NuPure[®] HPD SERIES

Palladium Membrane Hydrogen Purifiers



NuPure Corporation's HPD Series of Hydrogen Purifiers produce "State of the Art" hydrogen by utilizing the well established principle of permeation through a palladium alloy.

NuPure[®] HPD Series purifiers' standard size range is from 1 SCFH to 500 SCFH (0.5 slpm to 250 slpm), serving commercial and laboratory users in the electronic, analytical, chemical and metallurgical fields.

The product gas is particularly useful for critical epitaxy and MOCVD processes, and as a reference gas or carrier gas in high sensitivity analytical devices. NuPure[®] HPD-Series Purifiers operate on the "inside-out" principle, which allows the use of very impure feed gases with maximum hydrogen recovery. (See Technical Article: "OXYGEN SENSITIVITY IN OUTSIDE-IN AND INSIDE-OUT HYDROGEN PURIFIERS")

Smaller purifiers can be automated by the use of Automatic Temperature Control Systems ("**ATCS**") for "smart" purifier Start-Up and Shut-Down. These outboard Automatic Temperature Control Systems must be purchased separately with small flow purifiers. Larger systems are already fully automated as purchased. *(See Technical Description: "ATCS Purifier Start-Up & Shut-Down Automatic Temperature Control Systems")*.

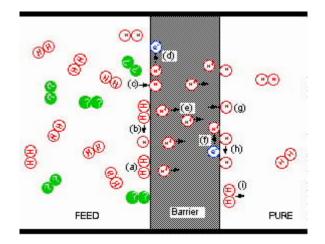
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PRINCIPLES OF OPERATION

NuPure Corporation's HPD Series of purifiers take advantage of the fact that only hydrogen and its isotopes (Deuterium, Tritium) can be made to pass through a palladium barrier. NuPure utilizes palladium alloy tubing which has been optimized for this use.

Palladium-purified hydrogen represents the "state of the art" in gas purity. Analytical techniques have not yet advanced to the point where impurities can be detected in this product.

Hydrogen permeation through palladium is a metallurgical process in that hydrogen "alloys" with the barrier material, diffuses through it as protons and "de-alloys" from the far side of the barrier. The barrier remains absolutely leak tight to all other species than hydrogen and therefore serves as an absolute filter to pass only hydrogen (and its isotopes.)



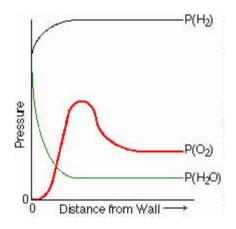
Diatomic hydrogen, at an elevated pressure, is adsorbed (a) on a palladium-alloy barrier, where it disassociates into monatoms (b) which are absorbed into the barrier (c). Other species of gases are adsorbed, but do not dissolve into the barrier.

The monatomic hydrogen donates electrons to the electron cloud in the metal (d) becoming protons (H+), which diffuse (e) toward the far side of the barrier. At the far surface, the protons pick up electrons (f), becoming monatoms again, move to the outer surface (g), recombine to form diatoms (h) and break free as gaseous hydrogen (i).

All NuPure HPD Series purifiers work with a pressure differential from the inside of the tubing outwards, avoiding all possibility of collapse or crushing of permeation tubing and guaranteeing flow characteristics such that there are no stagnant areas which might concentrate impurities.

OXYGEN SENSITIVITY IN OUTSIDE-IN AND INSIDE-OUT HYDROGEN PURIFIERS

1. In an Outside-In Purifier, hydrogen moves toward a palladium-alloy wall, where it is removed by diffusion. The vector for the direction of gas flow is essentially perpendicular to the wall.



2. Hydrogen carries all impurities toward the wall, increasing the concentration of the impurities as the wall is approached.

3. Oxygen moves, with other impurities, towards the wall. The innermost oxygen burns catalytically with hydrogen to form steam, which is trapped at the wall as an impurity.

4. The steam layer prevents further catalytic combustion and oxygen continues to build up on the far side of the steam layer in the same manner as the non-reactive (inert) impurities.

5. Oxygen can build up without limit until it reaches the Lower Explosive Limit, where it can flash spontaneously or until the inert shield layer of nitrogen, steam, etc. ruptures (because of eddy currents, geometric irregularities or the like) and the oxygen rich layer impinges on the catalytically active palladium surface and burns. The potential temperature change for the reaction is not a function of the initial oxygen concentration under these circumstances and can be much higher than calculated from the feed gas composition.

With the outside-in geometry, it is possible to generate destructively high surface temperatures at the permeation barrier with relatively small amounts of oxygen in the feed gas.

This problem does not exist in NuPure's Inside-Out Purifiers in which the permeation tubing is well swept and the major flow is parallel to the permeation surface rather than perpendicular to it. Oxygen is carried immediately to the wall and burns catalytically at low concentration and therefore with a small temperature change. Combustion products and inert impurities are pushed ahead of the incoming gas stream and are concentrated in a predictable manner as hydrogen is lost through the permeation tube wall.

ATCS

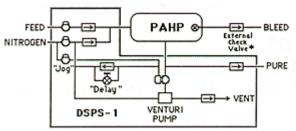
Purifier Start-Up & Shut-Down Automatic Temperature Control Systems

Palladium Alloy Hydrogen Purifiers (PAHP's) are among the class of devices that require considerable operator sophistication for safe operation, both with respect to obvious hazards in handling hydrogen and to less obvious requirements for maintaining the integrity of the palladium alloy barrier and thus the purity of the process stream.



ATCS Protection Systems are designed to respond appropriately to emergency situations, such as power loss, and to assist the operator to start up and shut down a PAHP under normal operating conditions.

ATCS units are mounted externally to PAHP's and replace the external networks recommended in PAHP instruction manuals.



*Required if unit does not have internal check valve in bleed line

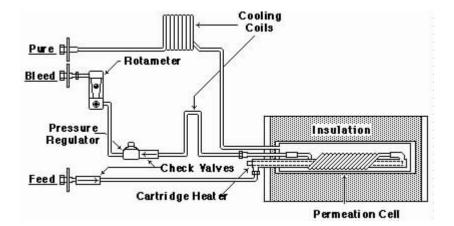
A primary rule for the reliable operation of a PAHP is to **never have hydrogen present** below 315°C.

By the proper choice of normally open and normally closed valves, the ATCS shuts off the feed hydrogen, flushes the crude side with nitrogen and evacuates the pure side whenever power is lost, preventing the PAHP from cooling in hydrogen.

Most power outages are of trivial duration and do not call for complete shut down. Timing circuitry is provided in the ATCS to allow the unit to come back on line whenever the power interruption is less than three minutes. When the power is off longer than three minutes, the feed hydrogen remains off when power returns.

A secondary function of the ATCS is to assist in normal PAHP start-up and shut-down. Whenever the Power Switch is turned On or Off, a 15 minute purge starts. When the unit is turned Off, the purge proceeds as in the case of an emergency shut-down, but is limited to 15 minutes and the evacuated PAHP cools safely to room temperature.

SINGLE CELL PURIFIER DESIGN (HPD-5 through HPD-100)



AIR COOLING

Pure and bleed gases exit the permeation cell at 425 $^{\circ}$ C. Both are cooled by natural convection, which cannot fail, rather than by fans or blowers, which can.

"SET AND FORGET" BLEED FLOW

Bleed flow control and indication is provided by a front panel rotameter and a line pressure regulator. As long as feed pressure is maintained above 50 PSI, a constant pressure is presented to the bleed rotameter, which then maintains a constant flow. A check valve in the bleed line prevents air from migrating back in the bleed line after the unit is shut down. This removes the need to close the bleed flow valve on shut-down

FORMAL FEED GAS PREHEATING

The Feed Gas enters the permeation cell by passing along the outside of a cartridge heater well. The Feed Gas traverses the full length of the heater well before it reaches the permeation tubing. **Competitive systems lack this preheating!**

FEED GAS CHECK VALVE

A check value on the feed line prevents sudden decompression of the feed side of the system and protects the permeation tubing from back pressure.

HIGH TEMPERATURE CONTROL AND ALARM (NOT SHOWN)

A second temperature control breaks the furnace line mechanically (and sounds an alarm) whenever the cell temperature rises 28 °C above the main control point. The second control temperature is still safe for operation and **the unit does not shut down!** The choice of when to shut down is left to the operator, who may wish to continue production for a reasonable period prior to removing the purifier from service.

Purifiers larger than 100 SCFH (Models HPD-150 and up) use multiple cells in parallel and incorporate appropriate modifications of the features listed above.

HPD Series Specifications

MODEL	CAPACITY	POWER	DIMENSIONS			WEIGHT
	SCFH*	Watts	Width (in.)	Depth (in.)	Height (in.)	Kg
HPD-1	1	90	12	16	5	11
HPD-2	2	150	12	16	5	11
HPD-5	5	300	22	13	16	20
HPD-10	10	300	22	13	16	20
HPD-25	25	450	22	13	16	25
HPD-50	50	800	27	13	16	32
HPD-75	75	800	27	13	16	32
HPD-100	100	800	27	13	16	32
HPD-150	150	1600	29	24	16	55
HPD-200	200	1600	29	24	16	55
HPD-250	250	2400	32	24	27	82
HPD-300	300	2400	32	24	27	82
HPD-400	400	3200	32	24	27	105
HPD-500	500	4000	32	24	38	136

* Based on a feed of Commercial Grade Hydrogen at 250 PSIG Inlet and 0 PSIG Outlet Pressure. Contact factory for assistance in DE-RATING to determine flows at different Feed and Product Pressures. NOTE: 1 SCFH = 0.5 slpm

HPD-100 & smaller units operate on 120 VAC, 50/60 Hz (240 VAC Available) HPD-150 and larger units operate on 240 VAC, 50/60 Hz.

- Cajon VCR Pure Gas Terminations are Standard (Swagelok Available)
- Swagelok Feed and Bleed Terminations Standard.
- Only Stainless Steel Components in Contact with Pure Gas.
- All interior connections on pure side are either welded or VCR.

(Construction Integrity Verified by Helium Mass Spectrometer)

* FLOW "DE-RATING"

HPD Series Capacity is defined in terms of throughput of ultra-pure hydrogen at one atmosphere from a feed gas of commercially pure hydrogen at 250 PSIG. For example, the Model HPD-100 will deliver 100 SCFH under these conditions. The relationship between pressure and throughput, however, is not linear.

NuPure can assist to calculate the size of a purifier required to deliver specific flows at various inlet and outlet pressure combinations. The following rules apply: (1) The Feed Pressure must be greater than the Product Pressure (2) The Feed Pressure must not exceed 270 PSIG.

To "de-rate" purifiers for extraction of hydrogen from highly crude streams (e.g. from ammonia), please contact:

<u>NuPure Corporation</u> 106-B Schneider Road, Ottawa, ON, Canada K2K 1Y2 Tel: (613) 836-0336 Fax: (613) 836-0297 E-mail: <u>sales@nupure.com</u>