

Generating Hydrogen for Head Space GC

Market Application Publication



Background:

Head space gas chromatography (GC) is commonly used for the extraction and monitoring of volatile compounds in complex matrices. Typical applications of headspace GC include the analysis of solvents in pharmaceuticals, alcohol in blood and aromatic hydrocarbons in environmental samples. In many laboratories, He is used as the extraction gas and carrier gas for headspace GC, but supply and cost issues have led chromatographers to employ H_2 or N_2 in its place. Of the two, H_2 can provide more rapid separations, increasing laboratory throughput.

H_2 can be readily generated by the electrolysis of water and is a very effective gas for the extraction of volatile compounds that can be safely used at elevated temperatures. H_2 is typically supplied in highly pressurized cylinders which may present a safety hazard. In addition, pressurized cylinders are inconvenient and involve significant handling and cost. In recent years, in-house H_2 generating systems using the electrolysis of water have been developed to provide the desired gas with a high degree of purity so that the chromatographer can obtain the gas at a flow rate and pressure which is compatible with the chromatographic system. The use of an in-house system is safer, more convenient and less expensive than the use of bottled gas.

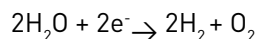


Features and benefits:

- Generates up to 99.99999 % pure H_2 at flow rates as high as 300 cc/min. Minimal O_2 (<0.01 ppm) and H_2O (1.0 ppm) content
- Requires minimum maintenance, maximizes up-time and laboratory efficiency
- Enhances laboratory safety, generates only the required flow rate of H_2 . Certified by CSA, UL, IEC1010 and CE
- Prevents running out of gas during instrument operation and eliminates the need for cylinders
- Extremely low cost of operation, no hidden costs (demurrage, maintaining inventory). Payback period typically less than 1 year
- Compact system frees up laboratory floor space; ideal for mobile laboratories
- Extremely reliable; operates 24 hours a day, 7 days a week

Hydrogen Generation:

H₂ is obtained by the electrolysis of water as shown in the equation below using a metallic electrode or an ionomeric proton exchange membrane.



The traditional method of generating H₂ via the electrolysis of water involves a metal anode (e.g. Pd), a metal cathode and a strong, water soluble electrolyte such as 20% NaOH (since water does not conduct an electric current very effectively). A cathode consisting of a bundle of palladium tubes is used to provide high purity gas (only H₂ and its isotopes pass through the cathode, oxygen and other

impurities collect at the anode). The use of a Pd tube cathode provides a considerable improvement in the purity of the generated H₂, compared to systems that use a desiccant as the final drying agent.

Proton exchange membranes such as Nafion® (a sulfonated tetrafluoroethylene polymer) or polybenzimidazole (PBI) are ionic polymers that are commonly used in fuel cells to generate a potential from H₂ and O₂ (with water as a byproduct). When an appropriate potential is applied to the membrane in the presence of water, the reverse process occurs creating O₂ and H₂. A significant benefit of this approach for

the generation of hydrogen is that the caustic 20% NaOH solution used with a metallic electrode is not required. A Pd membrane is included to further purify the H₂ by removing O₂ to less than 0.01 ppm and moisture down to <1.0 ppm.

A number of generators are available to allow the user to configure the systems to meet the specific requirements of the facility; as an example, the H2PD (Palladium electrode) series includes systems that can up to 99.9999+ % at flow rates as high as 300 cc/min while H2PEM and H2 systems (ion exchange membrane) can provide flow rates as high as 1200 cc/min.

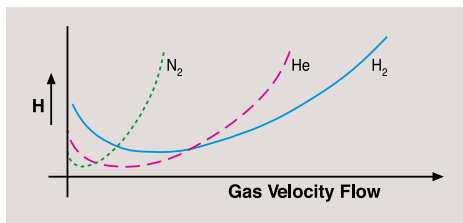
Applications:

In a typical application, Pd based hydrogen generators (H2PD-150 and 300) are used to provide H₂ for headspace GC at the Department of Forensic Sciences at West Virginia University. Dr. Suzanne Bell of WVU reports that her group analyzes a broad range of samples by headspace GC including blood (alcohol) and burnt materials such as wood in arson investigations. The headspace extraction was carried out using H₂ at 65°C. It should be noted that vials containing H₂ can safely withstand fairly extreme conditions as an example, Dr. Bell reports that H₂ was used to extract a series of terpenes from wood samples using a 20 minute extraction using H₂ at 120°C for 20 min.

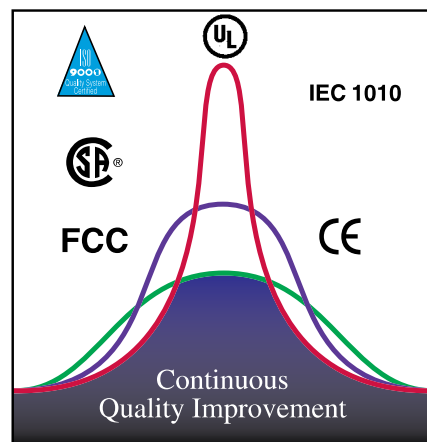
Full flow operation of a generator on a 24 hour/7 day basis requires approximately 4 L of water a week and can be automatically filled by installing a continuous source of DI water to ensure continuous operation. The conductivity of the water used for a PEM based system is continuously monitored (ionic materials in the water could foul the electrode and the system will shut down if the conductivity reaches a preset level). If a PEM based system is employed, the deionizer and filter are replaced every six months. Dr. Bell, reports that their in-house systems are very reliable, and always provides the required hydrogen as needed.

There are several benefits for using a in-house generator instead of a compressed gas tank for hydrogen purge gas including safety, cost and convenience.

A hydrogen generator supplies the gas at the desired flow and pressure to the system, while a high pressure hydrogen tank is a significant potential hazard due to loss of control of tank in transport or the rapid loss of the gas if the valve is compromised. Another advantage of an in-house generator is that once a system is installed, it provides the gas on a 24 hour /7day basis with essentially no user interaction; it is not necessary to replace tanks, a time consuming operation. The operating costs for a hydrogen generator are very low, compared to the use of tank gas and the use of an in-house generator dramatically reduces the environmental impact, since heavy gas tanks need not be transported from the source to the point of use.



The Van Deemter Curves (above) show a comparison of nitrogen, helium and hydrogen carrier gases. A Parker Balston Gas Station will also allow the user to exploit the benefits of using hydrogen carrier gas instead of helium. Increased flow velocity can shorten analysis time by 50%.



Principal Specifications:

Description	Model Number	
Hydrogen Purity	H2PEM Series:	99.9995 %
	H2PD Series:	99.99999+%
	H2-NA Series:	99.99999+%(O ₂ <0.01ppm, H ₂ O <1.0ppm) CO ₂ Concentration < 1 ppm
Maximum Flow	H2PEM-100:	100 mL/min
	H2PEM-165:	165 mL/min
	H2PEM-200:	200 mL/min
	H2PEM-510:	510 mL/min
	H2PD-150:	150 mL/min
	H2PD-300:	300 mL/min
	H2-500NA:	500 mL/min
	H2-800NA:	800 mL/min
	H2-1200NA:	1200 mL/min
Delivery Pressure	H2-PEM Series:	5-100 psig +/- 0.5 psig
	H2PD Series:	0-60 psig
	H2-NA Series:	0-100 psig ²
Outlet Port	H2-PEM Series:	1/8" Compression
	H2-PD Series:	1/8" Compression
	H2-NA Series:	1/4" Compression ²
Electrical Requirements ¹	H2-PEM Series:	120/230 VAC
	H2PD Series:	120V, 50/60 HZ , 3.15 A
	H2NA Series:	120V, 50/60 Hz, 5.3 A
Certifications	H2-PEM Series:	CSA, UL, IEC-1010, CE Mark
	H2PD Series:	CSA, UL 3101, IEC-1010-1, CE Mark
	H2NA Series:	CSA, IEC-1010, CE Mark
Dimensions	H2PEM Series:	17.12"H x 13.46"W x 17.95"D (43.48cm x 34.19cm x 45.6cm)
	H2PD Series:	22"H x 12"W x 12"D (58cm x 30cm x 30cm)
	H2-500NA:	13"H x 15"W x 18"D (33cm x 38cm x 46cm)
	H2-800NA, 1200NA:	15.5"H x 13"W x 17"D (39cm x 33cm x 43cm)
Shipping Weight	H2PEM Series:	59 lb (27 kg)
	H2PD Series:	58 lb (26 kg)
	H2-NA Series:	45 lb (20.4 kg)

Notes:

¹ Electrical requirements are for North America, see product catalog for electrical and plug configurations for other locations.

² H2NA-500 does not include automated water feed feature, maximum H₂ pressure is 90 psig, Outlet port is 1/8" compression.

Ordering Information H2-PEM Series*:

Description	Model Number
Hydrogen Generator	H2PEM-100, H2PEM-165 H2PEM-260, H2PEM-510
Desiccant Cartridge	MKH2PEM-D
6 Month Service Kit	MKH2PEM-6M
24 Month Service Kit	MKH2PEM-24M
Installation Service	H2PEM-100: MKH2PEM-100-INST H2PEM-165: MKH2PEM-165-INST H2PEM-260: MKH2PEM-260-INST H2PEM-510: MKH2PEM-510-INST
Preventative Maintenance Plan	H2PEM-100: MKH2PEM-100-PE H2PEM-165: MKH2PEM-165-PE H2PEM-260: MKH2PEM-260-PE H2PEM-510: MKH2PEM-510-PE
USB Remote Control Accessory	604970894

Ordering Information H2PD Series*:

Description	Model Number
Hydrogen Generator	H2PD-150 H2PD-300
Electrolyte Solution	920071
Pressure Regulator	W-425-4032-000
Preventative Maintenance Plan	H2PD-150-PM H2PD-300-PM
Installation Kit	IK7532
Extended Support with 24 Month Warranty	H2PD-150-DN2 H2PD-300-DN2

Ordering Information H2-NA Series*:

Description	Model Number
Hydrogen Generator	H2-500NA H2-800NA H2-1200NA
Deionizer Bags (2 each)	7601131
Preventative Maintenance Plan	H2-500NA-PM H2-800NA-PM
Extended Support with 24 Month Warranty	H2-500NA-DN2 H2-800NA-DN2

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