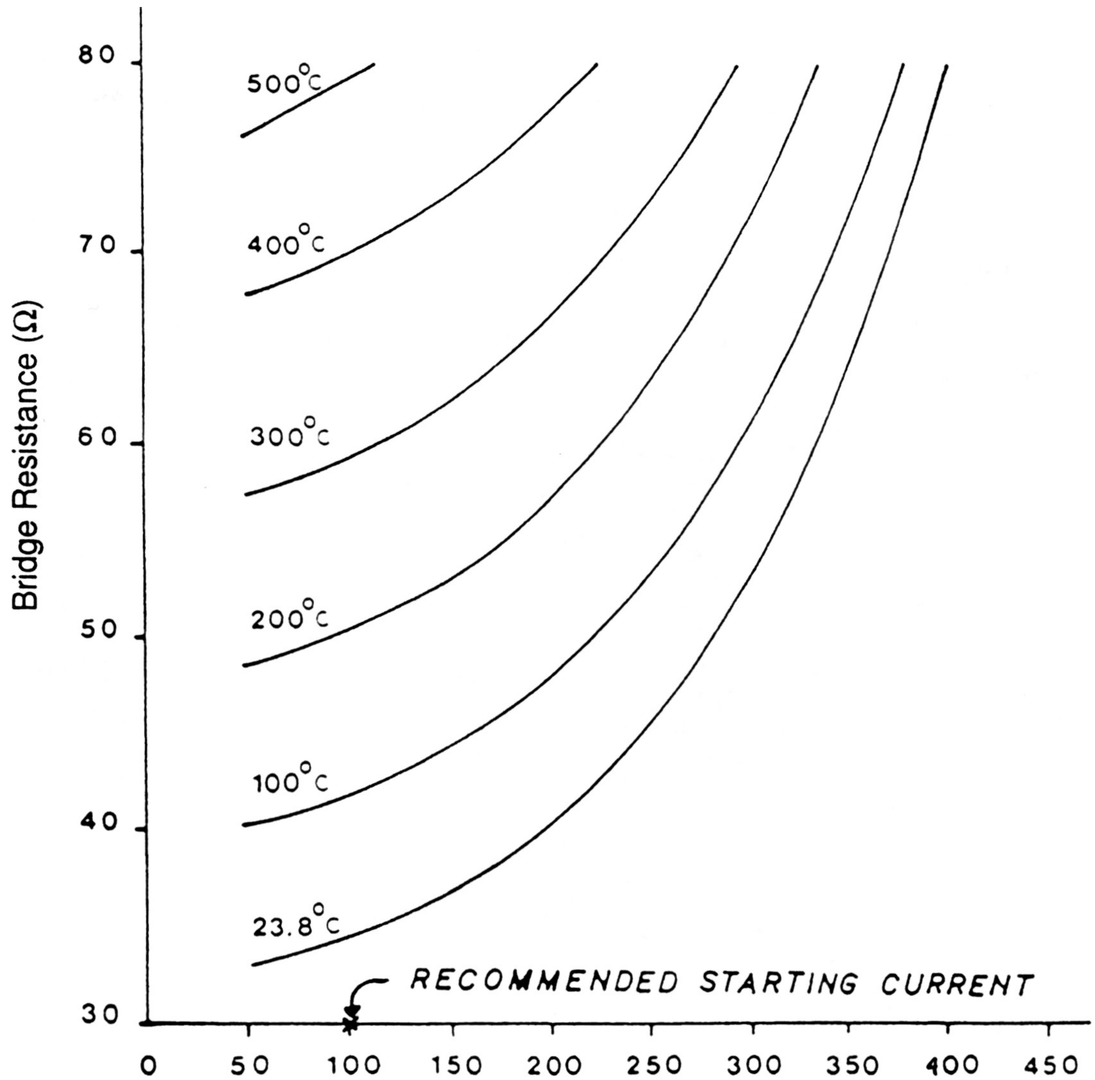
3. Richmond, A. B., “Thermal Conductivity Detectors for Gas Chromatograph: A Literature Survey,” Journal of Chromatographic Science, Vol. 9, February 1971.

Bridge Resistance vs. Current
Helium Atmosphere
WX7 Filaments, 10-955 TCD



Bridge Current (mA)

Bridge Resistance vs. Current
Helium Atmosphere
WX Filaments



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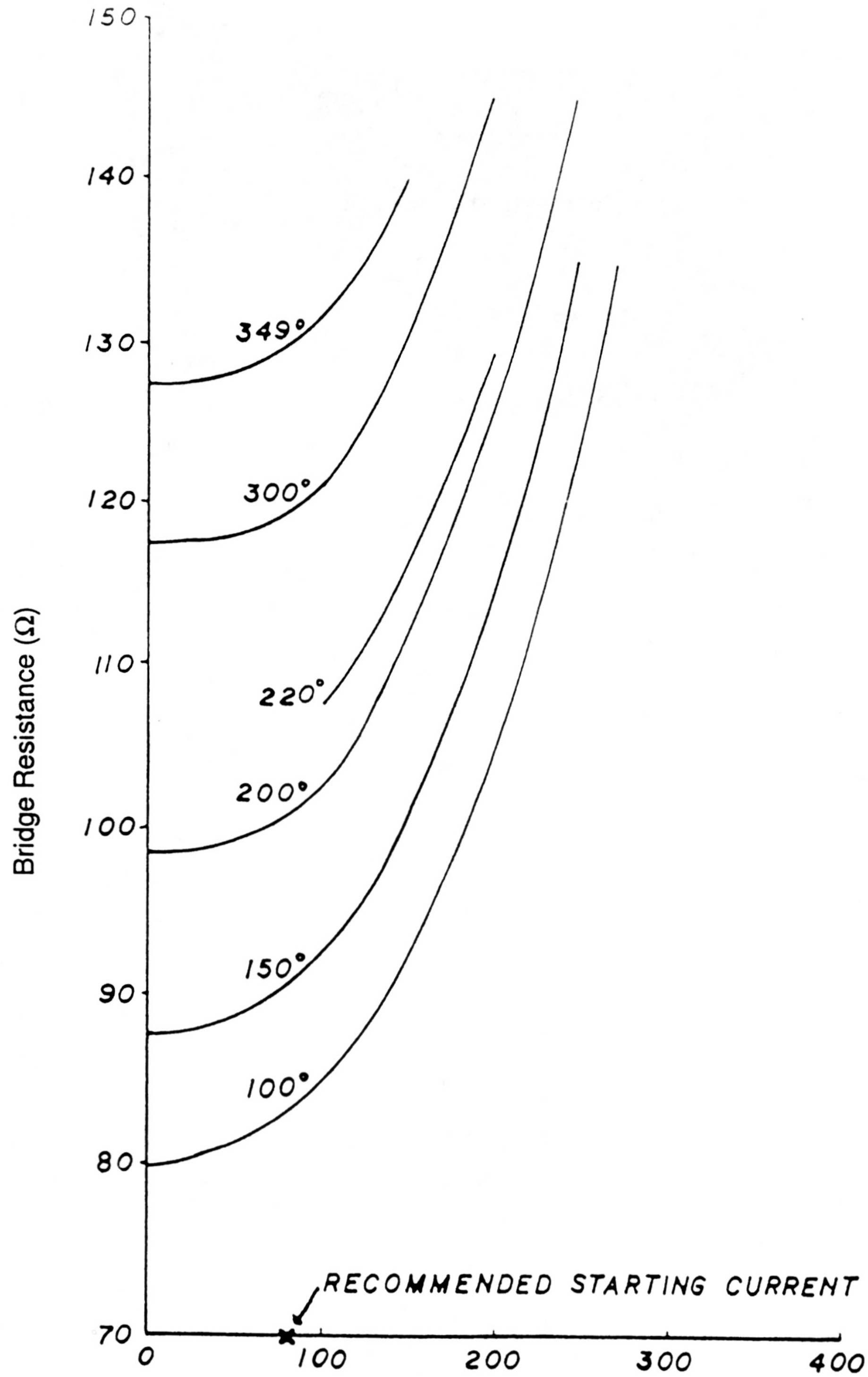

Bridge Current (mA)