

Polyarc[®] System v2.0 Installation Manual Agilent 8890, 8860, 7890, 7820, 6890 or equivalent Manual ID: PA-MAN-IM38

Quick Start Guide

Installation

- 1. <u>Shut down GC completely</u>: Turn off oven and detector temperatures and allow to cool; unplug power supply. (page 6)
- 2. Install CO₂ trap on the air supply to your flow controller (do not purify FID air supply). (page 12)
- 3. Plumb 1/8" tubing lines from air and H₂ supplies to Polyarc flow controller.
- 4. <u>Place the Polyarc</u> onto the GC with the capillary lines extending into the oven. (page 7)
- 5. <u>Connect the heater assembly</u> from the Polyarc system to the GC motherboard or external PID controller. (page 8)
- <u>Connect the Polyarc outlet</u> to the FID. (page 9)
 NOTE: Trim at least 0.5 inch of protruding tubing after putting on the 0.8mm graphite ferrule to remove any debris from the graphite ferrule (consult <u>Agilent website</u> tube cutting guide for information on how to properly cut tubing).
- Connect the Polyarc inlet to the capillary column using a zero-dead volume union. NOTE: Consult <u>Agilent website</u> for proper swaging instructions and to ensure the right ferrule is being used. Improper swaging can lead to leaky connections and bad chromatography.
- 8. <u>Connect Polyarc air and H₂ inlets</u> to their respective flow controller outlets. (page 11)
- 9. Power on GC; ensure carrier gas is flowing through column.
- 10. <u>Turn on Polyarc air and H2</u> flows and measure the flows independently out of the FID to confirm 2.5 sccm and 35 sccm, respectively; adjust if necessary. (page 11)
- 11. Configure the Polyarc heater
- 12. Identify the heater type. This will be indicated on the packaging and packing list, but the following can be used as a reference.
 - a. PT-100 RTDs will have a blue heater cable and/or a black Molex connector. They will display the actual temperature at room temperature.
 - b. ARC RTDs will have a tan heater cable and a white Molex connector. There will be an offset in the temperature readout, and you will see a negative readout at room temperature.
- 13. Condition the Polyarc depending at 350°C setpoint for one hour with an ARC RTD and at a 450 °C for two hours with a PT-100 RTD
- 14. Set the Polyarc temperature to the operating temperature using the following settings depending on heater cable:
 - a. PT-100: 450 °C
 - b. ARC RTD: 293 °C

Operation

- 1. Always turn the column carrier gas and air & hydrogen supplies on before heating the Polyarc.
- 2. Double check and leak test all connections.
- 3. Ensure the Polyarc is operating with gas flow rates of 2.5 sccm air and 35 sccm H₂. (pg 14)
- 4. <u>Configure GC methods</u> with aux temperature (293 °C for ARC RTD, 450 °C for PT-100 RTD) and FID H₂ flow rate to 1.5sccm. (page 14)
 - a. NOTE: if using hydrogen as a carrier gas, see important note in the Appendix.
 - b. Limit the on column injection amount to 0.1uL (i.e., 1 uL volume 10:1 split or lower).
- 5. Run your method. Avoid injecting more than 1,000 ppm sulfur and large amounts of silicon containing compounds such as BSTFA or TMS.

Shut Down or GC Maintenance

- 1. Shut off the FID.
- 2. Cool the reactor to room temperature (turn off the auxiliary temperature).
- 3. Shut off the air and H_2 flows to the reactor.
- 4. Perform maintenance or shut down GC.

Safety Notices

Throughout this manual, different signal words are used to indicate hazards during the installation process of a Polyarc[®] system on an Agilent GC. Failure to follow the safety messages accompanying the signal words can result in damage to the Polyarc system and GC systems, injury, or death.

ADANGER DANGER indicates a hazardous situation which, if not avoided, will result in death or serious injury.

AWARNING WARNING indicates a hazardous situation that, if not avoided, could result in death or serious injury.

ACAUTION CAUTION indicates a hazardous situation that, if not avoided, could result in minor or moderate injury.

NOTICE

NOTICE is used to address practices not related to physical injury.

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Introduction

This manual will guide you through the Polyarc[®] system installation process into an Agilent 8890, 8860, 7890, 7820, or 6890 GC. The installation is estimated to take two hours to complete (not including initial catalyst treatment).

EXAMPLING This manual is intended for use by those familiar with GC operation and installations, as well as those familiar with and competent using compressed gases including He, H₂, and air. Do not attempt installation if you are uncomfortable. Comply with all local and national safety requirements and be aware of the safety systems and procedures for the facility in which you are installing the system. Wear gloves and appropriate personal protection equipment.

Required Tools

- 1. T-15 and T-20 Torx drivers
- 2. 2 x 5/16" wrench
- 3. 2 x 1/4" wrench
- 4. 2 x 7/16" wrench
- 5. 3/8" wrench
- 6. 1.8 mm slotted screwdriver
- 7. Volumetric flowmeter (bubble-type or digital)
- 8. Leak detector (do not use liquids such as soapy water on GC components)
- 9. Ceramic column cutting tool
- 10. OPTIONAL: Column pre-swaging tool (e.g., pre-swaging wrench or pre-swaging nut; metal ferrules can also be tightened on the fitting without pre-swaging; recommend Agilent part G3440-80227)





Figure 2. (A). Polyarc[®] system installed in back detector position of Agilent 7890. (B). Polyarc[®] system installed, view from inside oven.

Part I – Gas Chromatograph Preparation

DANGER Hydrogen (H₂) is a flammable, colorless, odorless, compressed gas. It poses an immediate fire and explosive hazard when concentrations exceed 4% in air. Higher concentrations can also cause sufficient on any application using H_2 , turn off the supply at its source before working on the instrument and purge the lines to remove H₂. Failure to follow these precautions could result in explosion, fire and/or suffocation.

ACAUTION

Compressed air poses a pressure hazard. The high pressure gas coming out could rapidly pressurize an enclosed space or send particles rapidly out the tube. Turn off air at the source and depressurize the lines using an appropriate relief valve.

NOTICE

Column bleed from silicon containing columns (e.g., polydimethylsiloxane, or PDMS, and 5% Phenyl columns) can cause irreversible deposition of silica in the reactor leading to poor peak shapes and inaccurate peak quantification. High temperature, air/water leaks, old columns and large film thicknesses can all contribute to excessive column bleed rates. For the best performance operate all silicon-containing columns below 250C and maintain a leak free system or avoid silicon-containing columns altogether.

Prepare your Agilent 8890, 8860, 7890, 7820, or 6890 (if you are using an Agilent 5890 or any other manufacturer's GC please contact ARC prior to installation) gas chromatograph (GC) using the steps below.

- 1. Shut-down the GC and unplug the power cable. Allow all detectors, inlets and the oven to cool completely.
- 2. Detach the side panel on the detector side of the GC.
- 3. Detach the top covers of the GC.
- 4. Turn off the air supply to the GC and vent/depressurize the line (gas supply must be free of CO₂).
- 5. Turn off the H₂ supply to the GC and vent/depressurize the line.
- 6. The Polyarc flow controllers should be placed in a relatively isothermal area that is safe and level. We recommend setting on the top right or side of the GC.
- 7. Attach air and H_2 supply lines to Polyarc flow controller. For the manual or electronic flow controllers provided by ARC, attach copper lines to 1/8" Swagelok compression fittings by using supplied nuts and back/front ferrules, tightening as appropriate (3/4 turns past finger tight).

NOTE: Only use regulated and pure gas supplies. Supported supply pressures are indicated on flow controller inlets (60-80 psi is advisable). Fluctuations in the pressure of the gas supply will cause fluctuations in the flow rates of gases to the Polyarc system and could damage the catalyst. Activated Research Company is not responsible for damage to the Polyarc system from fluctuations due to negligent use.

8. Reconnect any tubes that were disconnected and make sure the shut-off valves are in the off position.

Part II – Location of the Polyarc[®] System

The Polyarc system is designed to be in a variety of locations on the GC depending upon the specifics of each GC configuration. The location needs to house the enclosure, which can reach

temperatures of 100 °C, and allow for the Polyarc system inlet/outlet tubes to connect into the GC oven.

The Polyarc system is optimally placed in the back (Figure 2) or front detector positions. Placement of the system elsewhere result in sub-optimal separation mav performance (such as peak broadening and/or tailing) if the transfer lines become cold enough to cause condensation or large interactions with eluents. If the two detector positions are full please contact us at contact@activatedresearch.com for other options.

For the remainder of the installation procedure, we will assume the Polyarc system is located in an otherwiseunused back detector position of an Agilent 7890.

Note: Do not place the electronic flow controller (EFC) on top or near the FID electrometer (see Figure 2)! This can lead to RF interference with the detector leading to a noisy baseline

- Clear a space for the system enclosure in the back detector opening (or other selected location). This may require that the aluminum cap is removed and the insulation cleared out. Be sure to clear out enough insulation to make room for the transfer lines (the final shape should look like a keyhole; see Figure 4 below).
- 2. Feed the two capillary transfer tubes into the GC while inserting the system enclosure in the back detector opening (or other selected location). Be careful not to bend or score the capillary tubes (they are flexible, but tight bends can destroy the tube).



Figure 2. Improperly placed EFC



Figure 4. Keyhole shape within detector position for transfer lines of Polyarc[®] system.

- Secure the Polyarc with the supplied enclosure screws to attach the Polyarc enclosure to the top of the GC oven.
- 4. Connect the heater assembly (4-pin Molex connector) from the Polyarc system to the back detector port as indicated in Figure 5 located on the detector side of the GC (behind the plastic panel). If the Polyarc is installed in an inlet position, use the inlet heater port located on the inlet side of the CC. If installing the Polyar



Figure 5. Heater connection to back detector port on Agilent 7890 GC (electronics located behind plastic panel on detector side of GC).

GC. If installing the Polyarc in a valve box or MS position, refer to Step 5 for instructions to connect to an auxiliary heater cable assembly.

- 5. If using an Agilent 6890 GC or if performing an installation on an Agilent 7890 GC in a position other than an inlet or detector position, follow this step for connection of the heater cable to a heater cable assembly. Otherwise, proceed to Step 6. Check to see that an auxiliary heater cable assembly (shown in Figure 6) is connected to the GC power board on the detector side of the GC. If the assembly is not present, install a cable assembly (specified during order; Figure 6). Feed heater assembly through side of GC down to its existing cable assembly. Perform step 'a' or 'b' below depending on cable assembly configuration.
 - a. <u>Using Existing Cable Assembly:</u> Connect the heater assembly (4-pin Molex connector) from the system to the cable assembly in the first (A1) or last (A2) position as indicated in Figure 6 located on the detector side of the GC (behind the plastic panel). Record its position (A1/A2).



Figure 6. Heater connection to A1 port on assembly cable of Agilent 6890 GC (electronics located behind plastic panel on detector side of GC).

b. Using Cable Assembly Supplied in Kit: Connect the heater assembly to the cable assembly and the cable assembly to the main power board (Figure 7)



Figure 7. Heater assembly connection with ARC cable assembly on Agilent 7890 GC (electronics located behind plastic panel on detector side of GC).

6. Close and fasten the GC side cover.

Part III – Polyarc[®] System Gas Connections

The following steps connect the GC column and FID to the Polyarc system.

NOTE: Procedure is the same for both capillary optimized and adaptable FID inlets.

NOTICE Use gloves to avoid contamination of the column

1. Connect the larger ID metal capillary tube (the outlet of the Polyarc system) to the FID following the steps below.



a. Remove the existing capillary column from the FID and cut the column to remove the nut to reuse it for the next step.

b. Place the larger metal capillary tube from the system through the FID nut (removed above) and through the included 0.8 mm graphite ferrule.

c. Trim 0.5" of the tubing using a ceramic scribe to remove any debris which may have accumulated during shipping and installation. Information about how to cut metal capillary tubing can be found online (<u>http://www.agilent.com/en-us/video/properlycuttubing</u>).

d. Insert the column up into the flame detector jet (you may need to gently move the tube around to find the jet hole) until it stops, then pull the column back out ~1-2 mm. Tighten the nut and ferrule onto the FID fitting by turning the nut about 90° past finger tight to secure the column (Figure 8).

NOTE: If you are having problems finding the jet hole and you have an adaptable FID inlet, try removing the 1/4" fitting.

Figure 8. Outlet of the Polyarc[®] system inserted into the FID jet.

 Connect the smaller ID metal capillary tube (the inlet to the Polyarc system) to the outlet of the capillary column using a zero-dead volume union by following the steps below. More detailed instructions can be found on the Agilent website: <u>https://www.agilent.com/cs/library/usermanuals/public/G3182-90580.pdf</u>.

ltem	Agilent Part No.	Ferrule Description	Degrees to tighten internal nut
1	G3188-27501	0.1 – 0.25 mm column ID	50-100
2	G3188-27502	0.32 mm column ID	30-70
3	G3188-27503	0.45 – 0.53 mm column ID	20-50
4	G3188-27504	Plug	60
5	G3188-27505	0.25 – 0.32 mm UltiMetal column ID	40-90
6	G3188-27506	0.53 mm UltiMetal column ID	20-50
			6

Table 1: Agilent Ferrules

- a. The smaller ID metal capillary tube should have a column nut and ferrule attached to a zero-dead volume union (Agilent ferrule Part No. G318827505). If these are not attached swage the ferrule onto the column following the procedure below (Steps c-f). Then tighten the nut and ferrule onto the zerodead volume union by tightening the nut 5-10° past finger tight (Figure 9b).
- b. Pass the GC capillary column (supplied by customer) through the supplied column nut, metal ferrule (with correct column I.D., Figure 9a) and swaging tool (wrench or nut). The union can be used as a swaging tool if no swaging tool is available. After swaging, the column must protrude 0.1-0.5 mm from the ferrule, or leaks and column breaks can occur.
- c. Tighten the column nut on the tool 10-20° swaging in increments using a wrench until the column just begins to grip. Overtightening can cause leaks and potential damage to the column. Remove the swaging tool.



Figure 9. Inlet of the Polyarc[®] system connected to GC capillary column. (a) From left to right: column nut, capillary metal ferrule for GC column, zero-dead volume union. (b) Completed connection of components.

- d. If the column does not extend 0.1-0.5 mm from the ferrule, trim the excess column using a cutting tool leaving 0.3 mm of column extending from the ferrule. Make sure no cracks extend into the ferrule or leaks may occur.
- e. Connect the column to the union and tighten 5-10° past finger tight (Figure 9b).

Part IV – Connecting Gas Flows to Polyarc[®] System

A DANGER Hydrogen (H₂) is a flammable, colorless, odorless, compressed gas. It poses an immediate fire and explosive hazard when concentrations exceed 4% in air. Higher concentrations can also cause suffocation. In any application using H₂, turn off the supply at its source before working on the instrument and purge the lines to remove H₂. Failure to follow these precautions could result in explosion, fire and/or suffocation.

ACAUTION

Compressed air poses a pressure hazard. The high-pressure gas coming out could rapidly pressurize an enclosed space or send particles rapidly out the tube. Turn off air at the source and depressurize the lines using an appropriate relief valve.

NOTICE Always measure air and H₂ flow rates from the FID outlet while the Polyarc system is at room temperature for accurate readings. Flow rates measured while the Polyarc system is warm are incorrect due to the reaction of oxygen and hydrogen to form water.

The following instructions can be used to connect and check the flow rates of air and H_2 to the Polyarc system. The electronic flow module supplied by ARC does not require any calibration.

- 1. Install a CO_2 trap on the air supply line to your flow controller. Split the air supply line so that one side goes to the FID and the other to the CO_2 trap and Polyarc system. This will prolong the life of the trap.
- 2. Measure and cut appropriate lengths of the supplied 1/16" OD tubing to connect your chosen flow controller to the Polyarc system for air and H₂ gases. The tubing can be routed through the back of the GC under the back cover.
- 3. Connect the 1/16" tubing to the flow controller 'air outlet' using two wrenches to tighten the nut without torqueing on the body of the flow controller. Connect the other side of the 1/16" tubing to the supplied 1/16" union.
- 4. Repeat Step 2 for H₂.
- 5. Turn on the H₂ at the source and adjust the flow rate to 35 (\pm 1.0) (see Adjusting Gas Flow Rates) as measured through the FID outlet. Check all fittings for leaks and adjust as needed. NOTE: If you are using hydrogen as a carrier gas, see important note with modified hydrogen flow rates in the Appendix.
- 6. Turn off the H₂ and turn on the air at the source and adjust the flow rate to 2.5 (\pm 0.5) sccm (see Adjusting Gas Flow Rates). NOTE: If you are using hydrogen as a carrier gas, see important note with modified air flow rate in the Appendix. Check all fittings for leaks and adjust as needed. NOTE: Ensure the Polyarc system is not heated for this step as the flow rate will be inaccurate.

Note: Do not place the electronic flow controller (EFC) on top or near the FID electrometer (see Figure 2)! This can lead to RF interference with the detector leading to a noisy baseline.

Part V – Final Configuration

Heater Configuration

- 1. Plug the GC into the wall outlet. Turn on the GC.
- 2. Turn configuration lock 'Off' under the following menu: Options/Keyboard & Display/Hard Configuration Lock.
- 3. Use Option 'a' or 'b' below depending on GC type

- a. For an Agilent 7890/7820, restart the GC and then;
 - i. Install cartridge heater/sensor as a 'Heater' in Thermal Aux 1, 2, or 3 (see step iii to determine Thermal Aux Number).
 - ii. Scroll to Config/Aux Temp#/
 - iii. Use the following table do determine which Thermal Aux number to install the Polyarc heater cartridge:

Heater Installation Location	Thermal Aux Number
Auxiliary Heater 1 (A1)	Thermal Aux 1
Auxiliary Heater 2 (A2)	Thermal Aux 2
Back Detector	Thermal Aux 2ª
Front Detector	Thermal Aux 3
Front Inlet	Thermal Aux 3
Back Inlet	Thermal Aux 1 ^b

^aIf there is currently a heater connected to A2 it will need to be reconfigured as Thermal Aux 3.

^bIf there is currently a heater connected to A1 it will need to be reconfigured as Thermal Aux 3.

- iv. Select the Thermal Aux number determined above.
- v. Text should say 'Unconfigured'
- vi. Press 'Mode/Type'
- vii. Install as 'Heater A1' or 'Heater A2', if the heater is installed in the auxiliary heater zone or 'Heater B-DET' if the heater is installed in the back detector position, by pressing enter over your selection. If the heater is installed in a different detector position, or an inlet position, select the appropriate option.
- viii. If using a PT-100 Polyarc heater (black connector on heater cable), proceed as follows:
 - 1. Once the heater has been configured, press [Config], then select your aux temp #, and press [Enter]
 - 2. Select Auxiliary Type: Unknown and press [Mode/Type]
 - 3. Select User Configurable Heater and press [Enter]
 - 4. Reboot GC by pressing [Options], selecting Communications, and selecting Reboot GC
 - 5. Press [Config] and select the thermal auxiliary being used
 - Scroll down to Maximum Setpoint, and update the value to 450 °C
 - 7. Reboot GC as in step 3.
- b. For an Agilent 6890 (method may vary depending on GC model; the goal is to configure the heater assembly into the appropriate auxiliary heater port as some recognizable heater, e.g., the MSD):
 - i. Install cartridge heater/sensor as a 'Heater' in A1 or A2 (determined in Part II)
 - ii. Scroll to Config/Aux Temp#/
 - iii. Select 1 or 2 to install as A1 or A2
 - iv. Under aux type, select 'MSD' using the Mode/Type key
- 4. Check that the Aux temperature reading is stable (it will be around -35 °C if at room temperature when using an ARC RTD; the offset is normal).

Clean-up

ACAUTION Insulation contains particles that are hazardous to your lungs and eyes. Avoid breathing insulation dust and contact with eyes. Wear long sleeves, gloves, safety glasses, and a disposable dust/mist respirator. Clean up insulation in the area. Dispose of insulation in a sealed plastic bag.

- 1. Clean up air and H₂ tubing by coiling or bending into its final location.
- 2. Clean up any insulation particles and dispose in a sealed plastic bag.

Test Fittings

- 1. Ensure all gas flows are on.
- 2. Using a leak detector, check all fittings for leaks.
- 3. Heat oven to 20 °C below maximum and hold for 10 minutes, cool GC down.
- 4. Recheck fittings for leaks and tighten as needed.

Initial Catalyst and GC Treatment

- 1. If not already completed, check and adjust (see "Adjusting Gas Flow Rates" in Appendix) the air and H₂ flow rates through the Polyarc system while the reactor is at room temperature. Never measure flow rates with the reactor warm and always measure from the FID outlet to obtain the proper pressure driven flow values. An electronic flow control module can be purchased from ARC to eliminate this flow calibration.
- 2. The Polyarc system requires an initial conditioning to prepare the catalyst and remove contaminants absorbed during shipping and installation. Leave the air supply and the H₂ supply on.
- 3. <u>Turn the column carrier gas on before heating the Polyarc system.</u>

NOTICE DO NOT heat the reactor without the carrier gas flowing through the inlet capillary of the Polyarc system. The reactor must be first cooled before disconnecting the column or before turning off the carrier gas supply.

- 4. Condition the Polyarc with the proper temperature setpoint according to the following instructions:
 - a. If your Polyarc is fitted with an ARC RTD (indicated by a tan heater cable and white Molex connector), condition the Polyarc at a 350 °C setpoint for one hour.
 - b. If your Polyarc is fitted with a PT-100 RTD (indicated by a blue heater cable and/or a black Molex connector), condition the Polyarc at a 450 °C setpoint for at least two hours.

NOTE: The heater has been specially engineered to ensure the Polyarc system runs at the ideal temperature to maximize analyte conversion. Please contact ARC if there are any questions about which heater settings should be used.

CAUTION DO NOT increase heater temperature beyond 350 °C with an ARC RTD (525 °C with PT-100 heater). Higher temperatures could damage the equipment and/or lead to fire.

- 5. Set GC oven temperature to 20 °C below max column/oven temperature.
- 6. Allow reactor to remain heated under these conditions for 1-2 hours, or until the baseline stabilizes.

- 7. If using an ARC RTD, reduce aux heater temperature setting to 293 °C and wait 30 minutes after temperature has reached 293 °C. Note: When using an ARC RTD, the 293 °C setpoint is not the actual temperature. The actual temperature of the Polyarc is 450 °C when heated to a setpoint of 293 °C. Use the actual temperature as a setpoint if using a PT-100 RTD (identified with a black connector and/or blue heater cable).
- 8. If the baseline is still decreasing, this procedure can be repeated. This may indicate oils on the fittings or column.

Method Configuration and Testing

NOTICE The Polyarc system requires minimal changes to your methods, such as adding the thermal auxiliary temperature and decreasing H_2 flow rates to the FID to compensate for the addition of H_2 to the Polyarc feed stream. Follow these guidelines:

- 1. Change the Auxiliary temperature setting to 293 °C (450 °C for PT-100 RTD) for all methods.
- 2. Reduce the H_2 flow rate to the FID to 1.5 sccm (most of the H_2 is now supplied by the Polyarc system). In some cases, this H_2 flow rate may need to be higher than 1.5 sccm.
- 3. Change the FID air flow rate to 350 sccm (this can be adjusted slightly to tune the FID for optimal performance). **NOTE: If you are using hydrogen as a carrier gas, see important note about FID air flow rate in the Appendix**.
- 4. Please note that the oven temperature should be kept above 30 °C when using standard Polyarc air and hydrogen flow rates, to prevent condensation in the Polyarc outlet and extinguishment of the FID flame. **NOTE: If you are using hydrogen as a carrier gas, see important note about minimum oven temperature in the Appendix**.
- 5. The column inlet pressure (flow rate) may need to be increased slightly to account for the slight increase in pressure drop due to the presence of the micro-channel reactor. An increase of ~10% is advised, but larger increases may be required for flow rates above 2 sccm.
- 6. Test the Polyarc system with a well characterized mixture such as the supplied Polyarc Test Mix. Use our one of our online calculators to determine the concentrations of components in the mixture to validate Polyarc system performance.
- Compounds containing sulfur at a concentration higher than 1,000 ppm can quickly contribute to catalyst poisoning, so limit the amount on column to below that concentration and/or below a 1 μL,10:1 split injection.
- 8. Compounds that contain silicon (e.g., BSTFA, TMS) lead to irreversible deposition of silica in the combustion chamber of the reactor resulting in peak tailing and broadening over time. In large amounts, single injections can lead to peak tailing, which is especially noticeable with sticky (those that bind strongly to silanols) molecules such as amines.

Part VI – Polyarc Replacement

Users typically replace the Polyarc every six months or more, depending on the analysis. When a replacement Polyarc is purchased, ARC will send a new unit to replace the existing one, and the replacement procedure is as follows:

- 1. Cool the existing Polyarc and FID to room temperature.
- 2. Once cool, disconnect the two Swagelok supply lines for air and hydrogen from the Polyarc, leaving the union connected to the lines for use with the next Polyarc.
- 3. Disconnect the Polyarc inlet transfer line from the Agilent Ultimate union. Keep the analytical column connected to this union.
- 4. Disconnect the Polyarc from the FID. The FID column nut and graphite ferrule can be re-used.
- 5. Disconnect the Polyarc heater cable from the GC or ARC Temperature Controller.
- 6. Carefully remove the Polyarc from its position on the GC.
- 7. Unpack the replacement reactor from its box and carefully place it onto the GC, taking care to avoid crimping or bending the transfer lines as they're guided into the GC.
- 8. Connect the air and hydrogen supply lines using the Swagelok unions. Ensure the proper gases are connected to the proper lines.
- 9. Connect the Polyarc inlet transfer line to the Agilent Ultimate union. This line will already have a nut and ferrule swaged on.
- 10. Connect the Polyarc outlet to the FID, re-using the existing 0.8mm graphite ferrule if possible.
- 11. Connect the Polyarc heater cable to the GC or ARC Temperature Controller.
- 12. Heat the Polyarc. The Polyarc may need to be conditioned for an hour to remove any residual buildup from shipping.

Thanks and Feedback

Thank you for your purchase and congratulations on your successful installation of the Polyarc system for quantitative carbon detection. The Polyarc system will eliminate standard time consuming and costly calibrations while enabling you to analyze your compounds with greater accuracy and ease than ever before. We are constantly demonstrating new uses for the Polyarc system and would love to hear how the Polyarc system has helped you. Please email any testimonials, technical literature or examples to <u>contact@activatedresearch.com</u> and we will include them on our website.

We at the Activated Research Company strive to bring you the best products with the ultimate customer experience. If you have any questions which aren't answered in the FAQ, troubleshooting or learning center sections of our website (<u>www.activatedresearch.com</u>), please contact us at <u>contact@activatedresearch.com</u> or call 612-787-2721.

<u>Appendix</u>

Using Hydrogen as a Carrier Gas

Because hydrogen reacts with air in the Polyarc system, the flows supplied to the Polyarc system must be modified from the typical operating flows when using hydrogen as a carrier gas. The following flow rates have been optimized for use with hydrogen as a carrier gas:

1. For hydrogen carrier gas flow rates up to 1.05 sccm:

- a. Set the Polyarc system air flow rate to 5.0 sccm.
- b. Set the Polyarc system hydrogen flow rate to 40 sccm.
- c. Set the FID air flow rate to 400 sccm.
- d. Note: The minimum oven temperature that should be used with these conditions is 40 °C. Operating at a temperature below this recommendation will lead to condensation of water in the Polyarc system outlet, and extinguishment of the FID flame. Contact ARC to obtain a cryo assembly if you are interested in operating at a lower oven temperature.

2. For hydrogen carrier gas flow rates from 1.05 to 2.1 sccm:

- a. Set the Polyarc system air flow rate to 7.5 sccm.
- b. Set the Polyarc system hydrogen flow rate to 40 sccm.
- c. Set the FID air flow rate to 400 sccm.
- d. Note: The minimum oven temperature that should be used with these conditions is 50 °C. Operating at a temperature below this recommendation will lead to condensation of water in the Polyarc system outlet, and extinguishment of the FID flame. Contact ARC to obtain a cryo assembly if you are interested in operating at a lower oven temperature.

3. For hydrogen carrier gas flow rates above 2.1 sccm, contact ARC.

Adjusting Gas Flow Rates

1. Manual Flow Control Module

The manual flow control module consists of two pressure regulators (bottom; Figure A1), one each for hydrogen and air, which supply gas at a constant pressure to two needle valves (top; Figure A1). The regulators are pre-set at the factory and should not need to be adjusted. If adjustment is necessary, the pressure supply to the needle valves can be increased/decreased by turning the respective regulator knob clockwise/counterclockwise. The needle valves are responsible for controlling the flow rate after a constant supply pressure has been set by the regulators. The needle valves have a knob to turn the flows off and on, and an inset screw for adjusting the flow rate. The knob can be turned clockwise to turn off gas flows. The knob should be completely open when adjusting flows and operating the flow controller. The inset increase/decrease screw is used to the flow turning rate by counterclockwise/clockwise.

The procedure for adjusting flows includes turning the valve knob counterclockwise completely (full open) and adjusting the inset valve screw until the desired flow rate is obtained. The flow controller is designed to be operated continuously without interference, but small adjustments may be necessary over time to correct for drift.



The pressure regulators mitigate changes in upstream gas pressures that could affect flows.

There are two options for checking the flow rates on the manual flow control option; a flow meter can be used for both options. The first option (recommended) is checking the flow rates out of the FID; it is important to note that the reactor must be cooled down, otherwise, water will be formed, and consistent flow rates won't be obtained. The second option, if cooling the reactor isn't possible, is to disconnect the air and hydrogen fittings and measure the flow rates from the manual flow control module itself; this option is not as accurate as the first because it does not account for the pressure drop when the system is connected.



Figure A1. Schematic of manual flow control module.

2. Electronic Flow Control Module

NOTE: The flow rates on the electronic flow control module are pre-set at the factory. The supplied Electronic Flow Control Communication Package (USB cable and software) can optionally be used to monitor flows, tare the device, and adjust flow rates (e.g., based on the carrier gas). The device should be tared every 6 months to correct for electrical drift. Instructions are included in the software manual.

The electronic flow control module consists of two mass flow controllers in a convenient enclosure (Figure A2 & A3). The enclosure is equipped with a power plug, a communication port and two on/off switches in addition to the supply and outlet fittings. Only use the supplied power plug (Figure A4). To operate the controllers, connect the appropriate air and hydrogen gases at supply pressures ideally between 60-100 psig. Plug in the device and turn the switches in the back from off (o) to on (|).

Flow can be turned off or on at any time as required for the operation of the Polyarc System. No calibration is necessary as the controllers are pre-set at the factory to the appropriate air and hydrogen flow rates for the Polyarc system. However, **if switching from an inert carrier gas to hydrogen**, **or vice versa**, **see the important note in the Appendix** regarding the recommended flows when using hydrogen as a carrier gas.



Figure A2. Schematic of front of electronic flow control module.



Figure A2. Schematic of back of electronic flow control module.



Figure A3. Schematic of EFC software communication cable.



Figure A4. Schematic of supplied power plug for electronic flow control module.

3. Calibrating the Agilent Auxiliary Electronic Pressure Control (EPC) **NOTE**: When measuring these flow rates, ensure that the Polyarc reactor is cold. Otherwise, water will be formed and accurate flow rates won't be obtained.

First, connect the hydrogen and air to separate auxiliary channels on the EPC; the hydrogen should use a medium-flow frit and the air should use a low-flow frit (frits can be purchased from Agilent). The exact pressure setting required for these flow rates can vary from system to system, however, most users have found that around 26.5 psi and 20.8 psi work for air and hydrogen, respectively. To measure the flow rates, with all fittings of the Polyarc reactor connected, connect a flow meter to the outlet of the FID. Start the hydrogen flow and measure the flow out of the FID, adjusting the pressure to obtain 35 sccm. Turn off hydrogen, start the air flow, and measure the flow out of the FID to obtain 2.5 sccm. Re-start the hydrogen flow to the set pressure. **NOTE: If you are using hydrogen as a carrier gas, see important note with modified air and hydrogen flow rates in the Appendix**.