# **Operating Manual**

### Series 580 FID Isothermal Gas Chromatograph

Series 580: 120 V, 50/60 Hz Series 582: 230 V, 50/60 Hz

> January 2024 Rev. 10

## READ INSTRUCTIONS BEFORE OPERATING



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## Series 580 Flame Ionization Gas Chromatograph Operation & Maintenance Manual

This manual provides operating instructions and maintenance requirements for the Series 580 FID GC to permit safe and efficient use of your instrument. Throughout this manual, special "NOTE", "CAUTION", "WARNING" signs appear for your protection. It is important that you thoroughly read the appropriate sections of this manual <u>before</u> operating your instrument. Operate the SERIES 580 FID according to these instructions. Any questions concerning the safe and proper use of your instrument should be addressed to:

GOW-MAC Instrument Co. 277 Brodhead Road Bethlehem, PA 18017 Tel: (610) 954-9000 Fax: (610) 954-0599 E-mail: sales@gow-mac.com URL: www.gow-mac.com This section is designed to bring special attention to specific areas or practices that may pose particular hazards to personnel and/or equipment safety only. For complete installation instructions, see Section 3.

It is in the operator's best interest to read this section to ensure the <u>safe</u> operation of the equipment.

#### A. BURN HAZARDS

The injection ports, columns, and column oven cover may reach very high temperatures, and remain hot for several hours after the instrument has been shutdown. To prevent painful burns resulting from contact with the hot surfaces, wear protective gloves.

#### B. ELECTRICAL HAZARDS

- 1. DISCONNECT the instrument from <u>all</u> power sources <u>before</u> removing front, side, and back panels and exposing potentially dangerous voltages.
- 2. Make sure that the actual line voltage is the value for which the instrument was designed. (for properly grounded outlet ONLY.)
- 3. DO NOT overload the ac outlet with other electrical equipment.
- 4. Adhere to the color coding descriptions when hooking up electrical connections.
- 5. Repair or replace faulty or frayed wiring.

#### C. COMPRESSED GAS CYLINDERS

Compressed gas cylinders are potential sources of serious accidents, injuries, and even death if proper precautions and safety practices are not followed. Therefore, during handling and use of these gases, be certain to use applicable safety precautions described by your compressed gas supplier, the Compressed Gas Association, and/or O.S.H.A. regulations.

- 1. Read the label on all cylinders **<u>BEFORE</u>** using to identify the cylinder contents. If the label is illegible, return the cylinder to the supplier. **<u>DO NOT ASSUME THE CONTENTS.</u>**
- 2. All gas cylinders in use and in storage MUST be properly secured to an immovable structure to prevent accidental falling or movement. Read all relevant safety codes.
- 3. Store or move cylinders ONLY in the vertical position.
- 4. DO NOT move or transport cylinders with regulators attached or without safety cap secured over the valve system.
- 5. Store cylinders in a well ventilated area away from heat or ignition sources.

- 6. When installing tubing, provide ONLY proper pressure reducing regulators and pressure relief devices to prevent overpressure of tubing and equipment.
- D. GENERAL
  - 1. Perform periodic leak checks on all fittings.
  - 2. Store organic solvents away from the GC in fireproof, vented, labelled cabinets.
  - 3. DO NOT allow flammable and/or toxic wastes to accumulate.
  - 4. Keep combustibles away from gas cylinders and eliminate ignition sources.
  - 5. DO NOT place papers, charts, samples, etc. on top of the GC.
  - 6. Maintain adequate ventilation.
  - 7. Dispose of wastes properly.

## Section 2 Specifications

The GOW-MAC Series 580 FID is a rugged, compact flame ionization detector (FID) gas chromatograph designed for high sensitivity performance. It is a single column, single detector unit for isothermal operation.

The right section of the chromatograph houses the electronics necessary for proper operation of the instrument. Control of the Series 580 is accomplished by solid-state temperature controllers with digital (LED) meter readout.

The center section of the chromatograph houses the columns, the FID, the injection port, and the flow system. The design of the oven enables the installation of optional accessories including a methanizer and/or capillary column.

The left section (optional) of the chromatograph houses any valves or other options you may have ordered.

#### OVERALL:

H 12 1/2" (317 mm) W 19 1/2" (495 mm) width varies due to various housings used and the options ordered. D 18" (457 mm)

Net weight: 70 lbs. (31.75 kg)

Shipping: 80 lbs (37.50 kg)

#### POWER REQUIREMENTS:

Series 580: 115 VAC, 60 Hz Series 582: 230 VAC, 50 Hz Fuse: Series 580: 10 amps Series 582: 5 amps

#### COLUMN OVEN:

H 7 1/2" (190 mm) W 10" (254 mm) D 8 1/2" (216 mm)

Temperature Range: ambient to 400 °C

Temperature Readout: 3 1/2 digit LED digital meter

Temperature Control: solid state time proportioning, RTD sensors, direct reading, ambient to 400  $^\circ\text{C}$ 

Column Oven Temperature Protection Circuit: shuts the column oven off if the temperature rises to 30 °C over the set point.

Oven Fittings: accommodates 1/8" or 1/4" o.d. metal, 6 mm glass, or capillary columns. Oven Capacity: can accommodate up to 30' of 1/8" columns

#### DETECTOR OVEN:

Temperature Settings: ambient to 400 °C

Temperature Readout: 3 1/2 digit LED digital meter

Temperature Control: solid state time proportioning, RTD sensors, direct reading, ambient to 400 °C

#### DETECTOR:

Detector Type: flame ionization Design: forced air, diffusion Linearity:  $1 \times 10^{6}$ Carrier Gas: N<sub>2</sub>, He, or H<sub>2</sub> Temperature Range: ambient to 400 °C Sensitivity:  $1 \times 10^{-12}$  g/sec hydrocarbon

#### **INJECTION PORT:**

Septum: standard 9 mm
 Temperature Control: solid state time proportioning, RTD sensors, direct reading, ambient to 400 °C
 Temperature Readout: 3 1/2 digit LED digital meter
 Injection Method: direct on-column or gas sample valves

GAS FLOW (conditions may vary depending upon the options chosen for your instruments) Single column with single injection port and exit One metering valve for control of the column

#### ELECTROMETER AMPLIFIER

Circuit: Monolithic FET input operational amplifier powered by a dual polarity tracking regulator to provide balanced positive and negative voltage to the amplifier. Sensitivity: 1.5 x 10<sup>-12</sup> A, full scale 1 mV recorder Dynamic Range: 1 x 10<sup>6</sup> Noise: with cable ±3  $\mu$ V at maximum sensitivity Drift: less than 2  $\mu$ V/hr under controlled environmental conditions Input Ranges: 10<sup>-9</sup>, 10<sup>-10</sup>, 10<sup>-11</sup>, 10<sup>-12</sup> A/mV Output Ranges: binary , 1 to 1024 plus infinity ( $\infty$ )

#### A. GENERAL

The customer should read and become familiar with this section before proceeding with the installation of the SERIES 580 FID GC.

#### B. ADDITIONAL EQUIPMENT REQUIRED

1. Use GOW-MAC Part No. 59-500 Installation Kit for Regulator hookup or 1/8" copper tubing and Swagelok<sup>®</sup> or equal compression fittings.

#### Pressure Regulators

Use high purity 2-stage regulators on gas cylinders installed to provide instrument carrier and/or detector gases (the first stage indicates cylinder pressure and the second adjustable stage controls delivery pressure to the instrument). Regulators should have non-contaminating metal diaphragms and a shut-off valve. The following table lists 2-stage regulators with shut-off valve and a brass 1/8 inch compression outlet connection that are suitable for an 580 FID installation:

GOW-MAC Part Number	CGA cylinder connection	Gas
180-514-3	580	helium, argon
180-515-1	350	hydrogen
180-354	590	air

- 2. Carrier Gas Cylinder: cylinder should be equipped with a regulator and rotameter\*\* terminating in a 1/8" fitting.
- 3. Hydrogen cylinder with regulator, needle valve, and rotameter\*\*.
- 4. Air cylinder with regulator, needle valve, and rotameter\*\*.
- 5. Potentiometric recorder with 1 mV span, 1 sec. response, or 10 mV span. <u>mV INPUT</u> <u>MUST NOT BE GROUNDED</u>. A computing integrator or chromatography software may also be used.
- 6. Gas flows; i.e., H<sub>2</sub>, Air, and carrier, can be measured using a rotameter assembly and a digital or bubble-type flow meter.

- 7. AC power source: Series 580: 1100 W at 115 V, 60 Hz Series 582: 550 W at 230 V, 50 Hz
- \*\* Rotameter and Needle Valve Assembly is available from GOW-MAC P/N 75-300-HE (Helium) or P/N 75-300-N<sub>2</sub> (Nitrogen)

Swagelok®- registered trademark of Crawford Fitting, Inc.



OPERATING INSTRUCTIONS FOR BOTH MODELS ARE THE SAME, EXCEPT FOR LINE VOLTAGE REQUIREMENTS. TO PREVENT DAMAGE TO THE INSTRUMENT, MAKE SURE THAT THE AC ELECTRICAL OUTLET IS THE CORRECT VOLTAGE FOR YOUR INSTRUMENT BEFORE PLUGGING IT INTO THE OUTLET.

8. Column suitable for your application.

#### C. UNPACKING-INSPECTION

- When unpacking the instrument, check it carefully for evidence of shipping damage or rough handling. Check to ensure that all components ordered have either been supplied or back-ordered. Notify the Company of any discrepancies. The packing box should be retained for use if the instrument needs to be returned to the factory for repair or modification. GOW-MAC does not supply field service. ALL repairs are made at Bethlehem, PA USA or by an authorized representative.
- 2. Remove all plastic and/or paper shipping caps and restraints before operating.
- 3. Fill out and mail the yellow WARRANTY-REGISTRATION CARD (included with this manual) to ensure that the warranty will be validated and that you will be kept informed of any improvements or other items of interest.
- D. LOCATION
  - The SERIES 580 FID should be placed in a location that is secure, vibration-free, protected from abrupt temperature changes (operating ambient temperature range is 15 °C - 40 °C), and drafts. Such changes may upset the temperature stability in the course of an analysis or preparation.
  - 2. Enough adjacent tabletop space should be allowed for the installation of recorders, integrators, computers, etc. Allow sufficient space on all sides of the GC for easy access.
  - 3. Make sure that there is adequate space for the installation of the gas cylinders. Cylinders should be securely fastened to the wall or table per CGA and/or OSHA regulations.



READ "SECTION 1 - SAFETY" TO ENSURE PROPER HANDLING OF GAS CYLINDERS.

4. An electrical outlet (ac) should be near the location where the GC is to be installed. If the outlet is not a 3-pin type, make sure that a good ground connection is available, since a good ground is necessary for proper operation. The ac outlet should be connected to a circuit that is not heavily loaded with other electrical equipment because input voltage to the instrument should be steady for optimum operating stability.

If the ac line voltage varies, consideration should be given to the installation of a stabilizing transformer at the ac outlet.



Both recorder and the Series 580 GC should be connected to the same duplex service outlet to prevent ground loops.

E. Power Requirements

The Series 580 GC requires a 115 volt/60 Hz power source capable of providing up to 10 amps. The ac power cord is terminated with a straight-blade 3-prong plug rated for 15 amp service that requires a matching receptacle.

The Series 582 GC requires a 230 volt/50 Hz power source capable of providing up to 5 amps. The ac power cord is terminated with a straight-blade 3-prong plug rated for 10 amp service that requires a matching receptacle.



MAKE SURE ALL SWITCHES ON FRONT AND BACK OF THE SERIES 580 GC ARE IN THE "OFF" POSITION BEFORE PLUGGING IN THE INSTRUMENT.

#### DO NOT PLUG THE INSTRUMENT IN AT THIS TIME!

F. Recorder Connection

Supplied with your GC is a recorder cable. Both ends of the cable terminate in three spade terminals.

Cable color code is as follows: Red lead, positive (+); black lead, negative (-), silver (shielded) lead, ground.

1. Connect one end of the recorder cable to the terminals located at the rear of the GC (Figure 3-1). Connection should be made as follows:

Black to black Red to red Silver to green

- 2. Connect the other end of the recorder cable to the proper terminals on the recorder.
- 3. A floating input must be provided at the recorder.

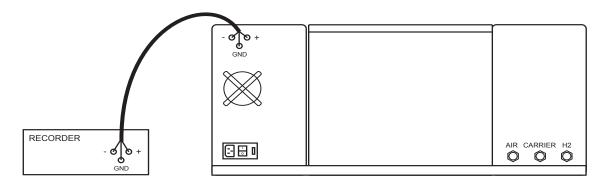


Figure 3-1 (For illustrative purpose only. Instruments may vary.)



The shielded ground wire should be connected to the proper terminal on the recorder, if provided. Follow the recorder manufacturer's instructions for grounding the recorder.

#### G. COMPUTING INTEGRATOR CONNECTIONS

The same cable referred to above can be used for connecting an integrator to the GC.

#### H. GAS CONNECTIONS

1. The flame ionization detector requires the following gases:

Hydrogen (H2) for the FID — Zero Grade (99.995%) Instrument Air for the FID — Zero Grade (99.995%) Helium (He), Nitrogen ( $N_2$ ) for Carrier Gas — Zero Grade (99.995%)

Each cylinder must be equipped with a two stage regulator.

All tubing connections for the carrier gas, hydrogen, and air are 1/8" o.d. While plastic or synthetic tubing may be used for the carrier and air, in the interest of safety, metal tubing should be used for the hydrogen. Either by using the GOW-MAC Installation Kit (part no. 59-400) or fittings and copper tubing of your own, connect 1/8" o.d. pieces of clean tubing from the gas cylinders to their respective inlets (Figure 3-2). High quality nuts and ferrules may be used for column connection. Swagelok<sup>®</sup> or equal compression fittings are recommended.

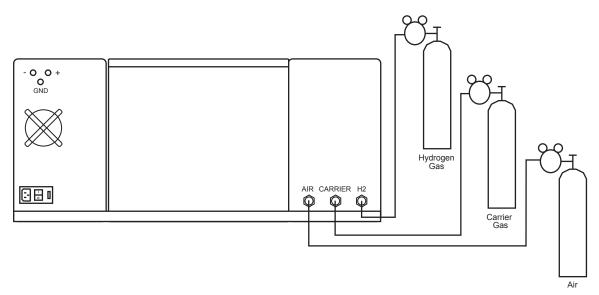


Figure 3-2 (For illustrative purpose only. Instruments may vary.)

To prevent contamination of your GC by grease, oil, or chemical residue, the following procedure should be followed for purging additional stainless steel or copper tubing PRIOR to connecting it to the Series 580 FID GC.

- a. Clean tubing by flushing with acetone to remove any oil residue that may be present .
- b. After washing, let tubing drain and dry.
- 2. All lines and tubing should be clean and free from moisture.

#### I. FLOW ADJUSTMENT FOR CARRIER GAS



MAKE SURE INSTRUMENT SERVICE CORD IS <u>NOT</u> PLUGGED INTO AC POWER SOURCE.

Helium (He) or Nitrogen ( $N_2$ ) may be used for carrier gas. Use the carrier gas for which the rotameter was calibrated.

- 1. Set secondary stage of pressure regulator on cylinder to 40 psig.
- 2. Carrier gas flow is controlled by the needle valve located on the front panel under the column.
- 3. Adjust carrier gas flow to 20-30 mL/min. using the needle valve.
- J. FLOW ADJUSTMENT FOR AIR

Adjust air flow in the FID to 250-300 mL/min. using the knob on the Air rotameter. Use the GOW-MAC 180-567 Digital Flowmeter to check the air flow.



#### HYDROGEN IS AN EXTREMELY EXPLOSIVE GAS! MAKE SURE THAT ALL CIGARETTES, CIGARS, MATCHES, ETC. ARE EXTINGUISHED BEFORE TURNING HYDROGEN "ON"!

Adjust hydrogen flow to 50 mL/min. to initially purge the system and to make ignition of the FID easier— then reduce to the optimum flow rate of 30 mL/min. (0.0636 SCFH). Use the GOW-MAC 180-567 Digital Flowmeter to check the hydrogen flow.



#### L. LEAK CHECK

After all connections have been made, it is important that they be tight and free from leaks. Leaks in the carrier gas system (particularly in the septum or column connections) will cause base line drift, noise, and may reduce sensitivity. Leaks in the air system are not as serious, but may cause erratic performance. Leaks in the hydrogen system are **HAZARDOUS!!** 



HYDROGEN IS AN EXTREMELY EXPLOSIVE GAS!

The lower explosive limit (LEL) of hydrogen with air is 4.0% and the upper explosive limit (UEL) of hydrogen in air is 75%. When oxygen is used the LEL remains the same but the UEL increases to 94%. Care must be exercised in handling this gas. The hydrogen should be turned "OFF" when not in use. The instrument should not be left unattended until the flame has been ignited and the operator is assured that FLAME OUT will not occur.

This instrument has been leak-tested prior to shipment. It is possible, but highly unlikely, that leaks may have developed during shipping. The most likely sources of leaks will be in subsequent connections or re-connections made by the user.



A LEAK CHECK SHOULD BE MADE OF THE ENTIRE GC SYSTEM PRIOR TO INSTRUMENT OPERATION. AND WHENEVER FLOW SYSTEM IS BROKEN. POWER TO THE INSTRUMENT IS TO REMAIN "OFF" THROUGHOUT THIS PROCEDURE!

1. Open the hinged COLUMN OVEN LID.

2. Check all column connections for tightness.

The column supplied with this GC is a test column: 4' x 1/8" stainless steel, packed with Molecular Sieve 5A, 80/100 mesh and requires a 9/16" open-end wrench for tightening or removal.

Columns ordered by customer will vary and may or may not be installed at the factory depending on the customer's instructions at time of order.

- 3. Check septum nut located on the front panel for tightness (should be finger tight).
- 4. Check the carrier gas connection at the back of the GC for tightness.
- 5. Check the hydrogen gas connection at the back of the GC for tightness.
- 6. Check the air connection at the back of the GC for tightness.
- 7. Check all connections at the gas cylinders for tightness.
- 8. The easiest way to locate leaks in the system is through the use of either a GOW-MAC Gas Leak Detector Model 21-080. If a leak detector is not available, the use of a leak testing solution (soap solution) and checking for bubbles may be used.



Leak checks should be run periodically and are a MUST when a new column or gas connection is made.

## Section 4 Operating Controls

This section of the manual will introduce you to the controls of your Series 580 FID GC.

A. Controls

With the exception of the MAIN POWER SWITCH (located on the rear panel), all of the operating controls are located on the right front panel of the GC. The operator should become familiar with these controls and their functions BEFORE operating the instrument. Refer to Figure 4-1.

- 1. DIGITAL PANEL METER: Displays the value of the operating function chosen by the SELECTION BUTTONS.
- 2. SELECTOR BUTTONS:
  - a. COLUMN OVEN TEMPERATURE (° C): Selects column temperature reading to appear on the DIGITAL PANEL METER.
  - b. DETECTOR TEMPERATURE (° C): Selects detector temperature reading to appear on the DIGITAL PANEL METER.
  - c. INJECTION PORT TEMPERATURE (° C): Selects in section port temperature reading to appear on the DIGITAL PANEL METER.
  - d. SET (IN)/ACTUAL (OUT): Selects either actual or set-point parameters for any of the above functions (a-c). This button should be left in the "ACTUAL" (OUT) position <u>EXCEPT</u> when settings are being changed.
- COLUMN TEMPERATURE CONTROL: Selects the temperature of the column oven. Temperature is indicated on the DIGITAL PANEL METER when the COLUMN TEMP. BUTTON is depressed. (See "2d" above). Knob is "locking type". Push locking ring "IN" to turn knob.
- 4. DETECTOR TEMPERATURE CONTROL: Selects the temperature of the detector oven. Temperature is indicated on the DIGITAL PANEL METER when the DET. TEMP. BUTTON is pressed. (See "2d" above). Knob is "locking type". Push locking ring "IN" to turn knob.
- INJECTION PORT TEMPERATURE CONTROL: Selects the temperature of the injection port. Temperature is indicated on the DIGITAL PANEL METER when the INJ. PORT. TEMP. BUTTON is pressed. (See "2d" above). Knob is "locking type". Push locking ring "IN" to turn knob.
- 6. IGNITOR BUTTON: This button operates the igniter on the FID. When pushed and the hydrogen and air are flowing, a glowing hot wire ignites the hydrogen inside the FID.
- 7. COARSE AND FINE ZERO CONTROLS: Adjusts the signal to establish the baseline on the recorder.

- 8. COLUMN HEATER AND FAN ON SWITCH: Controls the column oven heater and fan. The heater will operate only when the switch is in the "UP" position. This is the normal operating position. For rapid cooling of the oven, the heater may be turned "OFF" and the fan operated alone by placing the switch in the "FAN ONLY" position.
- 9. FLAME-OUT INDICATOR LIGHT: Illuminates when flame is not ignited. (optional)
- 10. FLAME-ON INDICATOR LIGHT: Illuminates when flame is lit. (optional)
- 11. RANGE SWITCH: Adjusts the amplification of the electrometer by a factor of 10. Four steps are provided from 10<sup>-9</sup> to 10<sup>-12</sup> amperes.



A change of range using the RANGE SWITCH can cause activation of the Flame Out Circuit (indicated by loss of signal and illumination of the red Flame Out Indicator Light).

Relighting the flame can be done immediately by switching to Flame Out Bypass and simultaneously pushing the Ignitor Switch.

- 12. ATTENUATOR: Attenuates the output of the electrometer, steps are binary from 1x to 1024 plus infinity ( $\infty$ ). The range switch should be set at the proper sensitivity so that the signal remains on scale on the recorder.
- 13. FLAME-OUT MODEL SWITCH (optional): The Flame Out Circuit detects the presence of the ionization current flow (only present when flame is lit). When the ionization current is not present (FLAME OUT), the circuit automatically deactivates the FUEL solenoid valve.
  - a. By-Pass Mode: Overrides fail-safe circuitry thereby allowing FUEL to pass through the system enabling ignition of the FID flame.
  - b. Operate Mode: Activates fail-safe circuit. To be engaged <u>after</u> the FID flame has stabilized. If the flame is accidentally extinguished, the "FAIL-SAFE" circuit will sense "flame-out" by an absence of ionization current, thereby closing the FUEL solenoid valve.
- 14. INJECTION PORT
- 15. METERING VALVE: Adjusts the flow of the carrier gas to the detector.
- 16. OVEN LID

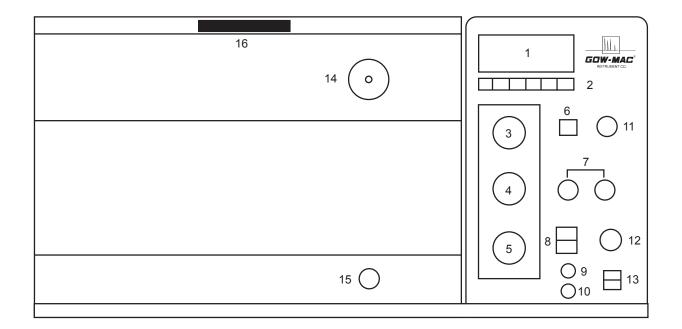


Figure 4-1 (For illustrative purpose only. Instruments may vary.)

- 1. Digital Panel Meter
- 2. Selector Buttons
- 3. Column Temperature Control
- 4. Detector Temperature Control
- 5. Injection Port Temp. Control
- 6. Igniter Button
- 7. Coarse/Fine zero
- 8. Column Heater & Fan On Switch

- 9. Flame-out Indicator Light (optional)
- 10. Flame-on Indicator Light (optional)
- 11. Range Switch
- 12. Attenuator
- 13. Flame-Out Mode Switch (optional)
- 14. Injection Port
- 15. Metering Valve
- 16. Oven Lid
- B. Power and Recorder Connections (refer to Figure 3-1)
  - 1. Power Switch with Fuse: This protects the entire instrument from AC voltage malfunctions. Should power fail to remain ON, remove power cord from AC outlet and check for shorted fuse.
  - 2. Service Cord: DO NOT PLUG IN INSTRUMENT AT THIS TIME. This power cord should be plugged into a grounded receptacle. If grounded receptacle is not available, use a three two adapter but make sure that the ground lead is attached to the screw on the receptacle plate.
  - 3. *Recorder*: A cable is supplied to connect the recorder to the instrument. Refer to Section 3F.
- C. Column

The Series 580 FID GC is complete with two Swagelok<sup>®</sup> fittings for an 1/4" o.d. column, or an 1/8" o.d. column with 1/4" ends can be ordered from GOW-MAC. One-fourth (1/4") to one-eighth (1/8") adapters may also be purchased from GOW-MAC.

The oven is designed to accept twenty-four (24) feet of 1/4" column tubing, using 6 1/4" mandrel. Correspondingly longer lengths of 1/8" tubing can be wound on a mandrel of 4" o.d. diameter (a standard 1 lb. coffee can is about 4" in diameter). Figure 4 illustrates the column supplied with your instrument.

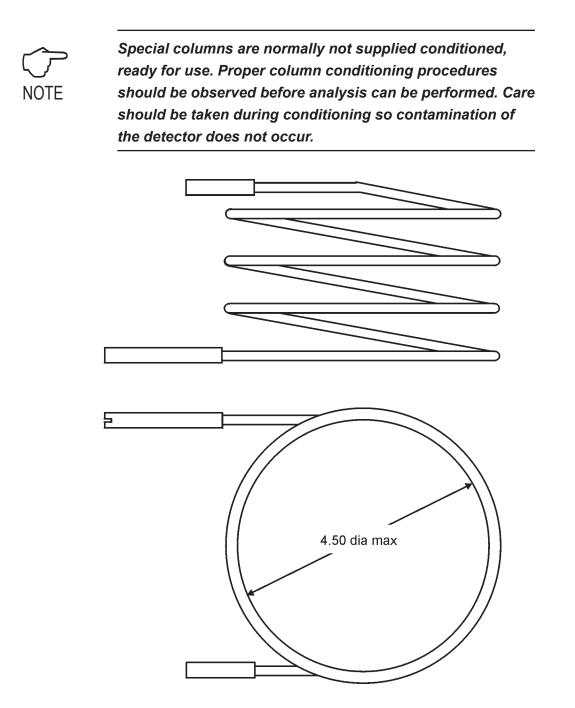


Figure 4-2

#### A. GENERAL

The chromatographer should be familiar with the techniques of chromatography, the functions of all instrument controls, the operation of the recorder, and the characteristics of the column used prior to running samples.

#### B. INITIAL OPERATION

- 1. Carrier Gas Flow Adjustment (Refer to Section 3, I)
  - a. Make sure that ALL switches are in the "OFF" position.
  - b. Set the Helium or Nitrogen carrier gas flowing with the secondary stage regulator on the gas cylinder reading 40 psig.
  - c. Adjust the carrier gas flow to 20-30 mL/min. using the NEEDLE (METERING) VALVE located on the front control panel. The needle valve is closed when it is turned fully clockwise (CW). Some instruments control the flow using a rotameter located on the front of the Accessory housing. Refer to the flow diagram for the location of the carrier flow control.
  - d. Check for leaks as described in Section 3, L.
  - e. Allow 10 minutes to purge the system before turning power on to the instrument.
- 2. Warm-Up
  - a. Plug the instrument into the appropriate ac outlet and turn instrument "ON".
  - b. Set DETECTOR TEMPERATURE to desired setting (e.g. 210 °C).



Allow detector oven to reach temperature selected <u>BEFORE</u> turning on injection or column ovens. The detector should be maintained at the highest temperature component of the system to avoid condensation and possible contamination of the flame tip.

c. Set desired COLUMN TEMPERATURE and INJECTION PORT TEMPERATURE.



DO NOT HEAT COLUMN ABOVE UPPER TEMPERATURE LIMIT OF COLUMN LIQUID PHASE. IF INSTRUMENT IS EQUIPPED WITH VALVES, DO NOT HEAT ABOVE 275 °C.

- d. Allow adequate time for the injector and column ovens to reach set temperatures and all temperature-controlled zones have stabilized.
- 3. Flow Adjustments For Hydrogen & Air
  - a. Adjust the hydrogen and air flows as described below:
    - i. Air

Adjust air flow to 300 mL/min. using the knob on the AIR Rotameter.

ii. Hydrogen

Hydrogen flow can be set to 50 mL/min. by adjusting the knob on the HYDROGEN Rotameter. This creates a hydrogen rich flame. Once the flame is ignited, decrease the hydrogen flow to 30 mL/min. for operation.

Recommended operation flows				
Carrier	30 mL/min.			
Air	300 mL/min.			
H <sub>2</sub>	30 mL/min.			



#### DO NOT LEAVE THE HYDROGEN FLOWING FOR MORE THAN 3 MINUTES WITHOUT IGNITION\*. READ REMAINDER OF INSTRUCTIONS BEFORE TURNING HYDROGEN ON.

\* May be longer on initial start up.

- b. For instruments equipped with a Methanizer, refer to Section 10 for a complete description of Methanizer operation.
- 4. Flame Ignition
  - a. Igniter

Heating the FID to operating temperature above 100 °C is recommended before igniting the flame in order to minimize water condensation in the detector. When ready to ignite the flame, initiate the flows of air and hydrogen to the FID. Press the IGNITER switch on the control section front panel. The hot wire igniter remains energized only as long as the IGNITER switch is pressed.



FLAME DETECTION CIRCUIT: In instruments with the flame detection circuit installed, the FLAME OUT MODE momentary switch must be held in the BYPASS position to allow the flow of hydrogen to the detector. Upon successful ignition, the circuit detects the flame, the FLAME ON indicator light illuminates, and the fuel shut-off valve remains open. The BYPASS switch can be released when the FLAME ON light is illuminated. Ignition is indicated by a slight "pop" sound along with a sharp <u>step</u> increase in instrument signal. Conversely, a transient <u>spike</u> in signal indicates momentary combustion in the detector, but not the desired steady flame. Condensation is a secondary method of confirming the presence of a flame. Hold a smooth glass or mirror to the FID chimney vent to catch and observe condensation.

- i. If the flame fails to ignite all flows should be rechecked.
- ii. If flows have been checked and the flame still fails to ignite, air and/or carrier gas flows may need to be reduced in order to concentrate the hydrogen and facilitate the ignition of the flame. After ignition, readjust the flows.

## iii. DO NOT LEAVE THE HYDROGEN ON FOR LONG PERIODS OF TIME WITHOUT IGNITION AND CONTINUOUS COMBUSTION.

iv. The purity of the air, hydrogen and carrier gas will have an effect on baseline drift and noise.

#### 5. Strip Chart Recorder Zeroing

The Series 580 FID GC is suitable for use with almost any strip chart recorder of the potentiometric type having a 1 mV full-scale span. An adjustable chart drive is also recommended: 40, 20, 10, 4, 2, 1 cm/min. and hour.

After the recorder is properly connected to the GC, it may be turned "ON". At this time the recorder zero should be established using the RANGE and COARSE & FINE ZERO CONTROLS. Proceed as follows:

- a. Set ELECTROMETER ATTENUATION SWITCH to infinity ( $\infty$ ), adjust the RECORDER ZERO CONTROL to the desired value, i.e. 0%, 5%, 10%, etc.
- b. Set the ATTENUATOR to "8" and adjust the ELECTROMETER ZERO CONTROL to position the recorder pen on the preestablished recorder zero.
- c. Observe the detector output signal for drift. If drift is more than 1% (of chart width) per minute, allow more time for the detector to stabilize or column bleed to minimize.
- d. When the baseline has stabilized, readjust the ELECTROMETER ZERO CONTROL for desired zero level on the chart.
- e. To establish the zero for the remaining three (3) ranges, follow the above procedure for the particular range you choose.

Refer to the recorder's operating manual for correct chart speeds, warm-up times, etc.

6. Computing Integrator Zeroing

The Series 580 FID GC may also be used with a computing integrator. The integrator makes full data acquisition more reliable and more accurate.

After the integrator is properly connected to the instrument, it may be turned "ON". At this time, the electrical or recorder zero should be established and reference should be made to the integrator's operating manual.

- 7. Calibration
  - a. After allowing sufficient time for the Series 580 FID GC to equilibrate and the operating parameters are rechecked, adjust the RANGE SWITCH and ATTENUATOR to the sensitivity required for the analysis.
  - b. Initial Analysis- The operation of the instrument cannot be evaluated without injection of a sample. Initial injections should be made with a relatively simple material that will elute in a short period of time. The sample should be as pure as possible to give 2 or 3 sharp peaks. <u>No attempt</u> should be made to start with complicated samples. Techniques of injections and analysis of data will require practice.
  - c. Make solutions of known concentrations of the components of interest. These standards should bracket (span) the concentration of interest. If the analysis is normally analyzing in the 0.2.% range, the standards should be 0.1%, 0.2%, and 0.3%. These standard solutions should be made up by either WT/WT or VOL/VOL, but this must be consistent and not switched from one method to the other.
  - d. Successive injections of the same sample size and standard should be made and the average output plotted to yield a curve of output vs concentration.
- 8. Standby and Overnight Conditions

When the instrument is used intermittently during the day or is needed right away the next morning, it is recommended that the instrument be kept in "STAND-BY" condition. This keeps the instrument ready to use without waiting for a long equilibration period. Proceed as follows:

- a. Reduce the carrier gas flow to save gas consumption.
- b. Turn Hydrogen flow "OFF".
- c. Turn Air "OFF".
- d. Turn recorder chart speed "OFF".
- e. Place ATTENUATOR on infinity ( $\infty$ ).



FOR CERTAIN SAMPLE COMPOSITIONS THAT INTERACT WITH ATMOSPHERICS (OXYGEN, MOISTURE, ETC), IT IS <u>NECESSARY TO THOROUGHLY PURGE</u> <u>THE ENTIRE</u> INSTRUMENT OF SAMPLE GAS <u>BEFORE</u> SHUTDOWN USING AN INERT GAS — INCLUDING THE SAMPLE INLET. THIS IS REQUIRED FOR SAMPLES WITH HIGH CONCENTRATIONS OF COMPOUNDS THAT FORM ACIDS (HF, HCI, HBr, ETC) OR REACT VIOLENTLY AND/ OR CORROSIVELY WITH ATMOSPHERIC ELEMENTS.

9. Shut-Down Procedure

The following should be performed in the given sequence to ensure proper cool down of your GC and longer life of the detector.

- a. Turn the recorder "OFF".
- b. Shut "OFF" the Hydrogen flow. Confirm that the flame has been extinguished.
- c. Shut "OFF" the Air flow.
- d. Reduce the temperature settings to 25° C. For a quicker cool-down of the oven, the oven lid may be raised and the COLUMN HEATER & FAN SWITCH placed in the "FAN ONLY" position.
- e. Turn the power "OFF".
- f. After oven has cooled considerably, shut the carrier gas "OFF".
- g. Recheck all gas cylinders to confirm flow has been shut off.

#### C. Daily Setup Check List

It is good practice to check the following items at the beginning of each day or shift, and when starting up the instrument after a weekend shutdown.

1. Electrical

\_\_\_\_\_ Additional instrumentation is connected properly.

2. Pneumatic

\_\_\_\_\_ Gas cylinder supply pressures are sufficient.

Air = 40 psig  $H_2$  = 40 psig He = 40 psig \_\_\_\_ Gas flow rates are adjusted properly.

Air = 300 mL/min.

- $H_2 = 30 \text{ mL/min.}$ He = 30 mL/min. or as necessary for analysis

\_\_\_\_\_ Appropriate column is installed.

\_\_\_\_\_ Leak check made.

#### 3. Front Panel

\_\_\_\_\_ All temperature settings are set to desired settings.

\_\_\_\_\_ Recorder/Integrator zeroed.

## Section 6 General Notes on FID Operation

A. Maintain flows approximately as follows:

Hydrogen (H <sub>2</sub> )	30 mL/min.
Air	300 mL/min.
Carrier	30 mL/min.

- B. At start up, some operators use a higher hydrogen flow to permit faster ignition. This is a dangerous procedure. CARE SHOULD BE EXERCISED.
- C. The flame cannot be seen; a small watch glass or mirror placed above the exhaust will show that the flame is ignited and burning by condensation on the glass. Tapers, paper, or matches should not be inserted into the exhaust as they will cause contamination inside the combustion chamber. They will usually put out the flame if already ignited.



<u>DO NOT</u> LOOK INTO THE EXHAUST TUBE TO CONFIRM IGNITION. PERMANENT EYE DAMAGE MAY RESULT!

- D. Excessive carrier flow will cause the flame to go out. The combination of hydrogen and carrier gas should not exceed 60-70 mL/min.
- E. Excess hydrogen will result in noise. Insufficient hydrogen flow will cause flameout.



INSUFFICIENT AIR FLOW IS DANGEROUS AND CAN RESULT IN AN EXPLOSION.

<u>DO NOT</u> LET HYDROGEN ACCUMULATE IN THE DETECTOR! UPON RE-IGNITION OF THE FLAME, AN EXPLOSION MAY OCCUR, UNLESS HYDROGEN HAS BEEN ALLOWED TO DISSIPATE.

G. Column/Injection Notes

The column supplied with this GC is a test column: 4' x 1/8" stainless steel, packed with Molecualr Sieve 5A, 80/100 mesh and requires a 9/16" open-end wrench for tightening or removal.

Care should be exercised to ensure that this column is not used below 30  $^{\circ}$ C or above 375  $^{\circ}$ C. This column has been preconditioned.

Columns ordered by customer will vary and may or may not be installed at the factory depending on the customer's instructions at time of order.

#### H. Injector Temperature

The injector should be operated at a temperature that assures complete vaporization of the sample. Care should be taken to use septa that will withstand the operating temperature of the injection port. If necessary, they must be replaced after several injections if a seal cannot be obtained. The septum nut acts as a heat sink, it should be kept clean and polished.

#### I. Sample Injection

Proper techniques have been outlined in the literature. Practice is necessary for consistent injection of samples. Sharp, straight needles, and smooth rapid injection are needed to produce proper and complete vaporization of samples.

## Section 7 Maintenance & Service

A. FID Cleaning & Servicing

**NO ATTEMPT** should be made to take the FID apart for repair or cleaning.

Call our Repair Department at (610) 954-9000 for assistance.

B. Columns

NOTE

When ordering a new column, please specify:
Instrument Model No. & Serial No.
Whether the column is for Side "A" or "B"
Column Length, Diameter, and material
% Liquid Phase
% Loading
Solid Support
Mesh Size

- 1. The Series 580 FID GC comes supplied with one test column (unless otherwise stated at time of order):
  - 4' x 1/8" stainless steel, packed with Molecular Sieve 5A, 80/100 mesh.

Care should be exercised to ensure that this column is not used below 30  $^{\circ}$ C or above 375  $^{\circ}$ C. This column has been preconditioned.

2. Columns for the Series 580 FID can be purchased from:

GOW-MAC Instrument Co. 277 Brodhead Road Bethlehem, PA 18017 Tel: (610) 954-9000 Fax: (610) 954-0599

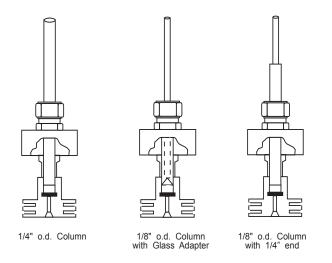


Figure 5-2

3. Changing Columns

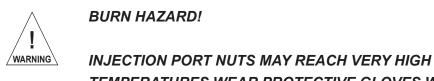
Figure 5-2 shows the injection port for low dead volume and on column injection. Either 1/4" or 1/8" o.d. columns can be used as shown. All 1/8" o.d. columns are furnished with 1/4" ends with a 2 mm i.d. opening.

Care should be exercised when changing or removing columns. Damage to adjacent threads may occur if they are hit with a wrench or other object which may result in nuts becoming cross threaded.

Care should also be used when inserting the columns into the injection port assembly. Insert the column until it stops, making sure that it has reached the beveled end of the injection port. Then back off about 1/4" to allow the carrier gas to sweep into the column. By using this technique, on-column injections can be attained. CAREFULLY tighten nuts with a 9/16" open-ended wrench (or 7/16" wrench for 1/8" fittings). CHECK FOR LEAKS!

C. Septums

The septa used in this instrument are standard 9 mm o.d. and may be obtained directly from GOW-MAC (Part No. 180-123S). The INJECTION PORT NUT may be removed and the SEPTUM replaced.



#### INJECTION PORT NUTS MAY REACH VERY HIGH TEMPERATURES WEAR PROTECTIVE GLOVES WHEN HANDLING INJECTION PORT NUTS

D. Temperature Control

The Series 580 FID can be operated at temperatures ranging from ambient to 400 °C. Temperature controls on the injection ports, column oven, and detector are independent solid state proportioning type. Proportioning cycle rate is approximately 2 1/2 seconds with a total band width of 5 °C. Temperature readout and "SET" are on a 3 1/2 digit LED digital meter.

Selector buttons are used to read the desired temperatures.

A centrifugal blower fan circulates and distributes the heated air, thus eliminating temperature gradients. The blower also provides rapid cool down when needed.

E. Column Oven

The column oven is heated by a 500 W tubular heater controlled by a proportional control. The solid state control incorporates a platinum RTD (Resistance Temperature Detector) and a 3 3/4 turn set potentiometer. Built into the circuitry is a fail-safe feature which disables the controller's triac output in the event of a shorted or open sensor.

The "Temperature Fail Safe" feature has independent shut down at 400 °C for injection ports and detector. The column oven shuts down 30 °C above the column set point. The heating units return to safe condition when the temperatures decrease to safe levels. The controller is easily removed for service or replacement.

F. Detector Temperature Control

The detector is heated by one (1) 100 W heater mounted in the DETECTOR BASE. The temperature control sensor is also located in the base.

The detector temperature is controlled in the same manner as the column oven.

G. Injector Temperature Control

The injection port is heated by a 60 W heater and is mounted inside the injector block. The temperature control sensor is also located in the block.

The injection port temperature is controlled in the same manner as the column oven.

H. Servicing

Allow 10 minutes for high voltage discharge after power is turned "OFF", before attempting to service the GC.

Servicing of the complex integrated circuits in the electronic housing of the unit should be performed by qualified personnel only. All calibrations and adjustments are made at the factory before shipping. Once these calibrations are performed, they should never need adjustment again during the life of the unit.

If questions arise that this manual does not answer or service of your GC goes beyond these instructions, please call our Repair Department at (610) 954-9000.

For a complete overhaul and cleaning of the FID or the entire instrument, please call us for a Return Authorization Number and further instructions for the return of your GC. Our receiving address for repairs is:

GOW-MAC Instrument Co. Attn: Repair Dept. RMA#\_\_\_\_\_ 277 Brodhead Road Bethlehem, PA 18017

Upon inspection of the instrument, repair costs will be furnished, IF REQUESTED, prior to repair.

If your instrument is not equipped with valves, go on to Section 9.

A. General

Many types of valve configurations are available with your Series 580 GC. For assistance in determining the proper valve for your application, contact GOW-MAC.

All valves must be treated with care. Foreign materials such as metal filings or abrasive particles can permanently damage the sliders of the valves. GOW-MAC installs stainless steel frits on the inlets of sampling valves to help protect against this type of damage.



HIGH TEMP VALVES: 175 °C - 300 °C

All valves may be fitted with pneumatic actuators which are available from GOW-MAC. Air pressure of 30-60 psig is required for actuation.

B. Valves and Their Functions

Valves are used to accomplish two basic operations in GC. One is to inject a sample onto the head of the GC column and the other, to reroute or "switch" the flow of the carrier gas or sample stream.

Within the broad category of switching, there are many valve functions, such as backflushing, detector switching or column selection. (See action G Typical Gas Sampling Valves).

Although most valves are categorized as either sampling or switching, some valves combine both functions and are termed "multifunction" valves.

C. Sampling Corrosive Materials

When dealing with harsh samples, such as chlorine and wet acid gases, valves made of Tantalum or Hastelloy C-276 are recommended.

#### Corrosion Resistance of Tantalum to Some Common Chemicals

<u>Excellent</u> Sulfuric Acid Hydrochloric acid <u>Slow Attack</u>

Strongly alkaline compounds <u>Not Recommended</u> Hydrofluoric acid Fluorine gas

Nitric Acid All organic corrosive chemicals

#### Corrosion To Resistance of Hastelloy C-276 Some Common Chemicals

#### <u>Excellent</u>

Acetic acid Amines Ammonia Chlorine (dry) Formic acid Hydrogen chloride (dry) Hydrogen sulfide Phosgene Sulfur dioxide **Good** Bromine Gas Chlorine (wet) Hydrochloric acid Nitric acid Phosphoric acid

### Not Recommended

Fluorine Hydrofluoric acid Hydrofluoride

D. Gas Sampling Valve

The gas sample valve is used to introduce gas samples into the chromatograph on a reproducible basis. The sample may be taken from a static system or from a flowing stream. Valves are also used to back-flush column, column selection, sample selection and detector switching.

Since the most common use of the valve is for sample injection, only that application will be discussed here in general terms. The valve may be installed in place of, or in series with the injection port. The valve may be permanently connected to a sample source or the sample may be passed through by means of a pump or other sample container.

The size of the sample loop is fixed but can be changed easily. (Section E Sample Loops).

The valve is first placed in the counter clockwise (CCW) position, that is, the valve handle is as far counter clockwise as it will go. At this time the sample is purged through the loop and the carrier gas merely passes through the valve to the column. When the valve handle is placed in the clockwise (CW) position, the carrier gas purges the sample from the loop and carries it through the column. The valve is then returned to the CCW position.



The sample is released to atmosphere in either valve position.

Care must be exercised to allow sufficient time for the sample loop to be completely filled with the sample before injection. This is easily calculated from the carrier gas flow and size of the sample loop. The same holds true for time allowed for the sample to enter the column.

## E. Sample Loops

Sampling valves are supplied with a .25 mL loop if not otherwise specified on the order. Other sample loops are available: 0.25 mL, 0.50 mL, 1.0 mL, 2.0 mL, 3.0 mL, 4.0 mL, 5.0 mL, 10.0 mL, and 20.0 mL.

## PROCEDURE FOR REMOVING AND REPLACING LOOPS IN GAS SAMPLING VALVES

- 1. Remove the four (4) screws holding the valve housing lid in place. Remove valve housing lid.
- 2. Unscrew loop mounting fittings (2) and remove loop.
- 3. Insert new loop and tighten fittings.
- 4. Replace valve housing lid.
- F. Pneumatic Actuated Valves

If pneumatic actuated gas valves are installed, you may override the auto feature by a switch on the accessory housing panel. An air pressure of about 30-60 psig is recommended to fully drive the valve.

Option 411 Interface PCB is used to activate the auto valves from a TTL closure generated by an external source, i.e. Computing Integrator. Option 411 will time a maximum of three (3) valves.

Option 412 accessory accepts up to three (3) valves in a separate oven assembly located in the accessory housing.

- G. Automatic Valve Operation (Option 411: TTL Interface)
  - 1. The valves in the Series 580 FID Gas Chromatograph can be operated either manually or through the use of the TIME FUNCTIONS control on a computing integrator.
  - 2. Time Functions

The valves correspond to the following "Time Functions":

T3 = Valve 1 T4 = Valve 2 T5 = Valve 3

## 3. Programming

Programming can be done as either part of the dialogue or by directly using the TFN command on the computing integrator keyboard.

4. Setting Time Functions

When programming the TFN, the computing integrator will ask for three (3) values:

TT = \_\_\_\_\_(when to activate)

TF = \_\_\_\_\_(what to activate)

The TT is the time the valves are to switch.

Example: Injection valve to inject sample after 1 minute, enter TT = 1.0

To activate Gas Sample Valve, enter TF = T3

To turn valve one, enter TV = 1

# Section 9 Capillary Chromatography

If you did not order a capillary system with this GC, go on to the next section.

### A. Introduction

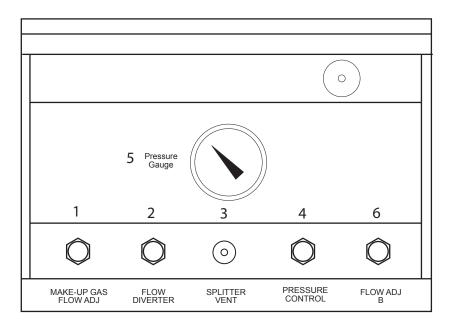
Capillary chromatography provides the utmost in GC performance. Advantages to using capillary techniques include high efficiencies — about 4000 plates per meter versus about 500 plates per meter for packed columns — and shorter retention times with better resolution.

If your sample does not have a complex mixture of components, it is possible to operate the system at higher than optimum temperatures and flow rates, therefore speeding up the analysis time. Using the capillary column samples that normally take 30 minutes to elute on a packed column, can be separated with greater resolution in one or two minutes.

These columns consist of open tubes from 10 meters to 100 meters in length and from 0.5 - 0.2 mm inside diameter; they may be made of glass, stainless steel or fused silica. The inside of the tubes are coated with the liquid phase in thickness of 5 microns to less than 1 micron. Because the liquid phase coating is relatively thin and the volume of these columns so small, these columns are easily overloaded. To overcome the effects of overloading, an inlet splitter is used which divides the amount of sample injected into the instrument. A small portion of the sample enters the column and the major portion is vented through a charcoal trap into the atmosphere.

Overloaded peaks can be identified easily by observing the peaks as they elute. If the pen goes up slowly and comes down rapidly (i.e. the slope of the front portion of the peak is resolved more gradually than the trailing part) then the peak is overloaded and a smaller sample should be injected or higher split ratio used to assure that smaller samples reach the column.

- B. Operation of the Capillary System
  - 1. MAKE-UP GAS FLOW ADJ: used to regulate make-up gas.
  - 2. FLOW DIVERTER: used to measure make-up gas.
  - 3. SPLITTER VENT: outlet from splitter where ratio is measured.
  - 4. PRESSURE CONTROL: used to set flow rate through the column.
  - 5. Pressure Gauge: measures pressure at the head of the column.
  - 6. FLOW ADJ B: use to adjust total system flow. Read from helium rotameter.





C. Installation of Capillary Column

**Fused Silica Columns** 

1. Fused silica columns are mounted exactly as above with two exceptions, 1) the column come mounted in a cage and 2) the smaller diameter columns can fit inside the glass tubing of the injector and the make-up gas tee, eliminating some of the dead volume.

Columns should be inserted 70 mm into the splitter. If the column will fit inside the flame tip it can be inserted 85 mm from the 1/4" nut on the FID.

- 2. It is recommended that ferrules with an inner diameter matching the size of the column be used for the installation of fused silica columns.
- 3. Injection Systems
  - a. Option 103 Split/Splitless Injection System
  - b. Option 104 Direct, Wide Bore System

Both systems include make-up gas connections for the FID. A metering valve controls the make-up gas to allow optimum flow to the FID.



A Rotameter Flow Assembly (p/n 75-300-HE or 75-300-N2) and the Hand-held Digital Flowmeter (p/n 180-567) are highly recommended for use with capillary columns.

When installing a new capillary column, the column should be placed into the detector as far as it will go. The other end of the column should be placed into the glass adapter approximately 3/4".

- D. Operation of the System
  - 1. Using FLOW METERING VALVE "B", set the flow to 30 mL/min. Read off of the helium rotameter.
  - 2. Put FLOWMETER onto the SPLITTER VENT.
  - 3. Using the PRESSURE CONTROL, set the flow out of the SPLITTER VENT to 27 mL/min.
  - 4. In theory, this should leave 3 mL/min. going through the column. This should yield a 27:3 or 9:1 split.
  - 5. Use the make-up gas measured at the MAKE-UP GAS FLOW VENT to bring your total gas into the FID up to 30 mL/min.
  - 6. In order to determine the flow rate of the column, inject a 5 10  $\mu$ L sample of methane and record the elution time in seconds.
  - 7. At this point, the split ratio can be calculated by measuring the flow rate using a bubble flowmeter connected at the SPLITTER VENT and adjusting the flow rate by adjusting the PRESSURE CONTROL to give a split ratio between 10:1 100:1 as required.

Split Ratio = <u>Amount of Gas coming out vent</u> Flow in mL/min through column

In most literature, capillary flows are expressed as a linear velocity.

Linear Velocity = <u>Column length in cm</u> Seconds

A flow rate in mL/min. is needed to calculate the split ratio. The flow rate in mL/min. can be calculated from the volume of the column.

 $V = \pi r^2 H$ 

Flow rate =  $V \times 60$ Sec for CH<sub>4</sub> to elute For a 15 m x 0.24 mm column, r = 0.0122 cm

 $V = \pi (0.0 \ 122)^2 \ x \ 1500$  $V = 0.70139 \ cc$ Flow rate =  $\frac{0.70139 \ cc \ x \ 60 \ sec/min.}{34 \ sec.}$ 

Flow rate range for 0.2 mm quartz columns is 0.5 - 1 cc/min.



For a direct read of the flow, connect the capillary column adapter (p/n 180-966-2) to the end of the column, then connect the other end of the adapter to a flowmeter.

- 8. Examine the methane peak; no tailing should be observed. If tailing is present, this indicates that the column is not installed properly.
- 9. Inject your sample with a swift smooth action of the plunger.

When injecting samples, make sure to insert the syringe needle about 2" through the injection port. This will ensure proper injection technique.

Sample volume should NOT exceed 0.2  $\mu L$  for capillary columns and 0.5  $\mu L$  for wide bore capillary columns.

# Section 10 Ruthenium Methanizer

If your GC is not equipped with Option 401-Ruthenium Methanizer, please go on to the next section.

A. General

The Option 401 Ruthenium Methanizer is designed to be used with FID gas chromatographs for the sensitive determination of CO and  $CO_2$ . With the broad linear range of the flame ionization detector, both high and low level concentrations of these gases can be analyzed with excellent results.

If your instrument was not equipped with Option 401 at the time of purchase, our Model 59-125 Ruthenium Methanizer Kit will retrofit on your Series 580 FID GC and all other gas chromatographs.

B. Operation

Conversion of CO and CO<sub>2</sub>

The Option 401 catalytic methanizing unit is designed for in-line conversion of CO and  $CO_2$ . The conversion is:

$$CO + 3H_2 \rightarrow CH_4 + H_20$$
$$CO_2 + 4H_2 \rightarrow CH_4 + 2H_20$$

Maximum conversion of CO and CO<sub>2</sub> can be achieved with fully reduced ruthenium catalyst which comes packed in the GOW-MAC methanizer. Operated at 350 °C, the hydrogen gas flow to the FID burner is routed through the methanizing unit. An auxiliary flow of carrier gas is then used to obtain proper carrier H<sub>2</sub> ratio for detector sensitivity.



WHEN METHANIZER IS INSTALLED ON GOW-MAC FID GC's, A CAP IS INSTALLED ON H<sub>2</sub> INLET TUBE.

- 1. Series 580 FID GC Methanizer Operation
  - a. Temperature control of the methanizer is achieved with a separate control unit mounted in the Accessory Housing (located on the left side of the GC). Included in the temperature control unit is:
    - Dial to set temperature
    - Meter for temperature readout

- b. In order to obtain the fast heat-up, the Methanizer Temperature Control should be turned to maximum for a specific time. The length of time for the control to be at maximum must be determined experimentally. The Ruthenium Methanizer should be heated to a temperature of 350 °C with the hydrogen flow "ON" for a period of 30 minutes, allowing the temperature to stabilize.
- c. For Series 580 FID GCs using a 6-port Series Bypass Valve
  - i. Methanizer H<sub>2</sub> IN connection is located on the back of the instrument.
  - ii. Use needle (metering) valve located on the front of the instrument for flow adjustment.
  - iii. Measurement of  $H_2$  flow must be made at the point where the carrier flow enters the FID. This requires disconnecting the flow system at the 1/16" Swagelok<sup>®</sup> fitting.



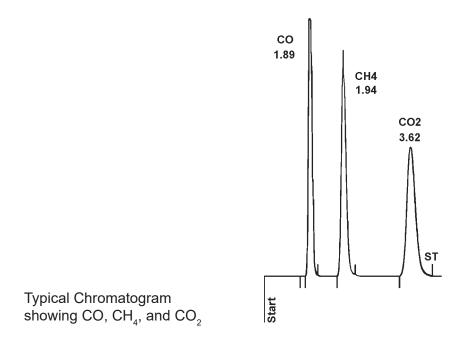
A leak check MUST be performed after the flow system is reconnected to ensure a hydrogen gas leak is not present.

- d. For Series 580 FID GCs using a 10-port Series Bypass Valve
  - i. Methanizer H<sub>2</sub> IN connection and flow adjustment are located on the 75-300 Rotameter Assembly.
- C. Example procedure for use of ruthenium Methanizer described in a typical analysis to determine the efficiency of the methanizer to reduce CO,  $CO_2$  quantitatively to  $CH_4$ .
  - 1. Ruthenium Methanizer Conditions
    - a. Prior to use, the Ruthenium Methanizer should be heated to a temperature of 350° C with the hydrogen flow "ON", for a period of "30 minutes, allowing the temperature to stabilize.
    - b. Analysis Parameters

Column:	6' x 1/8" S.S.	Sample:	1% CO, 1% CH <sub>4</sub> , CO <sub>2</sub>
Support:	Spherocarb	Sample	Size: 2 mL
Carrier Gas:	He	Temp:	
Carrier Flow:	30 mL/min.	Column	140 °C
H Flow:	30 mL/min.	Inj. Port	150 °C
Air Flow:	300 mL/min.	Detector	180 °C
Recorder:	1 mV	Attenuation:	2 <sup>2</sup> x 10 <sup>-9</sup>
Methanizer Temp:	350 °C	Chart Speed:	1 cm/min.

c. The sample used is a certified gas mixture containing 1% each of argon, carbon dioxide, carbon monoxide, hydrogen, nitrogen, and methane with the balance of helium being allowed to completely flush a 500 ml gas sampling tube at ambient temperature. After this procedure, 0.5, 1.0 and 1.5 mL quantities of the gas mixture should be removed with a gas tight syringe and injected into the gas chromatograph.

At this point, the following peaks should be observed:



c. RESULTS: For CO<sub>2</sub> the following results were tabulated:

<u>mL injected</u>	<u>peak area</u>
0.5	288,800
1.0	444,820
1.5	690,700

These results indicate a linear relationship with correlation coefficient of 0.9917, the equation of the linear regression line being y = 401900 + 12873.

Sigma-Aldrich

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## SAFETY DATA SHEET

Version 6.3 Revision Date 08/23/2023 Print Date 01/13/2024

## SECTION 1: Identification of the substance/mixture and of the company/undertaking

#### 1.1 Product identifiers

	Product name	:	Ruthenium(III) chloride trihydrate
	Product Number Brand CAS-No.	::	10452 Aldrich 13815-94-6
1.2	Relevant identified use	es	of the substance or mixture and uses advised against
	Identified uses	:	Laboratory chemicals, Synthesis of substances
1.3	Details of the supplier	of	the safety data sheet
	Company	:	Sigma-Aldrich Inc. 3050 SPRUCE ST ST. LOUIS MO 63103 UNITED STATES
	Telephone Fax	:	+1 314 771-5765 +1 800 325-5052
1.4	Emergency telephone		
	Emergency Phone #	:	800-424-9300 CHEMTREC (USA) +1-703- 527-3887 CHEMTREC (International) 24

Hours/day; 7 Days/week

#### **SECTION 2: Hazards identification**

## 2.1 Classification of the substance or mixture GHS Classification in accordance with 29 CFR 1910 (OSHA HCS) Skin corrosion (Category 1B), H314 Serious eye damage (Category 1), H318 For the full text of the H-Statements mentioned in this Section, see Section 16. 2.2 GHS Label elements, including precautionary statements Pictogram

Signal Word



Aldrich - 10452

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Causes severe skin burns and eye damage.
Do not breathe dust. Wash skin thoroughly after handling. Wear protective gloves/ protective clothing/ eye protection/ face protection.
IF SWALLOWED: Rinse mouth. Do NOT induce vomiting.
IF ON SKIN (or hair): Take off immediately all contaminated clothing. Rinse skin with water/ shower.
IF INHALED: Remove person to fresh air and keep comfortable for breathing. Immediately call a POISON CENTER/ doctor.
IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing. Immediately call a POISON CENTER/ doctor.
Wash contaminated clothing before reuse.
Store locked up.
Dispose of contents/ container to an approved waste disposal plant.

#### 2.3 Hazards not otherwise classified (HNOC) or not covered by GHS - none

#### SECTION 3: Composition/information on ingredients

3.1	<b>Substances</b> Formula Molecular weight CAS-No.	: Cl₃Ru · 3H₂O : 261.47 g/mol : 13815-94-6		
	Component		Classification	Concentration
	Ruthenium(III) chloi	ide trihydrate		
			Skin Corr. 1B; Eye Dam. 1; H314, H318	<= 100 %

For the full text of the H-Statements mentioned in this Section, see Section 16.

#### SECTION 4: First aid measures

#### 4.1 Description of first-aid measures

#### **General advice**

First aiders need to protect themselves. Show this material safety data sheet to the doctor in attendance.

#### If inhaled

After inhalation: fresh air. Call in physician.

#### In case of skin contact

In case of skin contact: Take off immediately all contaminated clothing. Rinse skin with water/ shower. Call a physician immediately.

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1illipore

#### In case of eye contact

After eye contact: rinse out with plenty of water. Immediately call in ophthalmologist. Remove contact lenses.

#### If swallowed

After swallowing: make victim drink water (two glasses at most), avoid vomiting (risk of perforation). Call a physician immediately. Do not attempt to neutralise.

- **4.2 Most important symptoms and effects, both acute and delayed** The most important known symptoms and effects are described in the labelling (see section 2.2) and/or in section 11
- **4.3 Indication of any immediate medical attention and special treatment needed** No data available

#### **SECTION 5: Firefighting measures**

#### 5.1 Extinguishing media

#### Suitable extinguishing media

Use extinguishing measures that are appropriate to local circumstances and the surrounding environment.

#### Unsuitable extinguishing media

For this substance/mixture no limitations of extinguishing agents are given.

#### 5.2 Special hazards arising from the substance or mixture

Hydrogen chloride gas Ruthenium oxide Not combustible. Ambient fire may liberate hazardous vapours.

#### 5.3 Advice for firefighters

Stay in danger area only with self-contained breathing apparatus. Prevent skin contact by keeping a safe distance or by wearing suitable protective clothing.

#### 5.4 Further information

Suppress (knock down) gases/vapors/mists with a water spray jet. Prevent fire extinguishing water from contaminating surface water or the ground water system.

#### SECTION 6: Accidental release measures

- 6.1 Personal precautions, protective equipment and emergency procedures
   Advice for non-emergency personnel: Avoid inhalation of dusts. Avoid substance contact.
   Ensure adequate ventilation. Evacuate the danger area, observe emergency procedures, consult an expert.
   For personal protection see section 8.
- 6.2 Environmental precautions Do not let product enter drains.
- **6.3 Methods and materials for containment and cleaning up** Cover drains. Collect, bind, and pump off spills. Observe possible material restrictions (see sections 7 and 10). Take up dry. Dispose of properly. Clean up affected area. Avoid generation of dusts.

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#### 6.4 Reference to other sections

For disposal see section 13.

#### SECTION 7: Handling and storage

7.1 Precautions for safe handling For precautions see section 2.2.

#### 7.2 Conditions for safe storage, including any incompatibilities

#### **Storage conditions** Tightly closed. Dry.

nghuy closed. Dry.

Air sensitive. hygroscopic

#### Storage class

Storage class (TRGS 510): 8A: Combustible, corrosive hazardous materials

#### 7.3 Specific end use(s)

Apart from the uses mentioned in section 1.2 no other specific uses are stipulated

#### SECTION 8: Exposure controls/personal protection

#### 8.1 Control parameters

#### **Ingredients with workplace control parameters** Contains no substances with occupational exposure limit values.

#### 8.2 Exposure controls

#### Appropriate engineering controls

Immediately change contaminated clothing. Apply preventive skin protection. Wash hands and face after working with substance.

#### **Personal protective equipment**

#### Eye/face protection

Use equipment for eye protection tested and approved under appropriate government standards such as NIOSH (US) or EN 166(EU). Tightly fitting safety goggles

#### **Skin protection**

Handle with impervious gloves.

This recommendation applies only to the product stated in the safety data sheet, supplied by us and for the designated use. When dissolving in or mixing with other substances and under conditions deviating from those stated in EN 16523-1 please contact the supplier of CE-approved gloves (e.g. KCL GmbH, D-36124 Eichenzell, Internet: www.kcl.de).

Full contact Material: Nitrile rubber Minimum layer thickness: 0.11 mm Break through time: 480 min Material tested:KCL 741 Dermatril® L

Splash contact

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Material: Nitrile rubber Minimum layer thickness: 0.11 mm Break through time: 480 min Material tested:KCL 741 Dermatril® L

#### **Body Protection**

protective clothing

#### **Respiratory protection** Recommended Filter type: Filter type P2

The entrepeneur has to ensure that maintenance, cleaning and testing of respiratory protective devices are carried out according to the instructions of the producer. These measures have to be properly documented.

required when dusts are generated.

Our recommendations on filtering respiratory protection are based on the following standards: DIN EN 143, DIN 14387 and other accompanying standards relating to the used respiratory protection system.

#### **Control of environmental exposure**

Do not let product enter drains.

#### **SECTION 9: Physical and chemical properties**

#### 9.1 Information on basic physical and chemical properties

	-	-
a)	Appearance	Form: solid
b)	Odor	No data available
c)	Odor Threshold	No data available
d)	рН	No data available
e)	Melting point/freezing point	No data available
f)	Initial boiling point and boiling range	No data available
g)	Flash point	()Not applicable
h)	Evaporation rate	No data available
i)	Flammability (solid, gas)	No data available
j)	Upper/lower flammability or explosive limits	No data available
k)	Vapor pressure	No data available
I)	Vapor density	No data available
m)	Density	No data available
	Relative density	No data available
n)	Water solubility	No data available
<b>o)</b> h - 10	Partition coefficient:	No data available

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n-octanol/water

- p) Autoignition No data available temperature
- q) Decomposition No data available temperature
- r) Viscosity No data available
- s) Explosive properties No data available
- t) Oxidizing properties No data available
- 9.2 Other safety information No data available

#### **SECTION 10: Stability and reactivity**

- **10.1 Reactivity** No data available
- **10.2 Chemical stability** The product is chemically stable under standard ambient conditions (room temperature) .
- **10.3 Possibility of hazardous reactions** No data available
- **10.4 Conditions to avoid** no information available
- **10.5 Incompatible materials** Zinc
- **10.6 Hazardous decomposition products** In the event of fire: see section 5

#### **SECTION 11: Toxicological information**

#### 11.1 Information on toxicological effects

#### Acute toxicity

Oral: No data available Inhalation: No data available Dermal: No data available No data available

**Skin corrosion/irritation** Remarks: No data available

Serious eye damage/eye irritation Remarks: No data available

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**Respiratory or skin sensitization** No data available

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Germ cell mutagenicity

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No data available

#### Carcinogenicity

- IARC: No ingredient of this product present at levels greater than or equal to 0.1% is identified as probable, possible or confirmed human carcinogen by IARC.
- NTP: No ingredient of this product present at levels greater than or equal to 0.1% is identified as a known or anticipated carcinogen by NTP.
- OSHA: No component of this product present at levels greater than or equal to 0.1% is on OSHA's list of regulated carcinogens.

#### **Reproductive toxicity**

No data available No data available

#### Specific target organ toxicity - single exposure No data available

**Specific target organ toxicity - repeated exposure** No data available

Aspiration hazard No data available

#### **11.2 Additional Information**

No data available

#### **SECTION 12: Ecological information**

- **12.1 Toxicity** No data available
- 12.2 Persistence and degradability No data available
- **12.3 Bioaccumulative potential** No data available
- **12.4 Mobility in soil** No data available
- **12.5 Results of PBT and vPvB assessment** PBT/vPvB assessment not available as chemical safety assessment not required/not conducted
- **12.6 Endocrine disrupting properties** No data available
- **12.7 Other adverse effects** No data available

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#### SECTION 13: Disposal considerations

#### 13.1 Waste treatment methods

#### Product

Waste material must be disposed of in accordance with the national and local regulations. Leave chemicals in original containers. No mixing with other waste. Handle uncleaned containers like the product itself.

#### **SECTION 14: Transport information**

#### DOT (US)

UN number: 3260 Class: 8 Packing group: II Proper shipping name: Corrosive solid, acidic, inorganic, n.o.s. (Ruthenium(III) chloride trihydrate) Reportable Quantity (RQ): Poison Inhalation Hazard: No

#### IMDG

UN number: 3260 Class: 8 Packing group: II EMS-No: F-A, S-B Proper shipping name: CORROSIVE SOLID, ACIDIC, INORGANIC, N.O.S. (Ruthenium(III) chloride trihydrate)

#### ΙΑΤΑ

UN number: 3260 Class: 8 Packing group: II Proper shipping name: Corrosive solid, acidic, inorganic, n.o.s. (Ruthenium(III) chloride trihydrate)

#### **SECTION 15: Regulatory information**

#### SARA 302 Components

This material does not contain any components with a section 302 EHS TPQ.

#### SARA 313 Components

This material does not contain any chemical components with known CAS numbers that exceed the threshold (De Minimis) reporting levels established by SARA Title III, Section 313.

#### Massachusetts Right To Know Components

No components are subject to the Massachusetts Right to Know Act.

#### **SECTION 16: Other information**

#### **Further information**

The above information is believed to be correct but does not purport to be all inclusive and shall be used only as a guide. The information in this document is based on the present state of our knowledge and is applicable to the product with regard to appropriate safety precautions. It does not represent any guarantee of the properties of the product. Sigma-Aldrich Corporation and its Affiliates shall not be held liable for any Aldrich - 10452

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damage resulting from handling or from contact with the above product. See www.sigma-aldrich.com and/or the reverse side of invoice or packing slip for additional terms and conditions of sale.

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# Section 11 Troubleshooting Chromatographic Interpretations\*

<u>Symptom</u>	Cause	Remedy
No Peaks.	Power supply malfunction	Check fuses.
	Incorrect mode selected on detector controller module.	Depress correct mode select switch.
	Detector flame out.	Ignite detector flame; check hydrogen and/or air flow; check for plugged flame tip.
	No carrier gas flow.	Turn on carrier gas; replace carrier gas cylinder if empty; check for obstructed carrier gas lines.
	Injector septum leaking.	Replace septum.
	Column connections leaking.	Tighten connections.
	Carrier gas connected to wrong injector.	Provide correct carrier gas connection to injector.
	Syringe leaking or plugged.	Replace syringe.
Poor sensitivity with normal retention times	Sensitivity Range too low or attenuation too high.	Select correct sensitivity with electrometer RANGE and/or ATTENUATOR switches.
<b></b>	Insufficient sample size.	Increase sample size.
	Decomposed sample.	Prepare fresh sample.
	Poor injection technique.	Review injection techniques.
	Syringe leaking or plugged.	Replace syringe.
	Carrier gas leaking at injector septum, column fittings, etc.	Locate and correct leak.

\* Taken from "Basic Gas Chromatography" by McNair and E.J. Bonelli (available from GOW-MAC, P/N 145-101).

<u>Symptom</u>	Cause	<u>Remedy</u>
Poor sensitivity with normal retention times (cont)	Carrier gas flow rate incorrect.	Adjust flow rate.
	Hydrogen or air flow rate incorrect.	Adjust flow rates.
	Obstruction in flame tip or incorrect flame-tip orifice.	Call GOW-MAC.
Poor sensitivity with increased retention time	Carrier gas flow rate too low.	Adjust carrier gas flow rate; check for depleted carrier gas supply or obstructed carrier gas lines.
	Carrier gas leaking at injector septum, column, etc.	Locate and correct leak.
	Column temperature too low.	Increase column temperature
Negative peaks.	Recorder leads reversed.	Check recorder connections.
	Detector controller OUTPUT polarity (+/-) switch in wrong position.	Select correct polarity for location of analytical column.
	Sample injected into wrong column.	Inject sample into correct column.
Irregular baseline drift when operating isothermally.	Poor instrument location.	Move instrument to a location where it is not subject to drafts and/or ambient temperature changes
	Instrument not properly grounded.	Make sure instrument and recorder are connected to good earth ground.
	Recorder defective.	Set detector controller ATTENUATOR switch to $(\infty)$ . If drift continues, recorder is defective. See recorder manual.
	Detector base contam- inated.	Call GOW-MAC.

<u>Symptom</u>	Cause	<u>Remedy</u>
Irregular baseline drift when operating isothermally (cont)	Carrier gas leaking at injector septum, column, etc.	Locate and correct leak.
	Column packing bleeding.	Condition column; operate column at a lower temperature; replace column or packing. Some packing materials cannot be operated at elevated temperatures without difficulty. Drifting may occur even on well-conditioned columns in which carrier gas flow rates have been carefully optimized.
	Poor carrier gas regulation	Check carrier gas supply pressure; check carrier gas regulator and flow controller to ensure proper operation.
	Poor hydrogen and/or air regulation.	Check hydrogen and air supplies; check gas regulators and flow controllers to ensure proper operation.
Irregular baseline shifting.	Column not properly conditioned.	Condition column.
	Excessive column bleeding from well-conditioned column.	Use different column. Some packing materials cannot be operated at elevated temperatures without difficulty. This symptom may occur even on well-conditioned columns in which carrier gas flow rates have been carefully optimized.
	Column contaminated.	Recondition column.
Baseline stepping. Baseline does not return to zero, attenuation is incorrect, peaks are flat-topped.	Instrument and/or recorder properly grounded.	Make sure instrument and recorder are connected to good earth ground.
	Recorder gain and/or damping control improperly adjusted.	Adjust recorder gain and/or damping control. Refer to recorder manual.

## Symptom

Baseline cannot be set properly.



Sinusoidal baseline drift.

<u>Cause</u>

Adjustable zero on recorder not properly set.

Recorder improperly connected.

Recorder defective.

Excessive background from column bleed.

Detector-oven temperature controller defective.

Column oven temperature controller defective.

Poor instrument location.

Carrier gas flow regulator defective.

Carrier gas supply pressure too low to allow regulator to control properly.

Detector temperature increasing (decreasing).

Leak in FID base.

Quick atmospheric pressure changes from opening and closing doors, blowers, etc.

Dust particles or other foreign material burned in flame.

Loose column fittings. Electronic circuitry defective.

## <u>Remedy</u>

Set detector controller ATTENUATOR switch to infinity  $(\infty)$  and adjust recorder zero.

Check recorder connections. Remove any connections between recorder inputs and ground or shield.

See recorder manual.

Condition column; use different column.

Replace temperature-sensing probe.

Replace temperature-sensing probe.

Move instrument to a location where it is not subject to drafts and/or ambient temperature changes.

Replace carrier gas flow regulator.

Replace carrier gas cylinder.

Allow sufficient time for detector to restabilize after changing its temperature.

Replace defective base or flame tip.

Relocate instrument to minimize problem. Do not locate under heater or air conditioner blowers.

Keep detector chamber free of dust particles. Blow out or vacuum detector to remove dust.

Tighten column connections. Consult GOW-MAC Engineering

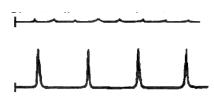
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one direction.

Constant baseline drift in

Sharp spiking at irregular intervals.

## Symptom



### <u>Cause</u>

Condensation in flow lines causing carrier gas to bubble through.

Water condensation in hydrogen line coming from hydrogen generator.

Contaminated column or excessive column bleed.

Contaminated injector.

Contaminated detector.

Water condensing inside flame detector shell.

New flame tip.

Defective recorder.

Recorder slidewire dirty.

Carrier gas flow rate too high.

Air and/or hydrogen flow rates incorrect.

Carrier gas leaking.

Bad ground connection.

Electronic circuitry defective.

#### <u>Remedy</u>

Heat lines to remove condensation while purging with dry gas.

Remove water from line and replace or regenerate filter.

Recondition column.

Clean injector and replace septum.

Call GOW-MAC.

Raise detector temperature to at least 50 °C above column temperature to eliminate condensation.

Bake new flame tip at 350 °C for 8 to 16 hours.

Set detector controller ATTENUATOR switch to infinity  $(\infty)$ . If noise continues, check recorder. See recorder manual.

Clean recorder slidewire.

Reduce carrier gas flow rate.

Adjust flow rates to proper levels.

Locate and correct leak.

Make sure all instruments are connected to good earth ground.

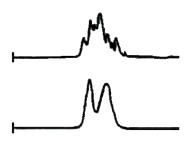
Consult GOW-MAC Engineering

<u>Symptom</u>



Leading peaks

Unresolved peaks too high.



Round-topped peaks



Square-topped peaks



## <u>Cause</u>

Injector temperature too high or too low.

Septum contaminated.

Column-oven temperature too low.

Incorrect column for application. Interaction between sample material and column mobile phase or solid support.

Column overloaded. Sample size too large for column diameter and length.

Sample condensed in system.

Column-oven temperature too high. Column too short.

Mobile phase has baked off of column support material.

Incorrect column for application.

Carrier gas flow rate too high.

Poor injection technique.

Operating beyond linear dynamic range of detector.

Recorder gain too low.

Detector controller output exceeds recorder input range.

Recorder slidewire defective or mechanism binding.

## <u>Remedy</u>

Readjust injector temperature.

Replace septum.

Increase column-oven temperature.

Use different column.

Decrease sample size.

Check that injector and detector settings are correct.

Reduce column-oven temperature. Use longer column.

Replace or repack column.

Consult GOW-MAC for column advice.

Reduce carrier gas flow rate.

Review sample injection techniques.

Reduce sample size.

Adjust recorder gain. See recorder manual.

Normal for solvent peak. Reduce detector controller sensitivity, if desired.

Check recorder operation. See recorder manual.

## <u>Symptom</u>

Extra peaks

Ш

#### <u>Cause</u>

Heavy residual material eluting from previous sample injection (chromatogram I).

Condensed moisture or other impurities from carrier gas eluting during temperatureprogrammed run. (chromatogram I).

Desorption from column packing when solvent is injected (chromatogram II).

Sample decomposition (chromatogram II).

Contaminated sample

Sample interaction with mobile phase or solid support of column packing (chromatograms I & II).

Contamination from glassware, syringes, etc. (chromatograms I & II).

Sample size too large.

Sudden drop-off of otherwise normal peak. Recorder pen returns to level below previous baseline. FID flame becomes extinguished.

Carrier gas flow rate too high.

Flame tip orifice too small.

Flame tip plugged.

supplies.

Loss of hydrogen or air.

Check hydrogen and air

#### <u>Remedy</u>

Allow sufficient time for previous sample to elute.

Install, replace or regenerate carrier gas filter.

Make several solvent injections. and recondition column.

Reduce injector temperature. Use different column if packing material is causing or catalyzing decomposition.

Ensure proper preparation of sample prior to injection.

Use different column. Consult GOW-MAC for column advice.

Make sure glassware, syringes, etc., are clean.

Reduce sample size. Reignite flame.

too high. Adjust carrier gas flow rate.

Call GOW-MAC..

Call GOW-MAC.

Reestablish proper flow rates.

## <u>Symptom</u>

Loss of hydrogen or air. (cont)

## <u>Cause</u>

Sample contains more oxygen than combustion air, causing flashback.

## <u>Remedy</u>

Dilute sample with inert gas, or use oxygen rather than air to support combustion.

# Section 12 Replacement Parts

When ordering replacement parts for your Series 580 FID GC, please specify the serial number of the instrument.

	<u>Description</u>	<u>Part No</u> .
Heaters (115 V)	Column oven heater 500 W Detector oven heater 60 W Injection port heater 60 W	124-183 124-152 124-152
Heaters (230 V)	Column oven heater 500 W Detector oven heater 60 W Injection port heater 60 W	124-184 124-153 124-153
Electronic Modules (115 V)	Temperature controller PCB Display/Switch Interface PCB Display	123-177 123-194 128-274
Electronic Modules (230 V)	Temperature Controller PCB Display/Switch Interface PCB Display	123-177-230 123-194 128-274
Mechanical Assemblies	Injection port assembly FID Detector assembly FID Detector assembly w/ flame-out option Column adapter kit (for one column with 1/8" ends)	069-86 12-580-N 12-580-NF 180-255
Electronic Parts and Controls		
	Zero potentiometer, 20K Receptacle, power switch w/ line filter (115/230 V) Fuse, 10 A (115 V) Fuse, 5 A (230 V) Attenuator board assembly Power cord (115 V) Power cord (230 V) Blower motor kit: motor, wheel, fan blade Neon pilot light Knob Switch, fan Switch, attenuator Switch, range Platinum sensor	111-178 129-152-10A 121-162 121-177 123-175 127-378 127-407 069-91 127-329 127-354 120-169 123-175 120-128 124-175

## SERIES 580 FID GC REPLACEMENT PARTS LIST (cont)

	<u>Description</u>	<u>Part_No.</u>
Miscellaneous	Injection port nut	176-125
	Metering valve Septa, 9 mm silicon	180-1030 180-123S
	Septa, High temperature Feet, rubber self adhesive	180-278S 141-452
	Carrier flowmeter, He 0-65 cc/min @ 40 psig	180-163
	Sample flowmeter, Air 0-130 cc/min @ STP	180-216
	Hydrogen Flowmeter w/valve Air Flowmeter w/valve	180-136 180-138
	Recorder Cable, 6'	141-354
	Rotameter Bank (He, Air, H <sub>2</sub> ) Rotameter Bank (N <sub>2</sub> , Air, H <sub>2</sub> )	75-300-HE 75-300-N2
	Capillary column adapter	180-966-2

# Section 13 Drawings and Schematics

Flow Diagram Wiring Schematic

## Health and Safety Declaration for the Return of GOW-MAC Instrument Co. Equipment

In order to protect our employees from exposure to various hazards, the following statements and/or questions <u>MUST</u> be answered by you. Fill out this document in its entirety and either fax or e-mail it to GOW-MAC Instrument Co., Attn: Repair Dept, **BEFORE** returning the product.

The instrument/part being returned <u>will not</u> be accepted into GOW-MAC's facility until we receive this completed document, along with a <u>PO or Credit Card</u>. Once approved for return by our Chemical Safety Officer, a <u>Return Materials</u> <u>Authorization (RMA) number</u> and shipping instructions will be issued. *All applicable regulations should be followed when returning instrumentation, and/or parts.* 

Model # / Part # \_\_\_\_

Serial #:\_\_

Service Technician spoken to:

Today's Date:

# IF THIS FORM IS NOT APPROVED BY OUR CHEMICAL SAFETY OFFICER, THE INSTRUMENT/PART <u>WILL NOT</u> BE PERMITTED INTO OUR FACILITY FOR SERVICING!

- A] Brief explanation of issue:\_
- B] Briefly list the application(s) for which the instrument/part was used, as well as any and all chemicals, gases, and/or materials analyzed and their concentrations. (Must be filled in):
- C] Is there the possibility of internal or external contamination on or in this instrument/part?
  - $\Box$  Yes see below  $\Box$  No proceed to D.

Please check the appropriate box.

- Chemicals or Substances That Are Hazardous to Health
- Blood, Body Fluids, (e.g. Urine, Secretions), Pathological Specimens
- Regulated Medical Wastes
- □ Infectious Substances or other Bio-Agents (e.g. Protein, Enzymes, Antibodies)
- Radioactive Isotopes used in the area. Detail type (ECD, Isotopic Labels, etc) and Activity in Micro Curies
- Biodegradable Material That Could Become Hazardous
- Other Hazards

If any of the above boxes are checked the following statements and/or questions must be answered.

- 1. Specifically describe where (on or in) the instrument/part there could be any residual contamination (for example: blood spill on the surface).
- Provide details of these hazards. Include names, Material Safety Data Sheets (MSDS), and concentration of contaminants, where
  possible.
- 3. Describe the method of decontamination used. Attach Procedure.
- D] I declare that the above information is true and complete to the best of my knowledge. I acknowledge that any inconsistencies between the condition of the instrument and the statements made on this form will delay the repair process.
   Authorized signature

<b>J</b>			
Name (Printed)		Phone number:	
Company name:		Fax number:	
Shipping address:			
City:	State/Country:	Zip :	
E-mail address:			

BEFORE item can be shipped, fax completed form to: (610) 954-0599 or e-mail it to: repairs@gow-mac.com

For GOW-MAC Use Only:	Signed:	Date/_	<u> </u>
<ul> <li>Passed Safety Inspection. OK to proceed to Repair Dept.</li> <li>Failed safetyInspection. <u>DO NOT</u> proceed to Repair Dept.</li> </ul>	Chemical Safety Officer RMA No:	Comments:	( ) None ( ) On Back >>>>
		REP-005	



Health-Safety Declaration Doc - ONLINE Rev.7 1/28/2022, kj