AIR PRODUCTS

Hydrogen, now a safer solution for the laboratory. . .
tell me more

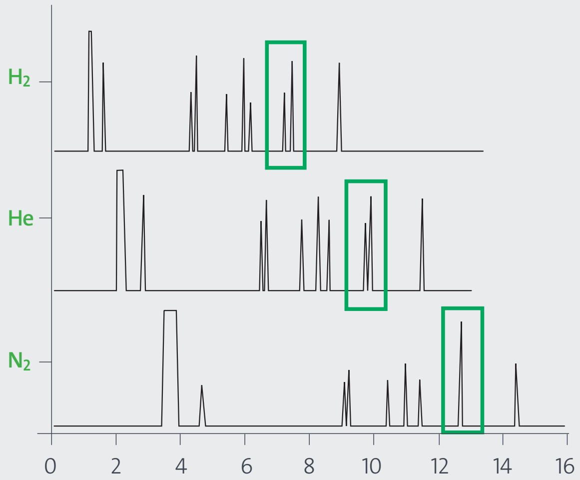
Hydrogen
as a carrier gas for Gas Chromatography

Hydrogen is considered by many to be the best carrier gas for gas chromatography and in some applications, it is the carrier gas of choice because of its specific characteristics - fast analysis, high efficiency and reduced costs.

Yet helium continues to be the most commonly used carrier gas. Things are changing, however, as the recent temporary helium supply restrictions prompted chromatographers to look again at hydrogen and reconsider its benefits to GC. Helium is still an excellent option for a carrier gas in GC and while there are enough reserves for several centuries, the use of hydrogen is increasing and GC manufacturers are introducing new equipment optimised towards hydrogen carrier gas.

Unlike helium, hydrogen is flammable. But its high diffusivity means faster linear velocities and shorter analyses, while still providing the same separation efficiency as helium.

Figure 1: Effect of carrier gas on resolution and analysis time



These chromatograms illustrate the differences among hydrogen, helium, and nitrogen as carrier gases in temperature programmed capillary GC.

The sample is a Supelco isothermal non-polar test mixture. Each carrier gas was set at its optimum linear velocity at the initial temperature of the temperature ramp. Notice the differences in the retention times and resolution. The column and conditions were not changed.

Shorter analyses mean increased throughput and lower costs per sample. However, it is worth noting that there could be a problem with reactivity in some situations (catalytic hydrogenation of unsaturated molecules at high inlet temperature) when using H_2 as the mobile phase. The potential for chemical reactions in your analytical system must also be evaluated.

Note: There are also some potential issues when using hydrogen as the mobile phase in gas chromatography-mass spectrometry (GC-MS). Consult your GC-MS supplier for advice.

The Air Products solution – Hydrogen BIP®

To meet increased demand for hydrogen as a carrier gas, we have now extended our unique BIP® technology to hydrogen.

Hydrogen BIP® cylinders are available with ultra low impurity specifications, not previously available from cylinder or generator sources. (See Table on the right)

Our unique BIP® cylinders use a sophisticated method to eliminate critical impurities as the gas is removed from the cylinder, offering the highest purity levels and suitable for the most demanding gas chromatography applications.

Every H_2 BIP® cylinder contains less than: 20 ppb of water; 100 ppb of oxygen and 10 ppb of total hydrocarbons. This makes Hydrogen BIP® gas many times purer than all conventional hydrogen grades.

Figure 2: Impurity specifications in ppb or ppm molar

Grade	H_2O	O_2	THC	CO+ CO_2	N_2	Certification
H_2 BIP® cylinder	20 ppb	100 ppb	10 ppb	0.5 ppm	2 ppm	Batch
H_2 BIP® PLUS cylinder	20 ppb	100 ppb	10 ppb	50 ppb	200 ppb	Individual

THC = Total Hydrocarbons measured as Methane

Case Study
MATGAS - Making the helium - hydrogen switch

MATGAS, a leading R&D organisation and Centre of Excellence based in the Universitat Autònoma de Barcelona campus in Spain, has made the change and is now using hydrogen as a carrier in gas chromatography. The switch, according to director Dr Lourdes Vega, has led to improved performance and reliability, and reduced costs.

When switching from helium to hydrogen, the modifications required to the gas chromatograph relate to the hazards of hydrogen. Hydrogen is flammable and can create an explosive atmosphere when it accumulates.

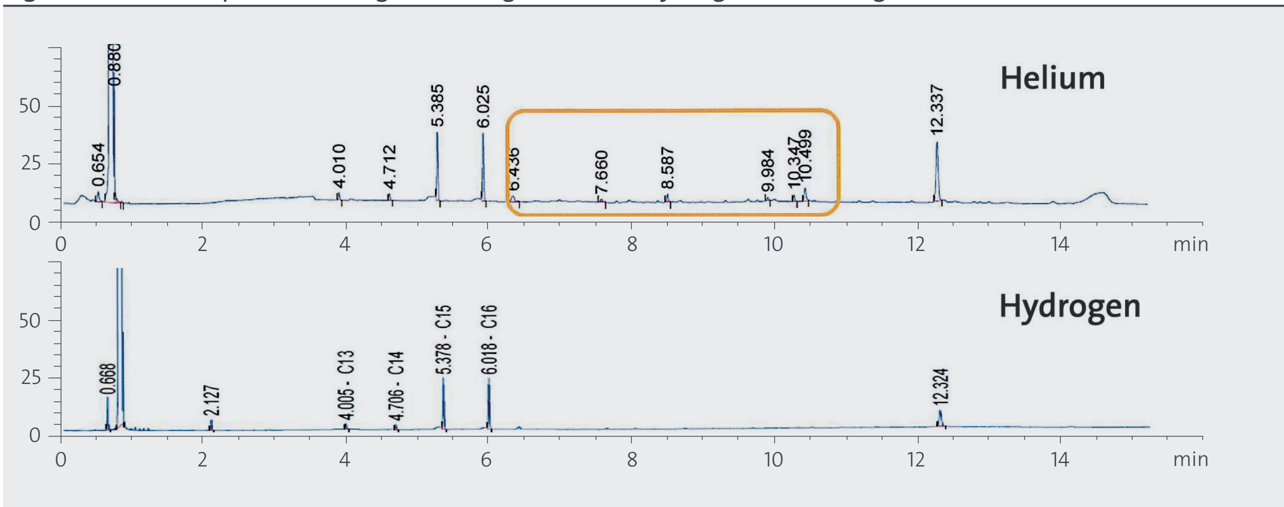
The priority is to use safety standards to ensure there is no build-up of gas, by avoiding and detecting leaks, and safely venting any hydrogen outlet stream.

Avoiding gas build-up

Modern gas chromatograph (GC) can detect leaks upstream in the column by monitoring gas pressure. This can be used directly for a GC working with hydrogen. If hydrogen leaks before reaching the column, the GC pressure drop is negligible – hydrogen pressure cannot build up and it reaches the defined set point. The GC interprets the permanent difference between the operative and the set point pressure as a leak and protects the GC by shutting off the hydrogen valve.

However, when the leak is downstream in the column, this safeguard is not efficient and a hydrogen sensor needs to be installed to detect any hydrogen build-up in the oven.

Figure 3: 0.5uL sample chromatograms using Helium or Hydrogen as carrier gas



Safe venting

The second modification needed when changing from helium to hydrogen is to safely vent the outlet stream. The ports involved are the outlet of the septum purge and the split from the injector. This is necessary because when helium is used it can be vented inside the laboratory, but when hydrogen is used the ports need to conduct it to the lab's flammable vent line.

The MATGAS lab was already prepared for working with hydrogen and had an FID detector installed, so no further modifications were needed. MATGAS also has a flow restrictor upstream of the pressure regulator, which limits the maximum leak possible and ensures lab ventilation can dilute any hydrogen leak. Flow restrictors are a low cost, useful and efficient way of controlling this type of gas leakage.

Once these measures were addressed, MATGAS verified the new GC configuration by running a standard FID performance evaluation test. This showed better performance was obtained with BIP® hydrogen compared with helium, with the base line being more stable and accurate.

"We are very happy with our decision to move from helium to hydrogen BIP® for GC," said Dr Vega. "It has allowed us to reduce the cost of using this analytical tool while improving its performance and reliability."

Provided it's used at the right purity and the correct safety measures are taken, she said: "Hydrogen can be a highly effective carrier gas that can improve quality, speed up your process and help you save money."

"Hydrogen can be a highly effective carrier gas that can improve quality, speed up your process and help you save money."

Dr Lourdes Vega, MATGAS.

MATGAS

H_2 Cylinders or Generators?

Hydrogen generators may offer an option for GC carrier and fuel gases and we should consider their relative advantages and disadvantages against cylinders before making a decision.

H_2 generators have two main advantages:

- They are a good solution in remote locations when cylinder supply is difficult or impossible.
- They produce H_2 on demand, so little H_2 is stored.

But H_2 generators are not always the best solution. There are often hidden drawbacks.

- Gas Specification – This should be checked carefully, as most manufacturers only give either the O_2 or H_2O impurity levels, not both.
- Cost - H_2 generators are usually more expensive than an H_2 cylinder supply.
- Reliability and Back-up- H_2 generators can fail catastrophically without warning, so back-up cylinder supply should be regarded as essential.
- Specialist Equipment - Deioniser bags purify the demineralised water supply. (These must be changed often or the generator will be severely damaged.)

Figure 4: BIP® valve and purifier design

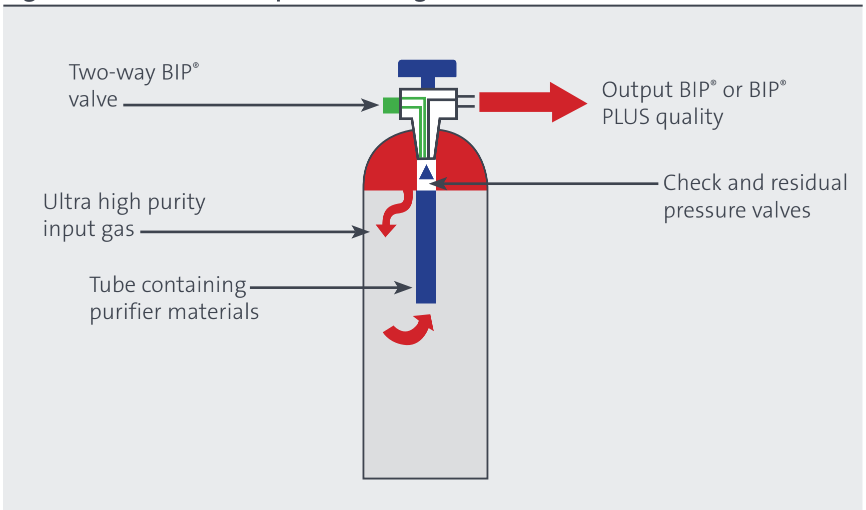
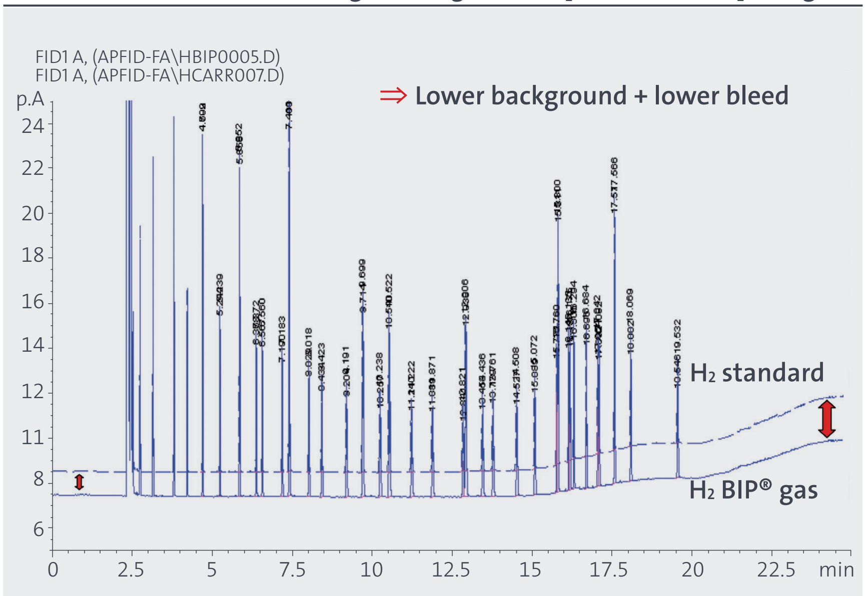


Figure 5: Baseline noise reduction and column bleed improvement with simultaneous fuel and carrier gas change from H_2 standard to H_2 BIP® gas



Safety Systems

An undetected gas leak can occur with a broken column or a leaking connection whether a cylinder or generator is supplying the carrier gas. The danger is that an undetected gas leak could result in an explosion in the GC oven, placing laboratories and personnel at risk.

Being able to safely detect hydrogen leaks in the GC oven is critical to any laboratory using hydrogen as a carrier gas. H_2 sensors are available from all major GC suppliers and they ensure the safe use of hydrogen in GC analysis. The H_2 sensor does this by constantly monitoring the H_2 concentrations in the GC oven and automatically switching to an inert gas when typically, 25% LEL is reached. This important feature eliminates risks and ensures safety.

For more information, please contact us at:

Air Products PLC
2 Millennium Gate
Westmere Drive
Crewe, UK
CW1 6AP
T 0800 389 0202
E apukinfo@airproducts.com

Air Products Ireland Ltd.
Unit 950, Western Industrial Estate
Killeen Road, Dublin 12
T 1800 99 50 29
E ieinfo@airproducts.com

AIR PRODUCTS

tell me more
airproducts.co.uk
airproducts.ie

 250-14-001-EN