



Agilent EI GC/MS Instrument Helium to Hydrogen Carrier Gas  
Conversion

## User Guide

# Notices

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# Introduction

Gas chromatograph/mass spectrometer (GC/MS) instruments are used for a wide variety of critical analyses in areas such as food safety, environmental, forensics, and petrochemicals. Historically, helium is the preferred carrier gas for GC/MS. Helium is an inert gas with favorable chromatographic characteristics for high resolution separations that minimize unwanted reactions of analytes in the chromatographic process. Helium is also optimal for use with electron impact (EI) MS in terms of sensitivity. The vast majority of spectra in reference libraries such as NIST are acquired with helium carrier gas.

In recent years, recurring difficulties with the availability and price of helium have resulted in users of GC/MS considering changing to alternative carrier gases. In GC without MS, several different carrier gases have been used successfully. Hydrogen, nitrogen, and argon (with or without methane) have all been used. However, for EI GC/MS, the only practical alternative is hydrogen. Hydrogen has superior chromatographic properties in terms of speed of analysis compared with helium but can have some limitations due to its reactivity. Hydrogen is also compatible with EI MS, although with some drawbacks based on its reactivity and reduced sensitivity.

In general, helium is always a better choice for GC/MS analysis. If helium is available at a reasonable cost, it is clearly preferred. However, if it is not an option, hydrogen can be considered. The purpose of this document is to help users of Agilent EI GC/MS systems determine if hydrogen can be used as a carrier gas for their analyses and what considerations and procedures are necessary to make the transition successful.

# Helium Conservation Techniques

Before deciding to convert your methods to hydrogen carrier gas, it is strongly recommended that you first consider helium conservation measures. Many of the conservation techniques can be implemented quickly and may result in substantial reductions in the rate at which helium is used. Even if you are going to pursue conversion to hydrogen, the helium conservation steps can be used during the transition period to save helium while the new hydrogen methods are being developed and validated.

A few of the key conservation measures will be discussed in this guide, but users are encouraged to review the extensive information regarding helium conservation techniques available from [Agilent.com](http://Agilent.com) at the [Alternate Carrier Gas Solutions](http://AlternateCarrierGasSolutions.com) website for more details.

## CAUTION

**Agilent does not recommend turning off the GC gas flow because the GC/MSD vacuum may draw air through the GC inlet and column into the hot source and quadrupole and damage the MS.**

Agilent recommends using one or more of the following approaches for gas flow during conversion.

- **Gas Saver Mode** - Most GC/MS systems use either the split/splitless (SSL) or multimode (MMI) inlets. The column flow is generally very low, typically ~ 1 mL/min. In addition to the column flow, there is the septum purge, which is typically 3 mL/min. The split vent flow defaults to a constant 50 mL/min rate, but can vary widely depending on the application and can be quite high during split injections and at the end of splitless injections. The Gas saver parameter can be used to reduce the split flow to 20 mL/min after the split or splitless injection has been completed. The split flow then remains at 20 mL/min at all times except during the injection. In many cases the total flow used by the GC/MS system can be reduced by 50% or more by simply using Gas saver.
- **Helium Use Audit** - Agilent recommends that laboratories using helium for multiple systems perform a periodic helium use audit and leak check. Record the helium use of each system and calculate the total flow for all systems. Compare this value with the rate at which helium is used from the cylinder that supplies it. For example, if the total flow for all the helium consuming instruments in a laboratory is 500 mL/min and it is supplied by a cylinder that contains 8,000 L of helium (at STP), the cylinder should last approximately 10 to 11 days. If the cylinder requires changing every five days, then the system should be checked for leaks and unknown points of consumption. Laboratories, especially those with large helium distribution manifolds, that have performed an audit often find substantial savings. As an example, Agilent reduced helium consumption at the Wilmington, Delaware site by 40% after performing an audit.
- **Programmable Helium Conservation Module** - Some GC/MS systems are not in constant use and can spend a significant amount of time in standby condition. For these systems, Agilent offers the Programmable Helium Conservation Module. This device is an option for the Agilent 8890 and 7890 GCs. The module provides the capability to automatically switch the inlet subsystem to nitrogen carrier gas for those times when the GC/MS is not in use. When the GC/MS is going to be used, the module then switches back to providing helium as carrier gas. It typically takes 15 to 30 minutes to purge the nitrogen from the system and be ready for use. Switching between gases is time programmable so that, for example, the system can switch to nitrogen on Friday evening and then automatically revert to helium 30 minutes before the GC/MS is needed on Monday morning. This provides a substantial savings in helium consumption while keeping the instrument ready for use upon short notice.

# Preparing for Conversion to Hydrogen Carrier Gas

Methods that will generally require less optimization include analytes that are:

- Durable compounds such as PAHs, hydrocarbons, and other low reactivity species
- Analyzed at high concentrations
- Analyzed with split injections
- Derivatized

Methods that will generally require more optimization include analytes that are:

- Fragile compounds
- Compounds that react with hydrogen
- Analyzed at trace concentrations

Regardless of the method to be adapted, there will be at least some changes required. Therefore, it is necessary to allot time for the necessary updates to SOPs and method validation.

It is important to recognize the differences between using hydrogen and helium carrier gas. Time should be allotted for adapting the methods, optimization, and resolving potential problems.

There are several topics to consider when converting from helium to hydrogen carrier for EI GC/MS.

Areas that will need attention are discussed in the following sections and include:

- Hydrogen safety
- Source of hydrogen
- Plumbing for hydrogen
- GC/MSD and GC/TQ hardware changes
- Choosing new chromatographic conditions
- Initial startup with hydrogen
- Potential reduction in signal-to-noise ratio (2 to 5 times or more) due to higher noise
- Changes in spectra and abundance ratios for some compounds
- Activity and reactivity with some analytes

# Safety Considerations When Converting to Hydrogen Carrier Gas

Safety is always the first and most important consideration when handling gases. Being an inert gas, helium has relatively few properties of concern. The concerns are mostly related to proper handling of high-pressure cylinders and the possibility of asphyxiation if used in a confined space.

Hydrogen has the added concern of flammability. It is, therefore, necessary to become familiar with the safety aspects of the use of hydrogen with your Agilent GC/MS systems. For detailed safety information see the *Agilent Hydrogen Safety Manual for GC/MS* (part number G7003-90053).

The entire safety manual must be read and understood before connecting and using hydrogen as the carrier gas. Here are some of the key concerns with hydrogen from the document:

- The use of hydrogen as a GC carrier gas, detector fuel gas, or in the optional JetClean system, is potentially dangerous.
- When using hydrogen as the carrier gas or fuel gas, be aware that hydrogen gas can flow into the GC oven and create an explosion hazard. Therefore, be sure that the supply is turned off until all connections are made and ensure that the inlet and detector column fittings are either connected to a column or capped at all times when hydrogen gas is supplied to the instrument.
- Hydrogen is flammable. Leaks, when confined in an enclosed space, may create a fire or explosion hazard. In any application using hydrogen, leak test all connections, lines, and valves before operating the instrument. Always turn off the hydrogen supply at its source before working on the instrument.
- The split vent and septum purge ports for any inlet operated with hydrogen must be connected to a negative flow laboratory vent system to prevent the build up of hydrogen in the laboratory. This is especially important in labs with multiple GCs using hydrogen as the carrier gas.

Hydrogen is potentially explosive and has other dangerous characteristics.

- Hydrogen is combustible over a wide range of concentrations. At atmospheric pressure, hydrogen is combustible at concentrations from 4 to 74.2% by volume.
- Hydrogen has the highest burning velocity of any gas.
- Hydrogen has a very low ignition energy.
- Hydrogen that is allowed to expand rapidly from high pressure can self-ignite.
- Hydrogen burns with a nonluminous flame that can be invisible under bright light.

The Hydrogen Safety manual contains specific details for the safe operation of the GC and MS. Following all necessary safety precautions and all necessary operating parameter differences, it has been demonstrated that the Agilent 597Xx, 7000X, and 7010X series GC/MS systems can be operated safely with hydrogen as carrier gas rather than helium.

**Figure 1** shows a letter from an Agilent safety engineer. It describes the hydrogen safety features of the Agilent 8890 GC. Letters are also available for the Intuvo 9000 and 7890 GCs. These may be helpful if requested by your facility safety personnel.

Some of the key hydrogen safety features of Agilent 8890 and 7890 GCs and MS systems are:

- **Safety shutdown** - When the gas pressure setpoints are not met, the valve and heater are shut off to prevent explosion
- **Flow limiting frit** - If a valve fails in the open position, an inlet frit limits the flow



- **Oven ON/OFF sequence** - A fan purges the oven before turning on heater to remove any collected hydrogen
- **Explosion test** - The GC and MS are designed to contain parts in case of explosion
- **Hydrogen sensor (optional)** - Agilent offers an optional oven hydrogen sensor accessory for its GCs. The hydrogen sensor will shut down the system if a hydrogen leak is detected in the oven, avoiding a possible hazardous situation. Benefits offered by the sensor include:
  - Integration into gas chromatograph
  - Detect potential leaks early to bring the system to a safe standby
  - Integrated calibration of sensor to ensure long-term reliability



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April 15, 2019

Subject: Use of Hydrogen in the 8890 Gas Chromatograph (GC)

Dear Customer:

I am pleased to respond to your request for information concerning the use of hydrogen as a carrier gas in the 8890 Gas Chromatograph produced by Agilent Technologies.

This instrument was designed to use hydrogen as a carrier gas. The Agilent 8890 Gas Chromatograph Safety Manual and the operation manual for the instrument contain safety instructions; but it is recommended that anyone working with flammable or explosive gases take a lab safety course covering proper gas handling and use.

Some laboratory precautions that are recommended for controlling hydrogen build up include directing vent lines into a fume hood and leak-testing the gas connections, lines, and valves before operating the instrument. Because hydrogen leaks frequently originate in tubing and connections external to the gas chromatograph (e.g., at the tank), hydrogen leak-testing throughout the lab should be performed at least weekly and whenever a tank is changed.

The Agilent 8890 gas chromatograph is not designed for use in hazardous atmospheres, but the GC has built-in safety features to reduce the risk of and the potential for injury from oven explosions when used in a standard laboratory environment. Enclosed is a set of frequently asked questions which should answer your questions about the use of hydrogen in the oven.

Lastly, it should be noted that we have not received any reports of injuries due to the use of hydrogen in this instrument.

Agilent appreciates your interest in ensuring safe use of your instruments. Should you require any additional information on this subject, please do not hesitate to contact us.

Sincerely,

Andrew Deionno  
Product Safety Engineer

Figure 1. Letter from Agilent safety engineer regarding suitability of 8890 Gas Chromatograph for use with hydrogen carrier gas.

# Sources of Hydrogen for Use as Carrier Gas

As with any carrier gas, it is important to use only ultra-high purity hydrogen. There are several grades of hydrogen available for applications like welding, which are unsuitable for use as carrier gas because of the levels of impurities such as water and oxygen.

There are two main approaches for providing hydrogen for a GC/MS system:

- High pressure gas cylinders
- Hydrogen generators

## High pressure cylinders

Cylinders are often less expensive initially. If you are evaluating hydrogen for the first time and are not certain you will be adopting it for routine use, cylinders may offer the simplest way to try hydrogen. Cylinders can be used while developing and testing new hydrogen carrier methods. Once the methods are determined, the hydrogen flow and pressure requirements of the methods can then be used to select a hydrogen generator, if desired.

Make sure to obtain hydrogen with a purity specification of 99.9999% or greater and with low oxygen and water levels. Also, use a cylinder regulator designed for use with high purity hydrogen applications. Consult your gas supplier for selecting an appropriate regulator.

### NOTE

**There may be restrictions on the use and placement of high pressure hydrogen cylinders in your laboratory, so it is a good idea to check with your facility safety personnel before purchasing any cylinders or regulators.**

## Hydrogen generators

Hydrogen generators are an alternative way of providing hydrogen carrier gas. They typically have a higher initial cost than cylinders but can be more economical over time.

As with cylinders, there are many different generators available with varying specifications of hydrogen purity. Only those with a purity specification of 99.9999% or greater and with low oxygen and water levels should be considered. When selecting a hydrogen generator, make sure the maximum delivery pressure and flow rate are high enough to meet the needs of your chromatographic methods and all the simultaneously operating instruments that will be supplied by the hydrogen generator.

Hydrogen generators also offer some useful safety features:

- Hydrogen is only generated at the needed pressure (for example 40 psi)
- Maximum flow is limited (for example 250 mL/min)
- Auto-shutdown if the setpoint pressure cannot be maintained
- Minimal stored gas (for example 50 mL at 40 psi) of hydrogen at any one time

# Plumbing the Instrument for Hydrogen Carrier Gas

## Carrier gas to the GC

Before making any plumbing changes, make sure that the GC and MS are turned off. The plumbing to provide hydrogen to the instrument must be clean and free of leaks. Dirty tubing can cause serious contamination problems. Chromatographic quality stainless steel tubing and fittings are often recommended for hydrogen plumbing and are the best choice if available.

### NOTE

**You may have to follow local codes or internal company guidelines.**

Copper tubing can also be used during the evaluation of hydrogen for conversion of methods. If copper tubing is used, it should be new 1/8-inch tubing, as old copper tubing becomes brittle and can break. Agilent sells new 1/8-inch copper tubing that has been cleaned for GC use (5180-4196 for 50 feet). If it is determined hydrogen will be used on a permanent basis, the system should be plumbed with stainless steel tubing and fittings.

Once the plumbing is completed, but with the GC turned off, pressurize the system briefly with hydrogen and check all connections with an electronic leak detector such as the Agilent G3388A Leak Detector. This step is necessary because the standard GC/MSD air and water background leak checks will not always find big outgoing hydrogen leaks.

When plumbing a hydrogen generator it is necessary to insert a moisture trap between the output of the generator and the GC. This is required to protect the GC EPC module in the event of water contaminating the hydrogen supplied by the generator.

### NOTE

**Hydrogen generators require periodic preventative maintenance. It is important that this be performed to prevent problems.**

Measurement of background levels of water, nitrogen, and oxygen levels should be performed later after all plumbing and column installation are completed, the system has been purged, and the GC/MS system pumped down with low temperature settings.

## Vent/Exhaust lines

The split vent and septum purge vent for each installed inlet are located on top of the 8890, 8860, and 7890 GC. (Figure 2) These vents should be connected to a negative flow laboratory vent system. Use tubing of a large enough inside diameter to assure that no backpressure is built up, as this can cause problems. The septum purge flow is generally very low (3 mL/min) but the split vent flow can be as high as several hundred mL/min.

### NOTE

**The longer the vent line, the larger the id must be to avoid building pressure. In practice, 1/8-inch od × 1.65 mm id copper tubing works well for the split vent line if it does not exceed 2 m. For longer lengths, consider using larger id tubing.**

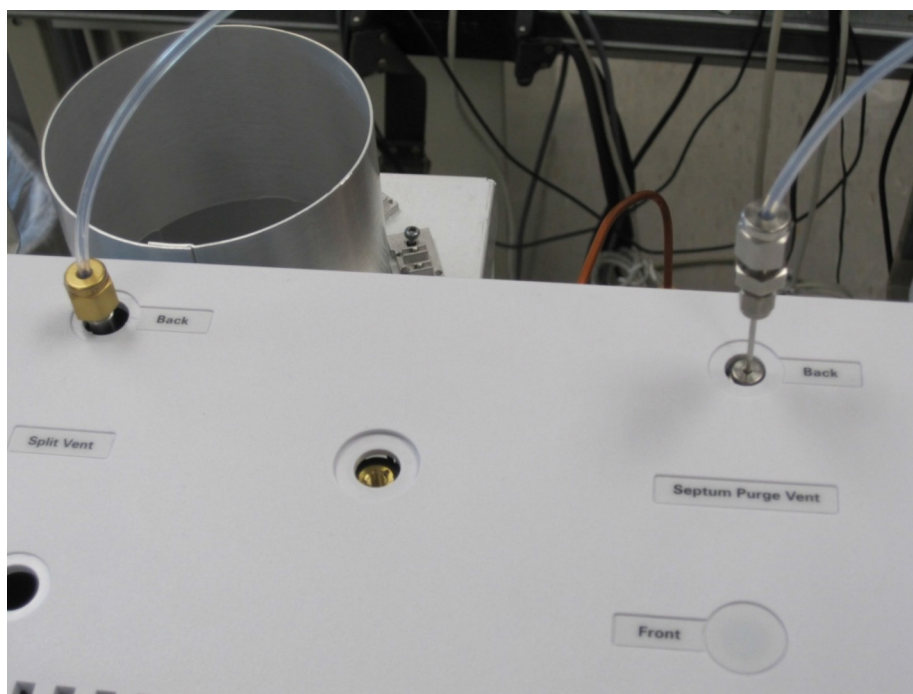


Figure 2. The split vent and septum purge vent for each installed inlet should be connected to a negative flow laboratory vent system.

## Foreline pump exhaust

The foreline pump exhaust should be connected to a negative flow laboratory vent system. If the foreline pump is not currently set up this way, purchase a barb fitting, O-ring, and tubing. These can be purchased from Agilent:

- 3/8-inch male NPT to barb fitting, part number G3170-80006
- 20 feet of 1/2-inch tubing, part number G3170-60100
- O-ring for the barb fitting, part number 0905-1193

Additional parts that may be required can be ordered from the Agilent GC and GC/MS Consumables catalog.

When installing the tubing, try not to exceed 20 feet in length and make sure there are:

- No restrictions
- No kinks
- Very few sharp radius bends
- No low spots where oil could pool

**Figure 3** shows a diagram of the plumbing for use with hydrogen carrier gas.

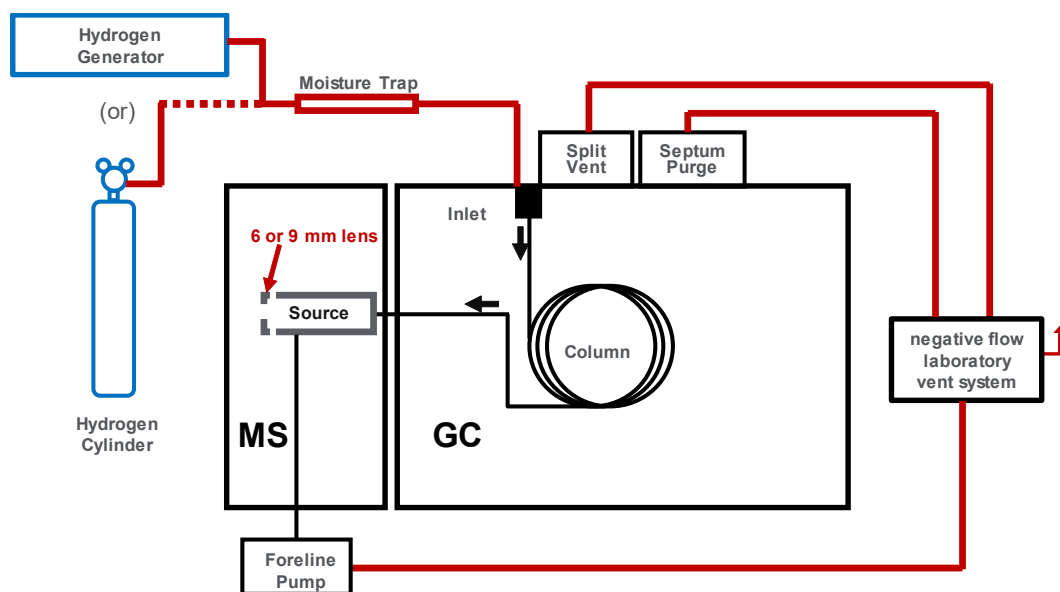


Figure 3. Diagram of the plumbing for use with hydrogen carrier gas.

# MS Source Components Required for Conversion

## MS Source Changes Required for Conversion

When converting the GC/MS from helium to hydrogen carrier gas, it is usually necessary to make changes in the source to optimize performance. The changes to be made depend on the model of MS and type of source it has. Here is a summary of currently available options:

- High-efficiency source (HES) in the Agilent 5977B/C GC/MSD or the Agilent 7010A/B/C triple quadrupole GC/MS. No changes required.
- Agilent 5975 Series GC/MSD with stainless steel or inert source EI source. Change drawout lens to 9 mm (or 6 mm).
- Agilent 5977/B/C GC/MSD or Agilent 7000C/D/E triple quadrupole GC/MS with inert plus (extractor) sources. Replace extractor source with the Agilent Hydrolnert source (recommended) or with inert 9 mm (or 6 mm) extractor lens.

Changing to hydrogen carrier gas has resulted in some problems being observed. Many were originally thought to be in the inlet or column, but experience has since shown that, while there can be inlet or column problems, most problems are related to the source. The problems result from in-source reactions due to the presence of heat, hydrogen, and the metal source components. The metal components can act as a catalyst for reactions like hydrogenation or dechlorination. These reactions can result in tailing, unwanted changes in ion ratios and spectra, and problems with calibration linearity.

Agilent CrossLab can support this conversion for you with knowledge transfer and troubleshooting tips and tricks for your workflow using hydrogen. Ask your sales representative about our method optimization service.

Agilent extractor, inert, and stainless steel EI MS sources all come standard with a 3 mm drawout lens (inert and stainless steel sources) or 3 mm extractor lens (extractor source). The 3 mm diameter works well with helium carrier gas but can cause problems when used with hydrogen. The most active area causing the in-source reactions (but not the only one) is metal around the aperture in the lens. Thus, expanding the aperture diameter to 9 mm significantly reduces the problems. Agilent offers both 6 and 9 mm lenses for this purpose. The 9 mm lens is recommended for most applications with hydrogen carrier gas, as it provides the best overall performance, balancing linearity, sensitivity, peak shape, and source reactivity. The 6 mm lens can be used if greater sensitivity is required, but it is best to start with the 9 mm first, as the 6 mm will have somewhat increased activity.

## Hydrolnert source

While the use of a 9 mm lens in the source reduces in-source reactions, it is not a complete solution to the problems. There are still many compounds affected to varying degrees. For this reason, Agilent has developed a new source technology called the Agilent Hydrolnert source.

The Hydrolnert source addresses in-source reaction problems; for the metal source parts, it uses a proprietary material that greatly reduces catalytic activity and tailing. The Hydrolnert source is installed in place of the inert plus (extractor) source using all the same connections.

Nitrobenzene is an example of a compound that is readily hydrogenated in the source with hydrogen present. This hydrogenation reaction produces aniline from nitrobenzene in the source. The result is dramatic changes in the relative ratios of ions. **Figure 4** shows the problem, and the improvements made using Hydrolnert. With hydrogen carrier without Hydrolnert, the spectrum more closely matches that of aniline instead of nitrobenzene. This problem persists even with the 9 mm extractor or drawout lens. Without Hydrolnert, the 93 ion is much too big and is tailing. The

65 ion also tails. Without Hydrolnert, the library match score (LMS) is only 68 relative to the nitrobenzene spectrum in the NIST 20 library. With Hydrolnert, the relative ratios of the ions more closely match those of the helium spectrum in NIST 20 and the tailing of the 93 and 64 ions is significantly reduced. Now the LMS is a much more acceptable 94.

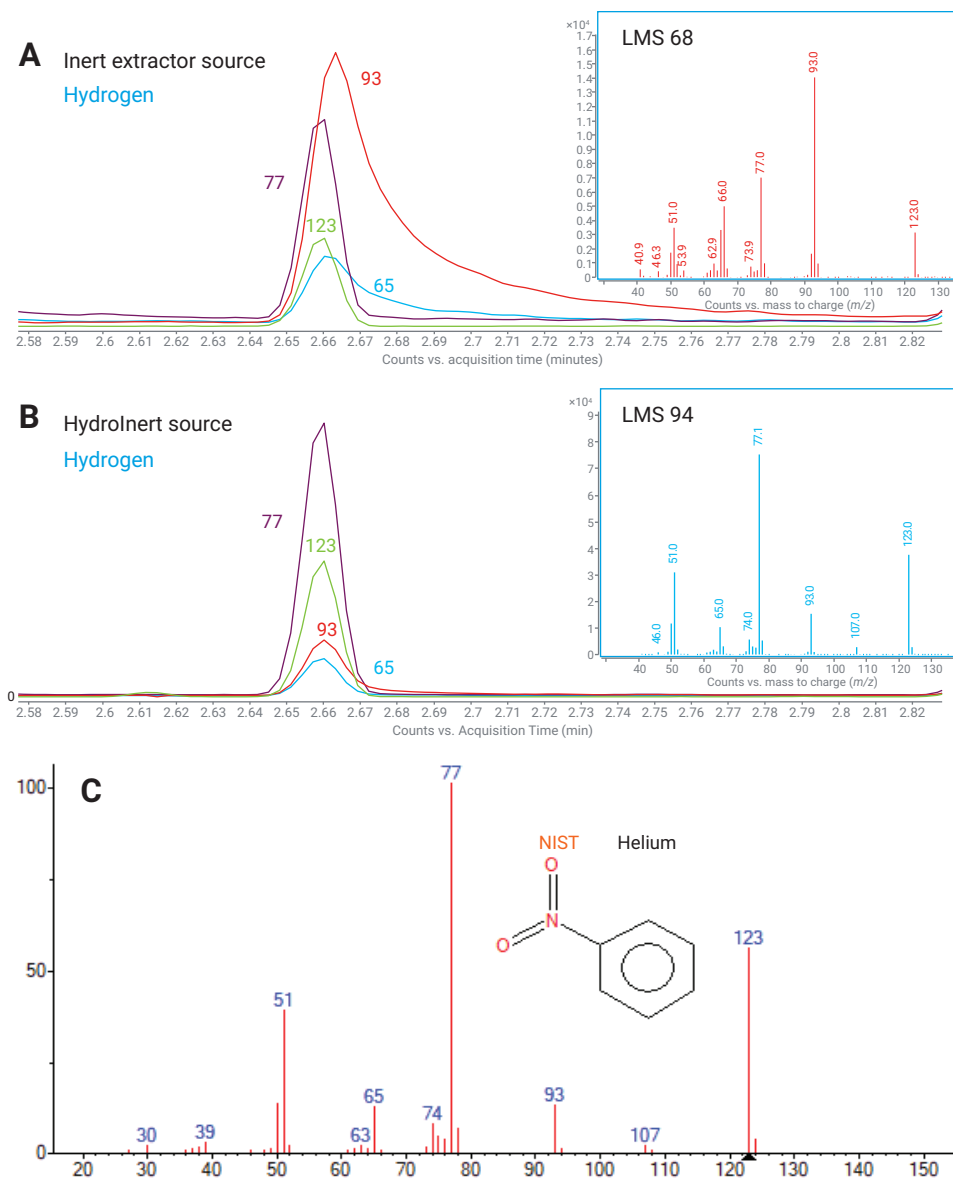


Figure 4. (A) Extracted ion chromatograms (EICs) of principle nitrobenzene ions with hydrogen carrier, Agilent inert extractor source, and 9 mm lens. (B) EICs of principle nitrobenzene ions with hydrogen carrier, Agilent Hydrolnert source, and 9 mm lens. (C) NIST 20 library spectrum of nitrobenzene with helium carrier.

Agilent also offers Hydrolnert 6 and 3 mm extractor lenses for use with the Hydrolnert source. These can be used to increase the signal-to-noise ratio (SNR) of analytes, but at the cost of increased reactivity for some compounds. If one of these is chosen, it is important to determine if increased reactivity is a problem for any analytes relative to the recommended 9 mm lens.

## HydroInert resources

The following links contain more information about the HydroInert source. The first link to the HydroInert product page lists the part numbers and pricing information. It is recommended that the complete HydroInert source assembly be purchased (part number G7078-67930 for the 5977 GC/MSD, or part number G7006-67930 for the 7000 GC/TQ).

- HydroInert product page (lists part numbers, pricing, and links to technical information):  
<https://www.agilent.com/en/product/gas-chromatography-mass-spectrometry-gc-ms/gc-ms-supplies-accessories/hydroinert-source-for-hydrogen-carrier-gas-on-gc-ms>
- HydroInert technical overview (detailed description of HydroInert and its performance characteristics):  
<https://www.agilent.com/cs/library/technicaloverviews/public/te-hydroinert-source-5994-4889en-agilent.pdf>
- EPA Method 8270 semivolatile organic compounds (SVOCs) with the 5977 GC/MSD using hydrogen carrier gas and the HydroInert source:  
<https://www.agilent.com/cs/library/applications/an-svoc-hydrogen-carrier-gas-hydroinert-gcms-5994-4890en-agilent.pdf>
- EPA Method 8270 SVOCs with the 7000E GC/TQ using hydrogen carrier gas and the HydroInert source:  
<https://www.agilent.com/cs/library/applications/an-svoc-hydrogen-carrier-gas-hydroinert-7000e-gcms-5994-4891en-agilent.pdf>
- Volatile organic compounds (VOCs) with the Agilent 8697 headspace sampler and the 5977C GC/MSD using hydrogen carrier gas and the HydroInert source:  
<https://www.agilent.com/cs/library/applications/an-voc-headspace-hydroinert-gc-msd-5994-4963en-agilent.pdf>
- Accelerate productivity, improve outcomes-CrossLab method and application services for the HydroInert source:  
[https://www.agilent.com/cs/library/flyers/public/flyer\\_method-application-services\\_5991-7028en\\_agilent.pdf](https://www.agilent.com/cs/library/flyers/public/flyer_method-application-services_5991-7028en_agilent.pdf)



## Inert plus (extractor), inert, and stainless steel EI sources

As mentioned previously, the use of an inert 9 mm (or 6 mm) extractor or drawout lens significantly reduces the problems with hydrogen carrier gas for many, but not all, compounds. If the HydrolInert source is not applicable (inert and stainless steel EI sources) or if a less costly option is desired for the extractor, the part numbers for the optional 9 and 6 mm lenses are listed in Table 1. Since the likelihood of problems is compound dependent, it is important to evaluate all target analytes. For systems that are used to identify untargeted unknown compounds, or for trace-level analyses like pesticides, the HydrolInert source is recommended.

In general, hydrogen flows <1.0 mL/min are suitable for 6-mm lens. For hydrogen flows >1.0 mL/min and <1.4-mL/min, the 9-mm lens is required. Higher flows are not recommended for data acquisition.

**Table 1** Optional lens part numbers for the extractor, inert, and stainless steel EI sources.

	6-mm	9-mm
Stainless Steel Drawout Lenses	G3163-20530	*
Inert Drawout Lenses	G2589-20045	G3440-20022
Extractor Lenses	G3870-20448	G3870-20449

\* A stainless steel 9-mm drawout lens is not available but the 9-mm Inert drawout lens, p/n: G3440-20022, can be used with a stainless steel source.

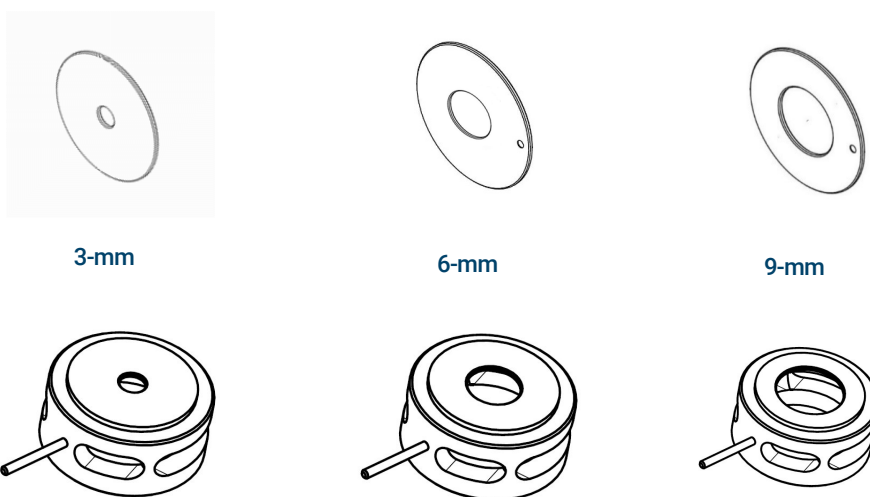


Figure 5. Drawout/extractor lenses for the extractor, inert, and stainless steel EI sources. Only the 6- or 9-mm (preferred) should be used with hydrogen carrier gas.

Additional parts required can be ordered from [www.agilent.com](http://www.agilent.com).

# Choosing a Column and Method Conditions

## MS Hydrogen pumping capacity

The initial step in choosing a column and method conditions for use with hydrogen carrier gas is to determine the flow rate of hydrogen your system can effectively pump. The pumping capacity for hydrogen is approximately 1/2 that of helium. This limits the choice of columns and flows. When choosing a GC/MS system for conversion to hydrogen carrier gas, turbo pumps are preferred over diffusion pumps because their added pumping capacity allows greater flexibility in column flows. **Table 2** lists the optimal, maximum recommended, and maximum flows for hydrogen with Agilent GC/MS systems.

**Table 2 Hydrogen flow rate recommendations for Agilent GC/MS systems.**

	Optimal flow (mL/min H <sub>2</sub> )	Maximum Recommended Flow (mL/min H <sub>2</sub> )	Maximum Flow (mL/min H <sub>2</sub> )
597Xx diffusion pump	~0.5	0.75	1.00
597Xx turbo pump	0.5 to 1.0	2.0	3.25
7000x/7010x	0.5 to 1.0	2.0	3.25
7200x/7250x	Hydrogen use not permitted		

It is helpful to have an ion gauge on the MS to monitor the vacuum versus column flow. Try to avoid flows that produce pressures higher than  $5 \times 10^{-5}$  Torr. Source performance starts to degrade above this pressure, and you may not get useful data.

Do not exceed the maximum flow into the MS at any time. If your method will use a pressure pulsed injection, verify the maximum column flow during the pulse does not exceed the listed maximum flow.

For methods that will use retention time locking (RTL), do not create the method with the nominal flow at the maximum listed. Since RTL requires changing the flow to match retention times, the locking flow for a different column or system may exceed the maximum flow. Create RTL methods with flow rates at least 30% below the maximum for your system to avoid this problem.

# Choosing a Column and Method Conditions

## Introduction

There are two major differences with using hydrogen versus helium as a GC carrier gas.

- The first is the viscosity of hydrogen is lower than that of helium, meaning that the inlet pressure to obtain the same flow rate with hydrogen is significantly lower. Column dimensions and flow setpoints must be chosen that maintain adequate inlet pressures, preferably 5 psi or greater, to permit precise control of column flow and retention times. Sub-ambient inlet pressures must be strictly avoided as these can cause inlet flow shutdowns and may damage inlet liners and columns due to air being drawn into the system.
- The second difference is in chromatographic performance. Hydrogen offers advantages for resolution and speed of analysis over helium. This results mainly from the shape of Van Deemter curve for hydrogen.

To determine appropriate column dimensions for the system, here are some important factors to consider:

- 1 If possible, use constant flow methods with GC/MS systems. Constant pressure methods with an oven temperature program have a negative impact on MS performance due to the changing flow into the source.
- 2 Determine the hydrogen pumping capacity of the MS hardware. With diffusion pump systems, try to use flows in the range of 0.5 to 0.7 mL/min and for turbo pump systems, 0.5 to 2 mL/min.
- 3 Optimal MS sensitivity and performance are obtained with 0.8 to 1.2 mL/min. Try to use a column flow in this range if you have a turbo pump.
- 4 The inlet pressure should be 5 psi or higher in the temperature range over which the analysis is run. At 25 °C, the inlet pressure should at least be 1 psi to avoid a carrier gas shutdown when the oven door is open or when the oven heater is off.
- 5 The optimum column average velocity range for helium is 20 to 40 cm/sec. For hydrogen, it is 30 to 55 cm/sec. If the flow of helium or hydrogen is too low, peak resolution is lost much faster than if it is too high. Always operate at or above the minimum average linear velocity if possible (see **Figure 6** on page 22).

# Simplified Approach to Column Conversion

If the current helium method uses a 30 m × 0.25 mm id × 0.25 µm film thickness capillary column, it may be possible to set up the hydrogen conversion method quickly with the following steps. This is a good starting point that can be optimized later, if necessary:

- 1** Purchase a 20 m × 0.18 mm id × 0.18 µm film thickness version of the current column with the same stationary phase type (DB-5ms, HP-5ms, etc.). Note, if the 30 m method uses a different film thickness than 0.25 µm, try to find a 0.18 mm id column with a phase ratio closest to the current one.
- 2** If the MS has a diffusion pump, set the hydrogen flow to 0.6 mL/min constant flow. If the MS has a turbo pump (preferred), set the hydrogen flow to 0.9 mL/min constant flow.
- 3** Set the new temperature program to the same values as in the current helium method. (This can be optimized later to speed up the method, if desired.)
- 4** If the helium method uses a pressure pulsed injection, and the MS has a turbo pump, set the pulse pressure of the new method so as not to exceed 3.25 mL/min during the pulse. Pulsed injections are not recommended with diffusion pumps due to insufficient pumping capacity.
- 5** If the MS has a high vacuum gauge, check to see if the pressure is  $>5 \times 10^{-5}$  Torr. If it is, reduce the flow until it is less than  $5 \times 10^{-5}$  Torr.
- 6** The solvent elution time will be different, so determine a new solvent delay time for the method.
- 7** After conditioning the system as described later, run a calibration standard and find the new retention times of the analytes.

The following sections provide more details on parameters that can be adjusted to optimize the converted methods.

# Column Dimensions

It is often necessary to change to a column of different dimensions when converting a GC/MS method from helium to hydrogen carrier gas. This is due to hydrogen's viscosity being lower than that of helium, meaning that the inlet pressure to obtain the same flow rate with hydrogen is significantly lower.

For example, 30 m × 0.25 mm id × 0.25 μm film thickness columns are widely used with helium in GC/MS applications. These columns are generally not suitable with hydrogen, because of insufficient inlet pressure as indicated in **Table 3**. **Table 3** lists the inlet pressures required for a flow of 1.0 mL/min for columns of various dimensions at ambient temperature (25 °C) and 100 °C with both helium and hydrogen carrier.

**Table 3** Inlet pressures required for a flow of 1.0 mL/min with helium and hydrogen.

Column Dimensions	Inlet Pressure (psi) for He at 25 °C	Inlet Pressure (psi) for He at 100 °C	Inlet Pressure (psi) for H <sub>2</sub> at 25 °C	Inlet Pressure (psi) for H <sub>2</sub> at 100 °C
30 m × 0.25 mm id	6.36	10.75	-0.56	2.40
60 m × 0.25 mm id	15.09	21.29	5.29	9.48
20 m × 0.18 mm id	18.47	25.39	7.57	12.22
40 m × 0.18 mm id	32.21	41.99	16.79	23.37

## NOTE

**The 30 m × 0.25 mm id column has a low inlet pressure (2.4 psi) with hydrogen at an oven temperature of 100 °C. At ambient, the inlet pressure would be sub-atmospheric and therefore not usable.**

A 60 m × 0.25 mm id column would give acceptable inlet pressures, provide better chromatographic separation, and have increased capacity, but would result in longer run times.

The 20 m × 0.18 mm id is a common choice for hydrogen use. It has acceptable inlet pressures with hydrogen and provides similar, and often better, chromatographic resolution than the 30m × 0.25 mm id columns. The sample capacity is about 1/3 of that for the 0.25 mm id column, so the amounts injected may need to be adjusted. These columns are a good starting point for method development.

**Figure 6** shows the Van Deemter curves for nitrogen, helium, and hydrogen. The plots have the height equivalent of a theoretical plate (H) as the Y-axis, and average velocity of the carrier gas as the X-axis. Optimal chromatographic resolution with a specific carrier gas is obtained near the minimum of the Van Deemter curve.

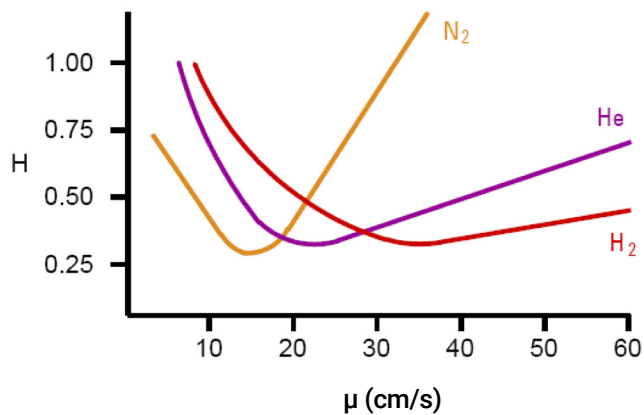


Figure 6. Van Deemter curves for nitrogen (N<sub>2</sub>), helium (He), and hydrogen (H<sub>2</sub>).

The optimum column average velocity range for helium is 20 to 40 cm/sec. For hydrogen, it is 30 to 55 cm/sec. However, since the slope of the Van Deemter curve for hydrogen is relatively flat with linear velocity, only a small amount of resolution is lost with increasing average velocity above the optimal value. This is helpful when optimizing the speed of analysis.

**NOTE**

**If the flow of helium or hydrogen is too low, you lose efficiency much faster than if it is too high. Therefore, operating at or above the minimum average velocity is important.**

# Flow Calculator Tool

As an aid to developing new GC methods, Agilent offers free downloadable calculators for the GC. Before installing new columns to try with hydrogen, it may be helpful to use the calculators to determine if they would be a good fit. The calculators are available at:

<https://www.agilent.com/en-us/support/gas-chromatography/gccalculators>

The Pressure Flow Calculator tool is shown in **Figure 7** and **Figure 8**. With the column dimensions, carrier gas type, column outlet pressure, and oven temperature entered, the average velocity can be entered to see what the inlet pressure and flow would be. In **Figure 7** entering an average velocity of 35 cm/sec (in blue) would give near optimal resolution, but the column flow and inlet pressure are unusably low (in red). In **Figure 8** the inlet pressure is entered as 5.0 psi (in blue) resulting in an acceptable linear velocity of 55.056 cm/s (in green). With this pressure, the outlet flow and inlet pressure are also at acceptable values.

Parameter	Value
Length (m)	20.00
Inner Diameter (µm)	180
Film Thickness (µm)	0.18
Temperature (°C)	50
Inlet Pressure (gauge)	-2.175
Outlet Flow (mL/min)	0.279
Average Velocity (cm/s)	35.000
Outlet Pressure	0.000
Split Vent Flow (mL/min)	0.000
Split Ratio (vent flow/col flow)	0.000 : 1
Holdup Time	0.95 min
Inlet Temp (°C)	175
Inlet Liner Flow	0.537
Liner Volume (µL)	900
Suggested Splitless	1.7 min
Carrier Gas	Hydrogen
Optimum velocity range (cm/s)	30 - 55

Figure 7. Using Agilent Pressure Flow Calculator to determine chromatographic conditions for 20 m × 0.18 mm id × 0.18 µm film thickness column with average velocity set near optimum 35 cm/sec.

Parameter	Value
Length (m)	20.00
Inner Diameter (µm)	180
Film Thickness (µm)	0.18
Temperature (°C)	50
Inlet Pressure (gauge)	5.000
Outlet Flow (mL/min)	0.690
Average Velocity (cm/s)	55.056
Outlet Pressure	0.000
Split Vent Flow (mL/min)	0.000
Split Ratio (vent flow/col flow)	0.000 : 1
Holdup Time	0.61 min
Inlet Temp (°C)	175
Inlet Liner Flow	0.845
Liner Volume (µL)	900
Suggested Splitless	1.1 min
Carrier Gas	Hydrogen
Optimum velocity range (cm/s)	30 - 55

Figure 8. Using Agilent Pressure Flow Calculator to determine chromatographic conditions for 20 m × 0.18 mm id × 0.18 µm film thickness column with inlet pressure entered at 5 psi.

Other useful values provided are the holdup time and a suggested splitless time calculated based on the entered liner volume and inlet liner flow rate.

Another example of the usefulness of the calculator is determining the maximum pressure that can be used for pulsed injections. With the possibility of analytes reacting with hydrogen in the hot inlet, pulsed injections are worth considering. Pulsed injections also aid in transferring analytes from the inlet to a narrower column. **Figure 9** shows the calculation of the pulse pressure (in green) at the maximum allowable flow with a turbo pump system, 3.25 mL/min (in blue).

Pressure Flow Calculator

Length (m)	20.00	Split Vent Flow (mL/min)	0.000
Inner Diameter (µm)	180	Split Ratio (vent flow/col flow)	0.000 : 1
Film Thickness (µm)	0.18	Holdup Time	0.28 min
Temperature (°C)	50	Inlet Temp (°C)	175
<b>Inlet Pressure (gauge)</b>	<b>28.044</b>	Inlet Liner Flow	1.833
<b>Outlet Flow (mL/min)</b>	<b>3.250</b>	Liner Volume (µL)	900
Average Velocity (cm/s)	119.471	Suggested Splitless	0.5 min
Outlet Pressure	0.000	Carrier Gas	Hydrogen
Pressure Units	<input type="radio"/> 1 Atm <input checked="" type="radio"/> Vacuum <input type="radio"/> Other	Optimum velocity range (cm/s)	30 55
	<input type="radio"/> KPa <input checked="" type="radio"/> psi <input type="radio"/> bar		

Figure 9. Calculation of the pulse pressure at 3.25 mL/min, the maximum allowable flow with a turbo pump system.

For the conditions used in the example, the calculator indicates that the maximum pulse pressure would be 28 psi. Because the inlet liner flow is higher, the suggested splitless time is lowered by more than two fold.

The Flow Calculator cannot be used for evaluating Intuvo GC flows due to the extra restrictions in the flow path. The Intuvo acquisition software must be used to determine flows.



# Method Translator

The Method Translator is useful for optimizing speed improvements when converting from helium to hydrogen. It is designed to evaluate different sets of conditions while maintaining the same elution order and relative elution times.

The method translation tool works best with constant pressure methods in that it accurately predicts the retention times on the new column or same column with different parameters. Since only constant flow methods are recommended for GC/MS, the predicted retention times are not as accurate, and some analytes may reverse elution order. However, the process is still very helpful.

Here are a few things to consider when using the Method Translator:

- If evaluating moving to a different column, the phase chemistry (DB-5, HP-5ms, Innowax etc.) must be the same.
- The process works best with columns of the same phase ratio. If the same phase ratio is not available for the new column, pick one with the closest ratio.
- When increasing the speed of a method, any oven ramps are increased by the factor of the speed gain. Be sure to check if your oven can ramp at the new rate. If not, translate to a lesser speed gain that gives an oven ramp the system can maintain.
- Keep the flow rate in the ranges suggested previously as dictated by the MS pumping system.

**Figure 10** shows the Method Translator evaluating moving from a 30 m × 0.25 mm id column with helium to a method with hydrogen carrier gas on the same column, but with a 2x gain in speed (that is, all the retention times on the new method would be approximately 1/2 of those in the original method).

The drawback with this translation is that it requires a relatively high flow, 1.8 mL/min, into the MS source. This is usable if the system has a turbo pump but would result in lower sensitivity. At 1.8 mL/min constant flow, the inlet pressure both during the analysis and with the oven off would also be acceptable.

## NOTE

**If the MS has an HES (high efficiency source) this flow rate would be too high.**

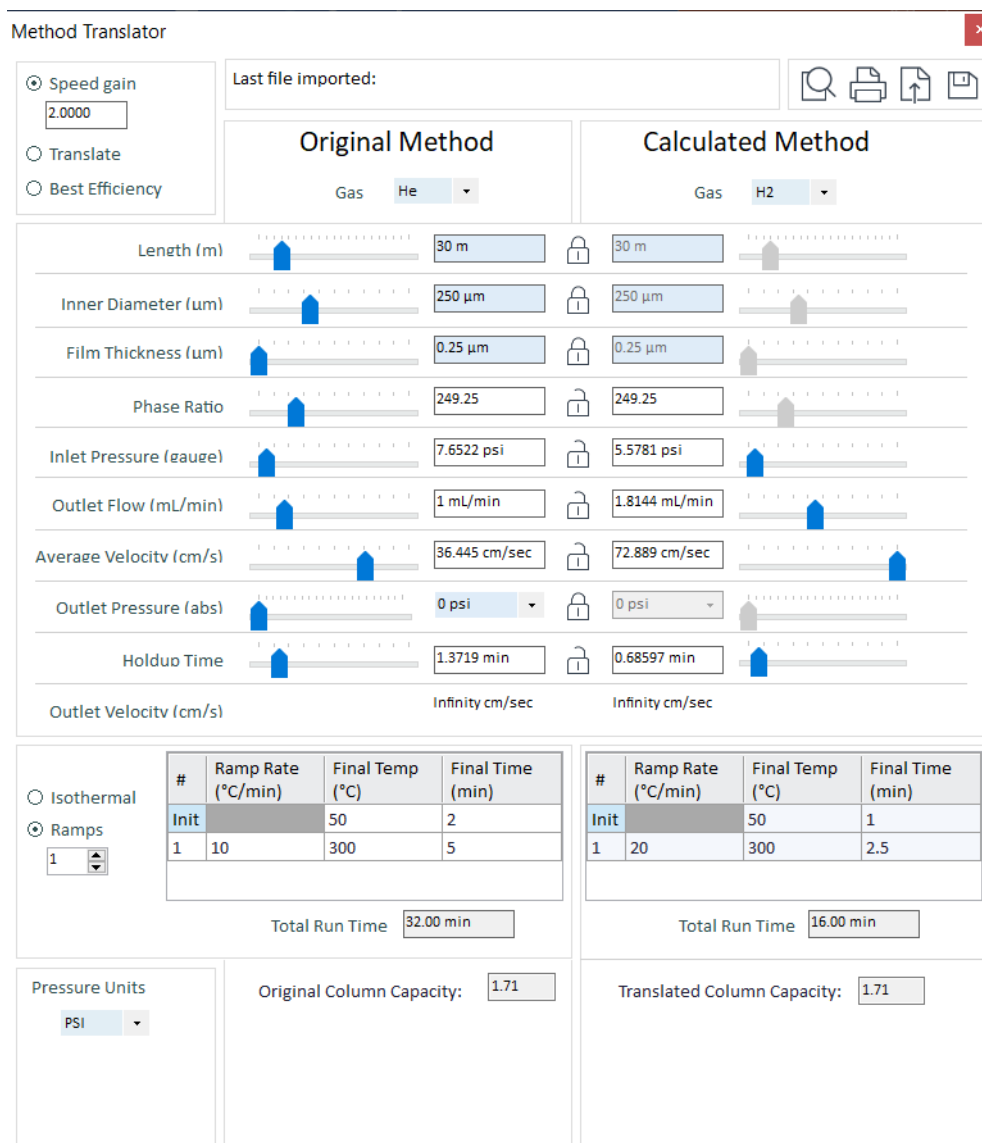


Figure 10. Method translation tool evaluating moving from a 30 m × 0.25 mm id column with helium to a method with hydrogen carrier on the same column with a 2x gain in speed.

**Figure 11** shows the translation of the method to a 20 m × 0.18 mm id column with hydrogen carrier gas with a 2.5x gain in speed.

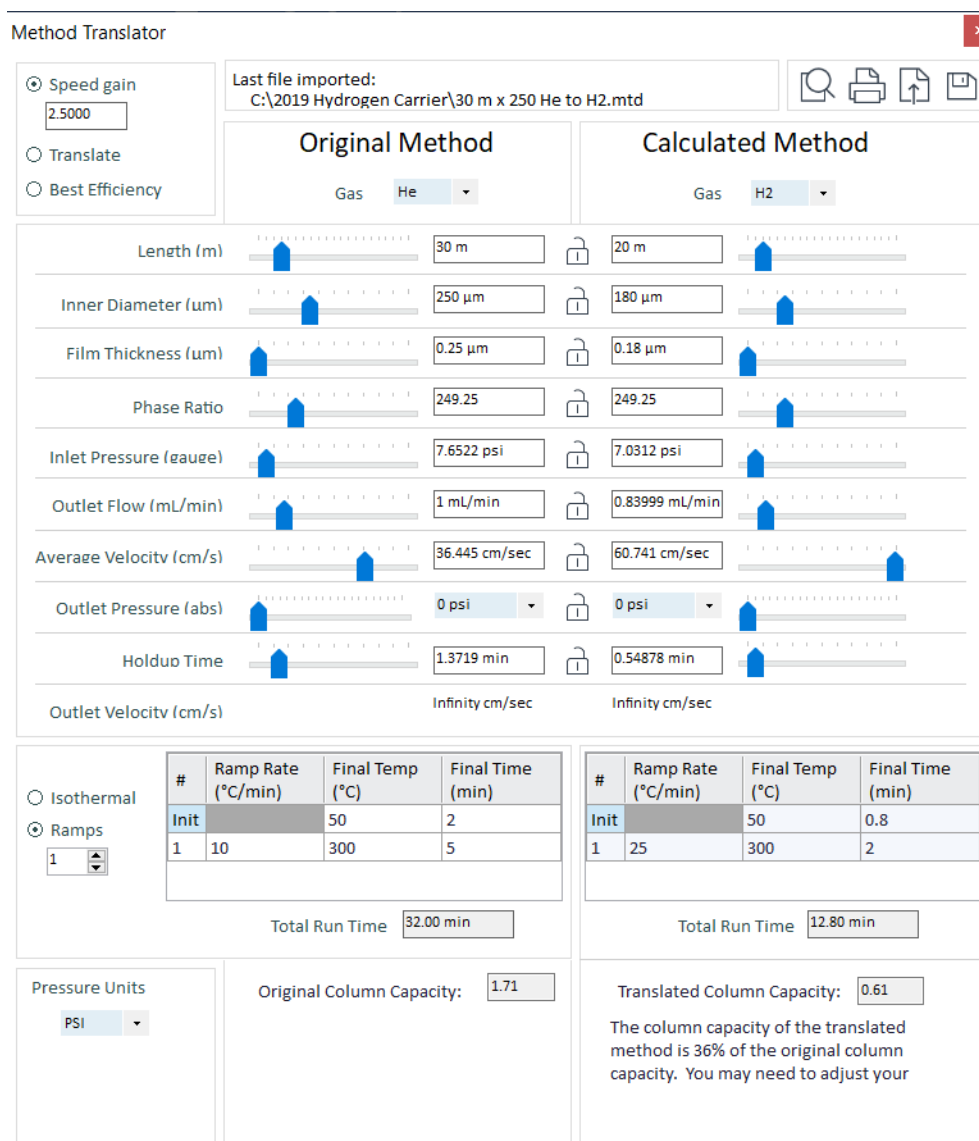


Figure 11. Translation of the method to a 20 m × 0.18 mm id column with hydrogen carrier gas with a 2.5x gain in speed.

With the translation to the 20 m × 0.18 mm id column, a speed gain of 2.5 is achievable with a flow rate in the preferred range. In the bottom right corner, the tool lists the translated column capacity, indicating the new column only has 36% of that with the original column. This method would be a good starting point for the conversion.

If the translation yields a ramp rate that is not attainable with the GC, try using a ramp rate that works (that is, does not cause the oven to go "Not Ready" during the analytical run) and raise the flow to a value in the acceptable range as discussed previously.

As with the Flow Calculator, the Method Translator cannot be used for evaluating Intuvo GC parameters due to the extra restrictions in the flow path. Use the Intuvo acquisition software to determine flows.

# Initial Startup with Hydrogen Carrier Gas

After the system is set up with:

- An appropriate supply of hydrogen connected to the instrument and leak checked.
- The source has the correct larger diameter (preferably 9-mm) drawout or extractor lens installed.
- The column is chosen, installed, the appropriate flows set, and leak checked.
- Pumped down for at least one hour with heated zones (inlet, transfer line, source etc.) turned off.
- Heated zones brought to method temperature and allowed to stabilize for one hour.
- MS is tuned.

The system can be started, and testing of the method can begin.

There are typically three problems observed upon initial startup.

- High spectral background with ions at most masses up to about 300  $m/z$ .
- Reduced signal-to-noise resulting in a worse MDL.
- Significant tailing for many compounds.

## High spectral background

**Figure 12** shows a typical background spectrum acquired with hydrogen carrier gas. The spectrum has a response at virtually all masses up to 300  $m/z$ . The background is very high at first but should drop gradually as the source bakes out. The high background is presumed to result from active hydrogen species cleaning the interior of the source, as is seen with JetClean. A detailed procedure for reducing the background is discussed in a later section.

## Reduced signal-to-noise ratio

The high background results in additional baseline noise that reduces the signal-to-noise ratio (SNR) of analytes. As the background drops, the noise drops, and the SNR gradually improves.

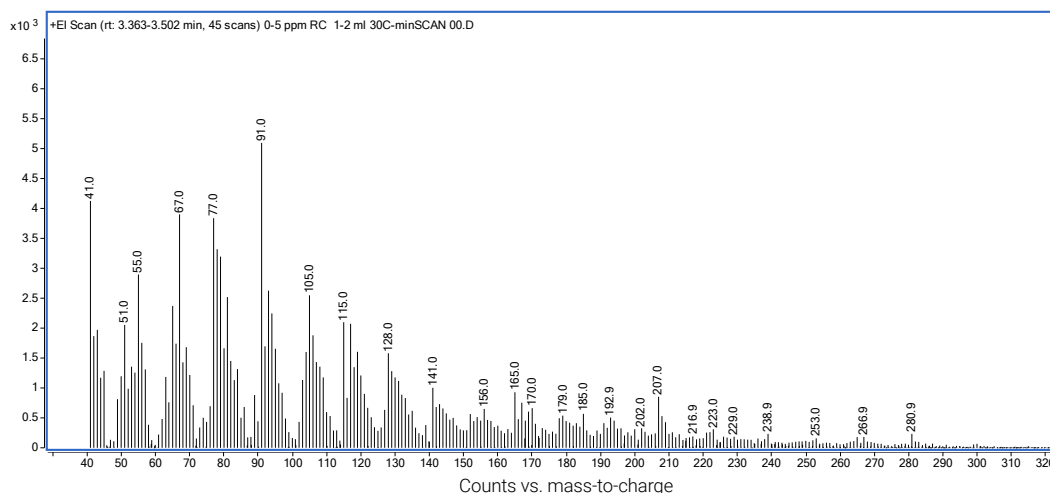


Figure 12. Mass spectrum of background with hydrogen carrier gas.

## Severe tailing of some compounds

The third problem, significant tailing for many compounds, is shown in **Figure 13**. The top chromatogram is the TIC of a mixture of underivatized drugs at 5 ng/compound acquired with helium carrier gas. Some of the compounds exhibit tailing due to their polar nature, but most have acceptable peak shapes.

The bottom chromatogram is the TIC of the same mixture shortly after the carrier gas was switched to hydrogen. The peak shape for phencyclidine (peak 9) is quite good, but the rest of the peaks have become significantly smaller and many exhibit severe tailing.

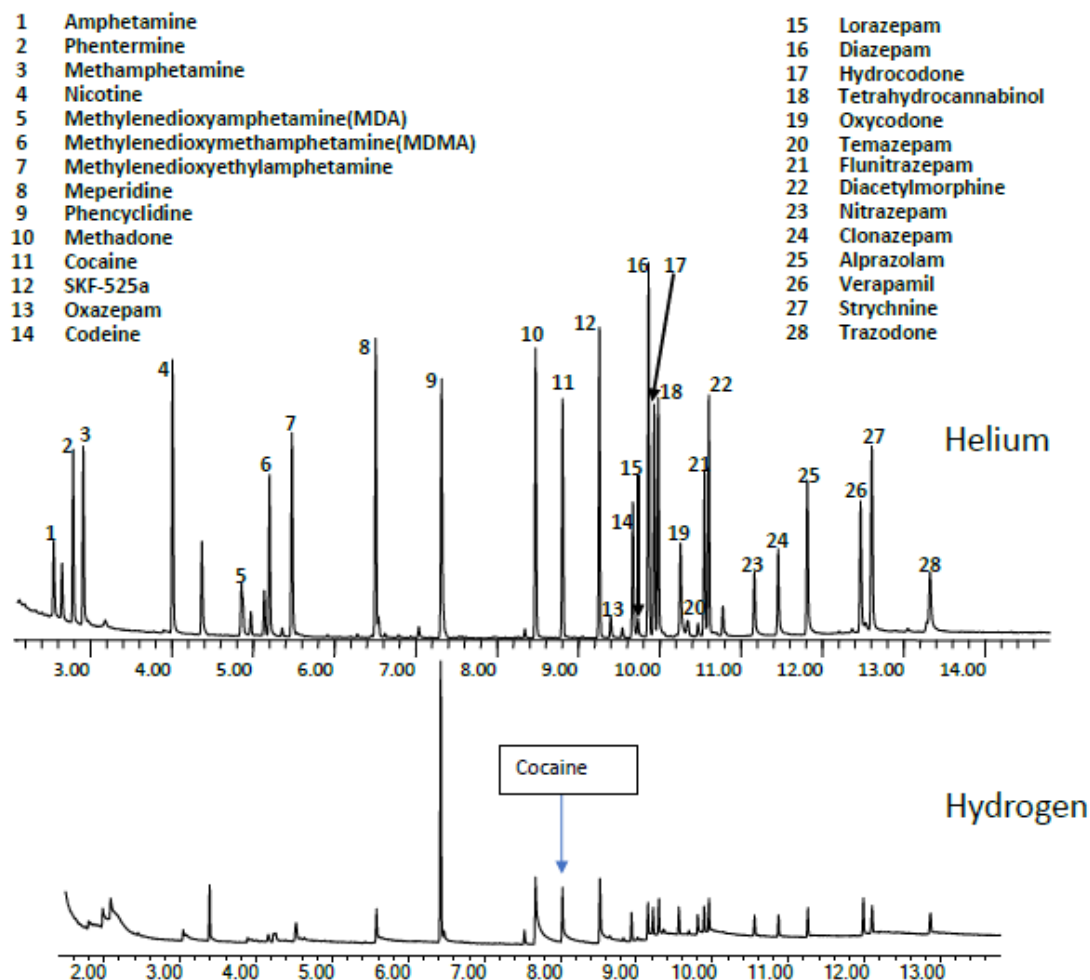


Figure 13. Drug test mix (5 ng/component) run with helium carrier gas (top) and hydrogen carrier gas shortly after initial startup (bottom).

Inspection of the cocaine peak in the TIC of the hydrogen chromatogram shows severe tailing not seen with helium carrier gas. This tailing is not typical chromatographic tailing.

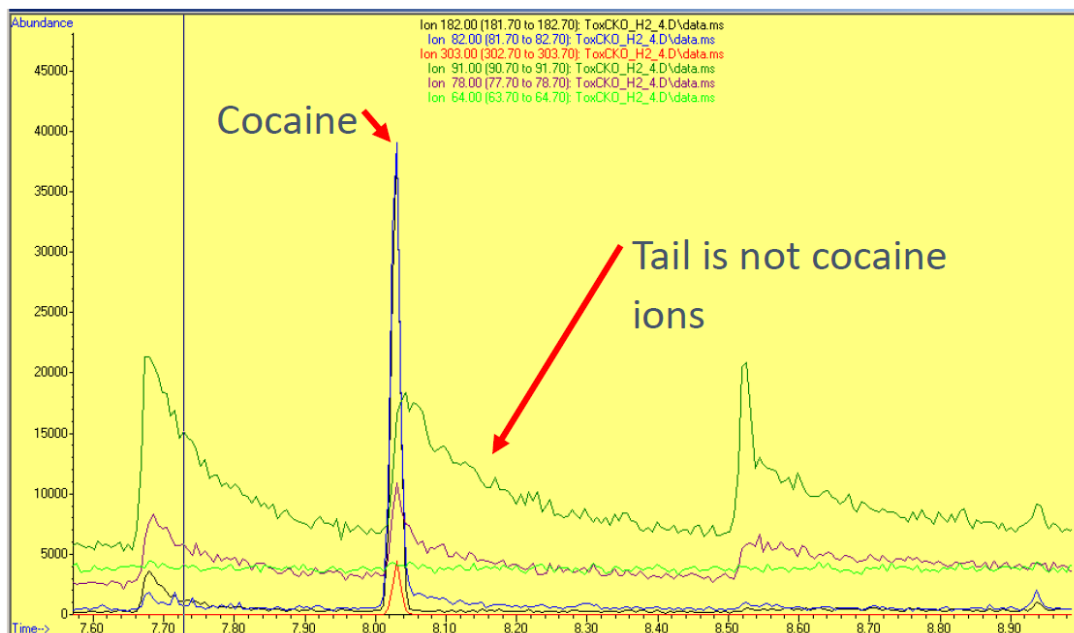


Figure 14. EICs from cocaine peak in **Figure 13** (bottom) with hydrogen carrier gas.

**Figure 14** shows the extracted ion chromatograms (EIC) for the principal cocaine ions at  $m/z$  182 (black), 82 (blue), and 303 (red). Note they have excellent peak shape. The tails are made up of ions unrelated to cocaine, with  $m/z$  91 (green) being the most prominent.

The hydrogen carrier chromatograms in **Figure 13** and **Figure 14** were acquired with the 6-mm extractor lens. This tailing effect is more pronounced with the 6-mm than with the 9-mm lens. The 6-mm was used here to more clearly demonstrate the problem, but the 9-mm is preferred.

Obviously, this performance is unusable. Fortunately, there is a procedure to greatly reduce these problems.

## Conditioning the source

The key to reducing the above three problems is to condition the source overnight. The recommended steps are:

- 1 Have a good source of clean hydrogen such as a >99.9999% purity hydrogen generator.
- 2 Keep the hydrogen flow limited to a value suitable for your pumping system.
- 3 Use an optional Hydrogen drawout lens/extractor lens. Start with the 9-mm first. If your maximum sensitivity is insufficient, try the 6-mm.
- 4 After setup, purging and pump down, go the MS acquisition program and:
  - a Set the source to the max temperature for your source (usually 350 °C, but check).
  - b Reduce the EMV to 800 V.
  - c Set the program to scan from  $m/z$  40 to 300 continuously. This will keep the filament on, which is key to the conditioning process. Leave the FILAMENT ON overnight.
- 5 Peak shape will be much better, and background will be much lower in the morning.
- 6 Lower the source temperature to method value, retune, and run some samples.
- 7 Always have extra filaments on hand in case one burns out. Like all GC/MS ion source parts, filaments are normal operator replaceable consumable parts.

The preceding procedure works well with the 6-mm drawout or extractor lenses. With the 9-mm lens, and a source that is clean, it may not require overnight cleaning. It may be conditioned in 2 to 4 hours as determined by a lower background and improved peak shapes.

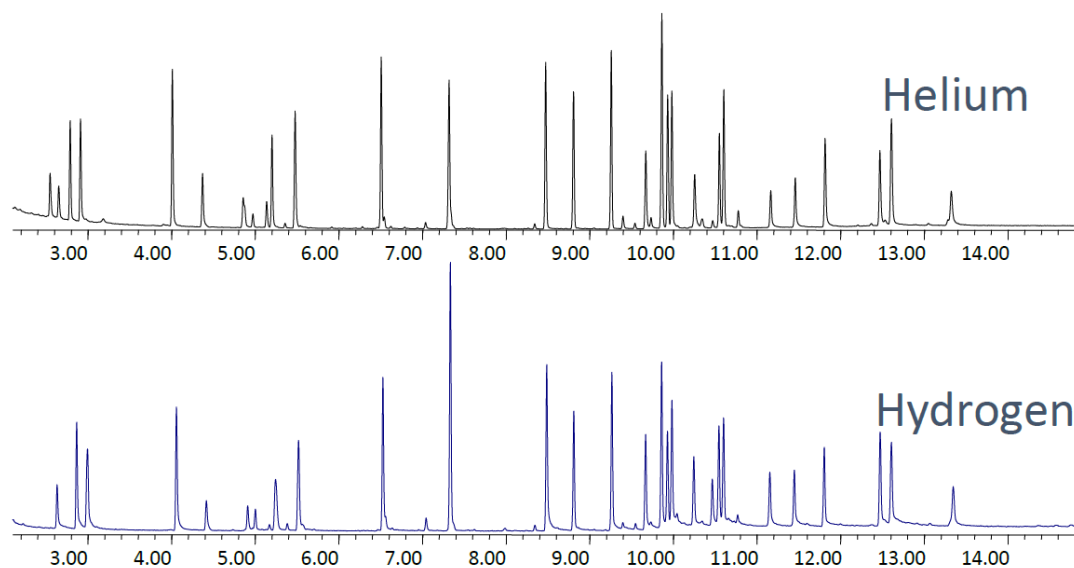


Figure 15. Drug test mix (5 ng/component) runs with helium carrier gas (top) and hydrogen carrier gas (bottom) after conditioning overnight.

**Figure 15** shows the dramatic improvement obtained with the overnight conditioning step, greatly reducing all three problems of high background, high noise, and severe tailing.

# Performance Expectations

## Signal-to-Noise ratio

Signal-to-noise ratio is often worse by about 2 to 5 times or more with hydrogen carrier gas. This varies from compound to compound, so it is important to check all analytes to be determined with the hydrogen method.

Some compounds may disappear at low levels. Examples include: some nitrogen and oxygen containing compounds (alcohols, aldehydes, ketones) such as those found in flavor samples.

## Spectral fidelity

While most spectra obtained with hydrogen carrier remain the same as those acquired with helium carrier gas, there are always exceptions. Users should check the reference spectra for important targets to make sure they have not changed.

As an example, **Figure 16** shows the chromatograms of a test mixture acquired with both helium and hydrogen carrier gas. The mixture contains a diverse set of compound types to evaluate changes in spectra.

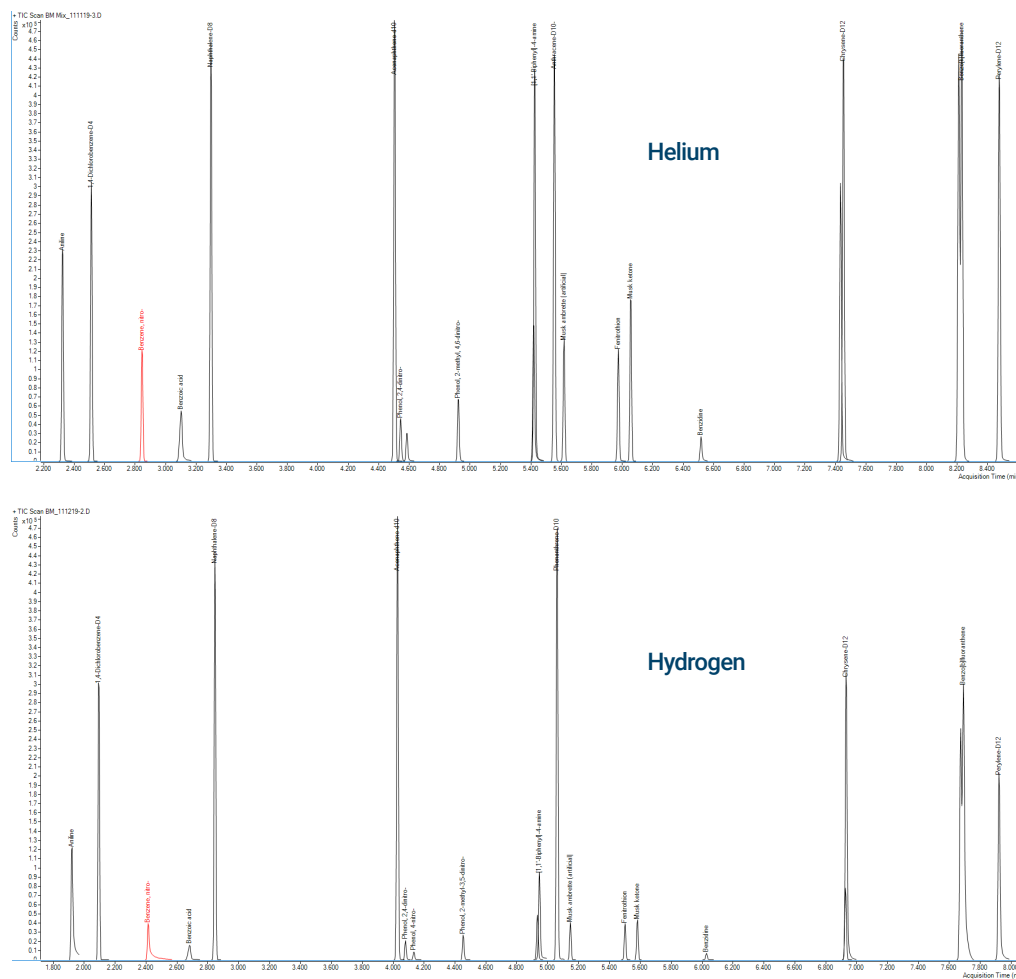


Figure 16. TIC chromatograms of test mixture to compare library match scores against NIST 17 for helium carrier gas (top) and hydrogen carrier gas (bottom). Compounds are listed in **Table 4**.



**Table 4** lists the library match scores (LMS) for test mix in **Figure 16**. Spectra were obtained by spectral deconvolution and searched against NIST 17 in MassHunter Unknowns Analysis. For most compounds, the LMS values are comparable between the two carrier gases.

As demonstrated in **Table 4**, while the 3 mm inert extractor works well with helium, 11 of the 21 compounds show decreased LMS values with hydrogen. The 9 mm inert extractor improves this substantially, with only 2 of the 21 compounds having significantly decreased LMS values with hydrogen. The 9 mm HydroInert provides the best LMS overall with hydrogen, especially for nitrobenzene and benzidine.

**Table 4** Library match scores (LMS) for test mix in Figure 16.

Analyte	3 mm Extractor with He (LMS)	3 mm Extractor with H <sub>2</sub> (LMS)	9 mm Extractor with H <sub>2</sub> (LMS)	9 mm HydroInert with H <sub>2</sub> (LMS)
Aniline	98.6	92.3	98.2	97.7
1,4-Dichlorobenzene-d4	96.7	94.7	98.9	97.5
Nitrobenzene	95.7	68.1	63.3	94.3
Benzoic acid	93.3	87.7	95.2	97.2
Naphthalene-d8	96.9	93.9	97.4	97.6
Acenaphthene-d10	98.3	97.3	98.5	97.8
2,4-Dinitrophenol	95.6	90.6	95.4	94.8
4-Nitrophenol	89.5	83.1	90.5	94.8
4,6-Dinitro-2-methylphenol	94.3	93.6	95.2	95.7
Pentachlorophenol	90.7	85.4	85.5	89.1
4-Aminobiphenyl	96.9	95.8	97.1	97.9
Phenanthrene-d10	97.7	93.3	96.7	97.2
Musk ambrette	86.6	86.3	89.0	89.5
Fenitrothion	95.6	88.3	94.3	97.0
Musk ketone	95.8	89.8	94.4	98.2
Benzidine	92.6	70.1	83.5	97.5
3,3'-Dichlorobenzidine	97.3	89.8	95.8	95.3
Chrysene-d12	96.0	84.8	95.2	93.5
Benzo[b]fluoranthene	97.8	70.1	97.7	98.4
Benzo[k]fluoranthene	97.8	96.8	97.7	98.4
Perylene-d12	94.4	79.4	98.2	93.1

## Other Considerations

Below is a list of items to consider when converting to hydrogen carrier gas:

- Hydrogen is not an inert gas, and therefore inertness problems will still exist or may be worse.
- Use the lowest inlet temperature that works (to reduce reactions with hydrogen).
- Use pulsed injection, especially with small bore columns.
- Consider using an MMI in cold splitless mode for fragile compounds.
- Using the optional deactivated split/splitless weldment might help reduce inlet reactions.
- Avoid using methylene chloride as a solvent (especially wet). At higher inlet temperatures (such as, >280 °C), HCl is formed and causes problems.
  - If it must be used, use the lowest inlet temperature, the optional deactivated SSL weldment, or an MMI inlet with the temperature ramped starting at 200 °C.
- Avoid carbon disulfide as a solvent.
- Use liners with a taper at the bottom to minimize sample contact with the gold seal.
- Install the column high enough in the inlet to minimize sample contact with the gold seal.
- Use Agilent Ultra Inert inlet liners.
- Have extra filaments on hand, as their useful life is usually shorter with hydrogen carrier gas.
- Source cleaning may be needed much less frequently due to the cleaning action of hydrogen.

## Summary

For many GC/MS applications, hydrogen can be successfully substituted for helium as the carrier gas. Changes are often required in gas plumbing, instrument hardware, consumables, sample preparation, and method parameters. As discussed in this document, there are several topics that need to be addressed to achieve successful conversion.



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