Operating Manual

Model 75-810 & 75-810-AR Noble Gas Purifier

75-810-HE: 120 V, 50/60 Hz 75-812-HE: 230 V, 50/60 Hz

75-810-AR: 120 V, 50/60 Hz 75-812-AR: 230 V, 50/60 Hz

> July 2023 Rev. 2

READ INSTRUCTIONS BEFORE OPERATING



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Models Affected

75	-81	0-	HE	
75	-81	2-	HE	
75	-81	0-	AF	2
75	-81	2-	AF	2

Warranty

THE MODEL 75-810 NOBLE GAS PURIFIER SOLD BY GOW-MAC[®] INSTRUMENT CO. IS WARRANTED FOR A PERIOD OF ONE (1) YEAR AGAINST DEFECTS IN MATERIALS AND WORKMANSHIP. THE TERMS OF THIS WARRANTY ARE AS FOLLOWS:

- 1. The warranty period begins with the shipping date of the equipment to the original purchaser.
- 2. Certain parts such as batteries, fuses, glass accessories, septa, columns, etc., are expendable in normal use, and their service life is unpredictable. Such items are not covered by this warranty.
- 3. Filaments of thermal conductivity detectors are not covered by this warranty.
- 4. Hydrogen Palladium Tubes are not covered by this warranty.
- 5. All requests for service or repair under this warranty must be received within the warranty period by GOW-MAC® or its authorized representative. All repairs are made at GOW-MAC plants or at the office of authorized representatives.
- 6. All repairs, adjustments, and other services under this warranty shall be performed free of charge to the purchaser. However, warranty service and repairs shall be limited to equipment malfunctions which, in the opinion of GOW-MAC®, are due or traceable to defects in original materials or workmanship. Instrument malfunctions caused by abuse or neglect of the equipment are expressly not covered by this warranty.
- 7. Instrument parts which have been repaired or replaced during the warranty period are themselves warranted only for the remaining unexpired portion of the original one-year warranty.
- 8. Repairs, adjustments, and service performed after expiration of the one-year warranty period shall be charged to the purchaser at the then current prices for parts, labor, and transportation.
- 9. This warranty attaches to the equipment itself and is not limited to the original purchaser. Unexpired portions of the warranty are thus transferable to subsequent owners.
- 10. GOW-MAC® expressly disclaims any liability to users of its products for consequential damages of any kind arising out of or connected with the use of its products.
- 11. Except as stated in Sections 1 through 8 above, GOW-MAC® makes no warranty, expressed or implied (either in fact or by operation of law), statutory or otherwise; and, except as stated in Sections 1 through 8 above, GOW-MAC® shall have no liability under any warranty, expressed or implied (either in fact or by operation of law), statutory or otherwise.
- 12. Statements made by any person, including representatives of GOW-MAC® which are inconsistent or in conflict with the terms of this warranty shall not be binding upon GOW-MAC® unless reduced to writing and approved by an officer of the Company.
- 13. This warranty shall be governed by the laws of the Commonwealth of Pennsylvania.

Noble Gas Purifier Models 75-810-HE/75-812-HE & 75-810-AR/75-812-AR

1 Introduction

The 75-810 series of purifiers are designed for the purification of noble gas and under no circumstances shall they be utilized for the purification of hydrogen, nitrogen, oxygen, air or other reactive gases. Please read and understand the following manual and documents prior to beginning the installation of the 75-810. If there is anything not understood, please call for clarification prior to operation. 75-810-HE/AR designates models for 120 VAC service and 75-812-HE/AR for 230 VAC service. All versions may be generically referred to as 75-810 throughout this manual where appropriate.

The GOW-MAC 75-810 series are point-of-use gas purifiers that remove impurities (H2O, O2, CO, CO2, H2, N2, CH4) from ultra-high purity (99.999%) argon or helium. The bench-top configuration is compact with $\frac{1}{4}$ inch VCR connections for gas in and out on the top panel. The getter material is heated to about 400 °C when the power switch is ON and illuminated. The heated cartridge is wrapped in insulation that is easily removed for service. A second stage getter for hydrogen removed is directly downstream from the first stage. The hydrogen trapping is more efficient when colder so that section of the cartridge is wrapped in a heatsink to transfer heat away from the cartridge. The final feature of the flow path is an integral 0.003 micron particle filter. The 75-810 purifiers are factory-conditioned for optimal performance with helium or argon (denoted in the part number by –HE and –AR).

UltraPure[™] is a registered trademark of UltraPure Systems Inc.

2 Safety Precautions



WARNING

This purifier is intended for the purification of NOBLE* gas only. Under NO circumstances should it be used for the purification of hydrogen, nitrogen, oxygen, air, or reactive gases. Failure to comply can result in personal injury and/or mechanical damage to purifier and all warranties shall be void. *Noble gas refers to Ar, He, Ne, Kr, Xe.

WARNING

This equipment processes gas using electrical equipment. Serious injury can result if maintenance is performed with the power on. Always shut power off at the facility circuit breaker before maintenance is to be performed. In addition, lock out the associated circuit breaker in accordance with local safety directives to prevent an advertent operation by facility personnel until maintenance has been completed and all personnel are clear of the machinery.

WARNING

This equipment processes gas at high temperatures. Serious burns can result. If maintenance is to be performed, allow unit to cool to room temperature prior to any maintenance or service.

WARNING

Electric shock hazard potential exists inside the unit. Take the necessary safey precautions to avoid touching exposed terminals and wiring. Failure to comply can result in death, personal injury or damage to the purifier.

WARNING MAXIMUM VESSEL OPERATING PRESSURE: 250 PSIG

SAFETY CAUTIONS

CAUTION

All gas lines and connections MUST be leak-checked prior to operation of the unit in order to ensure the integrity of the system and to prevent any exposure of the getter bed to atmosphere. The getter material in the purifier may be destroyed if exposed to atmosphere while its temperature is above 150 °C.

CAUTION

If a gas line should become disconnected during normal operation, the purifier must be shut down and all inlet/outlet valves should be closed in order to isolate the unit from the facility lines. This will limit the exposure of the getter bed to atmosphere while it is in excess of 150 °C. Do not restart the purifier until temperature has cooled to below 150 °C and all gas lines have been reconnected/repaired and certified. Failure to comply can result in personal injury or damage to the purifier.

CAUTION

The purity guarantees are void if installation is other than that approved by GOW-MAC Instrument Company

CAUTION

During heated operation, this unit MUST be pressurized with noble gas and isolated from any exposure to atmosphere or the getter bed will be destroyed.

3 75-810 Specifications

Gases Purified	He, Ne, Ar, Kr, Xe
*Impurities Removed:	H ₂ O, H ₂ , O ₂ , N ₂ , CO, CO ₂ , CH ₄
Impurities Not Removed:	He, Ne, Ar, Kr, Xe
Operating Temperature:	400 °C <u>+</u> 25 °C
Temperature Sensor:	K-Type thermocouple is integrated in the heater, monitor the cartridge temperature by connection with a flat pin connector to the receptacle on the top panel
Maximum Recommended Operating Pressure:	17.0 atm (250 psig)
Suggested Maximum Flow Rates (ccpm):	Model 75-810: 1500
**Gas Connections:	1/4" VCR® Male
Leak to Atmosphere	<2 X 10 ¹⁰ atm-cc/sec helium
***Lifetime Purifying He or Ar:	75-810: 1 yr. @ 200 ccpm
Power Requirements:	75-810: 80 Watts, 120 VAC 75-812: 80 Watts, 230 VAC
Circuit Breaker	In power switch, fast acting, 1A rating
Weight (net):	3.5 lbs.
Dimensions:	5"D x 4"W x 10"H

 * Each impurity removed to < 10 ppb based on 10 ppm total inlet impurities at suggested maximum flow rate

** VCR® is a registered trademark of Swagelok Corporation

*** Lifetime based on suggested maximum flow rates; a higher flow rate will proportionally decrease the lifetime while a lower flow rate will increase the lifetime. The lifetime is rates at 10 ppm total inlet impurities; higher or lower inlet impurity levels will decrease or increase the lifetime proportionally. The purifier bed is considered an expendable item. System leaks, poor quality gas, and improper handling will adversely affect its useful life.

4 Installation

Locate the 75-810 on a table top with the rubber feet on the surface. In this position, the gas in and out connections are on top and the purifier cartridge is in the correct vertical orientation which reduces gas channeling through the getter material.

Connect the 75-810 fitting marked GAS IN to the source of carrier helium or argon. A diaphragm shut-off valve in the supply line will facilitate servicing the 75-810. Recommended tubing specification is 1/8 inch OD X 0.028 wall, 316L stainless steel, cleaned or electropolished. Orbital butt weld a ¹/₄ inch VCR® gland including a female nut for connection to the 75-810. Test any new weld to verify there is no leak. Likewise prepare a tube to connect to the GAS OUT fitting. Connect the 75-810 GAS OUT to the CARRIER IN connection on the gas chromatograph or ZERO GAS IN connection on an analyzer.

The flat pin receptacle on the top panel allows monitoring of the cartridge temperature. For usual operation, monitoring is not necessary because the heater should operate at optimum temperature when the 75-810 is powered on. It is during transitions to start-up and shut-down that knowing the temperature is important, especially the 150 °C point that is referred to throughout this manual. Use a flat pin thermocouple connector (not provided) and K thermocouple leads connected to a suitable meter to monitor 75-810 temperature.



(4) RUBBER FEET





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SIDE VIEW

5 Operation

Start Up

The purifier should be purged with an inert gas (helium if -HE or argon if -AR model, minimum purity 99.99%) for at least 10 minutes before powering on. Apply continuous gas flow of 50 to 200 ccpm before and during initial activation. After the 10 minute purge period, switch on power to the 75-810. The heater is selected to heat the purifier to about 400 °C. At this temperature, the purifier material will take 3 hours to fully activate. After this activation period, the purifier is ready to operate at the desired flowrate.

CAUTION

IF THE 75-810 SUPPLY OR VENT LINE IS ACCIDENTALLY VENTED TO ATMOSPHERE, ASSUME AIR HAS ENTERED THE PURIFIER. IMMEDIATELY SWITCH OFF POWER AND FLOW HELIUM OR ARGON THROUGH THE PURIFIER AT 50 TO 200 CCPM TO PURGE OUT ANY AIR. EXPOSURE OF THE GETTER MATERIAL TO OXYGEN WHEN IT IS ABOVE 150 °C WILL DESTROY THE MATERIAL AND MAY INITIATE AN EXOTHERMIC REACTION.

Examples of the performance of the 75-810 for helium and argon gases are in the Appendix.

Shutdown

If the gas line containing the 75-810 is to be vented to air, for example during purifier replacement, then the purifier must be at room temperature before venting to air. If the purifier is hot, switch off power, maintain gas flow and wait 3 hours to allow the purifier to reach room temperature.

6 Maintenance & Servicing

Purifier Lifetime

In order to maintain proper purification efficiency, do not exceed estimated lifetime of the purifier cartridge. The estimated lifetime (first impurity breakthrough) is 1 year at 400 °C operating temperature, 150 ccpm flowrate, and inlet gas purity of 99.999%. First breakthrough of H2 or O2 at these conditions should be after about 4 years. Lifetime is inversely proportional to the total inlet impurity level and to the average flow rate.

The getter material in 75-810 models cannot be regenerated so the cartridge must be changed. In the unlikely event of H2 or O2 breakthrough, the hydrogen removal stage can be regenerated in the field.

Hydrogen stage regeneration

- Power off the 75-810 and allow 3 hours to cool while maintaining minimum 50 ccpm inert gas flow through the cartridge. Monitor the temperature using the K thermocouple on the unit. Ensure cartridge temperature is below 150 °C before turning off gas flow. Keep all gas connections intact if possible to prevent ingress of air.
- Switch off power on the 75-810 and UNPLUG THE POWER CORD.
- Open the case
- Loosen the hose clamp on the heatsink which is on the hydrogen removal stage. Slide the heatsink away from the cartridge.
- Move the heater and insulation from the first stage to the hydrogen stage and re-tighten both.
- Check integrity of wire connections inside the case
- Follow the steps for initial activation in order to regenerate the hydrogen stage. Refer to Section 5 Operation/Start Up for activation procedure.
- After regeneration, replace heater, insulation, and heatsink to their original locations, check wire connections, and close the case.

Replacing the cartridge and/or heater

- Power off the 75-810 and allow 3 hours to cool while maintaining minimum 50 ccpm inert gas flow through the cartridge. Monitor the temperature using the K thermocouple on the unit. Ensure cartridge temperature is below 150 °C before turning off gas flow. Keep all gas connections intact if possible to prevent ingress of air.
- Switch off power on the 75-810 and UNPLUG THE POWER CORD.
- Open the case
- Loosen the hose clamp on the heatsink which is on the hydrogen removal stage. Slide the heatsink away from the cartridge. Retain heatsink for re-assembly.

- Remove the insulation and retain for re-assembly
- Remove the heater and retain for re-assembly unless also replacing the heater
- With one each 5/8 inch and ³/₄ inch open end wrenches, undo the VCR® connections on the cartridge and remove the two gaskets, which should be discarded.
- Insert the new cartridge, TAKING CARE TO PLACE THE FIRST STAGE CLOSEST TO THE GAS IN FITTING. Use new VCR® gaskets provided with the replacement cartridge. Assemble the VCR® nut finger tight and then use the wrenches to tighten a further quarter-turn.
- Replace heater, insulation, and second stage heatsink. Tighten all hardware and secure insulation and heatsink on the first and second stages respectively
- Check integrity of wire connections inside the case and close the case
- Follow the steps for initial activation. Refer to Section 5 Operation/Start Up for activation procedure.

Waste Disposal

This procedure is for the purifier cartridges in all models 75-810.

If the purifier is used, then the getter material inside the purifier is already in a nonreactive state, (i.e. will not react with air).

If the material is still in a reactive state (i.e. the purifier is not completely "used up"), it still will not sustain combustion in air except at temperatures over 350 °C, and with a continued exposure to air.

The operator should place a VCR® cap on the inlet and outlet fittings for sufficient precaution before disposal. It is then acceptable to deposit in an approved landfill, in accordance with local, state and/or federal regulations. When disposed of in this manner, the waste poses no known environmental problems.

7 Replacement Parts

<u>Part No.</u>	Description
124-240	Heater, 115 VAC, 80 W (includes integral K thermocouple)
124-241	Heater, 230 VAC, 80 W (includes integral K thermocouple)
180-1000-HE	Purifier cartridge for helium, 1/4" VCR® male connections. Includes hydrogen removed stage and internal 0.003 micron filter and two (2) VCR® gaskets. Does not include insulation or heatsink.
180-1000-AR	Purifier cartridge for argon, 1/4" VCR® male connections. Includes hydrogen removed stage and internal 0.003 micron filter and two (2) VCR® gaskets. Does not include insulation or heatsink.

8 Appendix

Summary of Helium Purifier Performance in Removing Impurities

Instrument	: GOW- I	MAC Series	200 GC
Detector	: DID		
Detector Temperature	: 35°C		
Detector Flow	: 10 ml/m	nin	
Column	: 4.5' x 1.	.8" Molecula	r Sieve 13X
Oven Temperature	: 50°C		
Sample Gas	: 2 ppm s	tandard atm	ospheric gases in Helium
Sample Flow	: 60 ml/m	nin	1 0
Carrier Gas	: Helium		
Carrier Flow	: 30 ml/m	nin	
Purifier Operating Temperature	: 400°C (Factory pre-	set)
		CH4	— — - No Purifier — — Purifier @ 400°C
			0
Ar			
ä	N2	: :	co



Table 1: Remaining Concentration of Impurities with Helium Purifier at 400°C

Key Findings:

- 1. CO is totally removed.
- 2. N2 peak is negative: N2 is removed by purifier so less N2 remains in sample line compared to in carrier gas.
- 3. Other peaks (except for Argon) are reduced to ppb level.

Summary of Argon Purifier Performance in Removing Impurities



Impurities	Responses (mV.s)	Concentration (ppm)
H2	259.943	0.215
02	0.000	0.000
N2	0.000	0.000
CH4	58.359	0.034
СО	0.000	0.000

Table 2: Remaining Concentration of Impurities with Argon Purifier at 400°C

Key Findings:

- 1. O2, N2, and CO are totally removed.
- 2. H2 and CH4 are reduced to ppb level.