Conversion of GC/MS Methods From Helium To Hydrogen Carrier Gas

poster 1210-10P

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Introduction

Concerns regarding the cost and availability of helium have resulted in many GC/MS users considering conversion to hydrogen as the carrier gas. This poster will discuss the steps required in this conversion and some of the limitations to be expected with using hydrogen as a carrier gas.

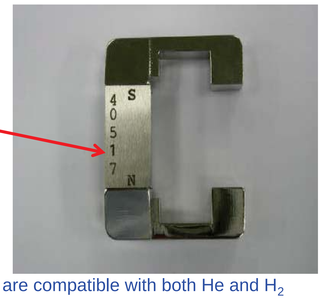
Safety and Gas Source

- **Hydrogen Safety:**
  - –Check with your laboratory safety coordinator to make sure you are allowed to use hydrogen in your lab.
  - –Make sure your GC/MS system was designed for use with hydrogen. If there is any question, check with the manufacturer.
  - –Review any safety documentation provided with the instrument.
- **Source of H_2 Carrier Gas:**
  - –If possible, use a high purity (>99.9999%) hydrogen generator.
  - –Higher initial expense than cylinders, lower cost over time.
  - –Very clean H_2, >99.9999% available
  - –More consistent purity
  - –Make sure to buy a good one with a low spec for water and oxygen
  - –Safety considerations
    - H_2 is only generated at needed pressure (like 40 psig)
    - Flow is limited (like 250 mL/min)
    - Auto-shutdown if set point pressure cannot be maintained
    - Minimal stored gas (like 50 mL at 40 psig) of H_2 at any one time

Source Magnet

If the magnet in the 5975 does NOT have a serial number along the left edge (as viewed through the source window), it is suitable for use with Helium, but not Hydrogen.

If not, please contact your Agilent service engineer to change it to a Helium and Hydrogen compatible magnet before converting the system to H_2 carrier.



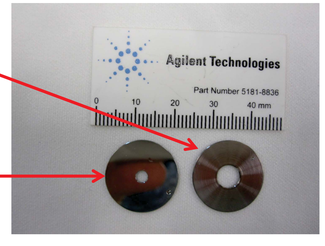
Note: All 5973 magnets are compatible with both He and H_2

Source Drawout Plate

As shown later, results with hydrogen carrier gas are improved by using a larger diameter source drawout plate.

G2589-20045
Hydrogen Draw Out Plate, Inert

Standard 3 mm
Draw Out Plate, Inert



H_2 Flow Rate Into MS

These are approximate values for the 5975. Maximum flow of H_2 to maintain reasonable source pressure:

- –Performance turbo: 2 mL/min
- –Standard turbo: 1 mL/min
- –Diffusion pump: 0.75 mL/min
- –Pressure pulsing: turbo ≤ 3 mL/min, diffusion ≤ 2.5 mL/min

It is very helpful to have an ion gauge on the MS to monitor the vacuum vs. column flow.

Try to avoid flows that produce pressures higher than 5 x 10^{-5} torr. You can get useful data above this pressure, but performance may start to degrade

Plumbing Considerations

- –Chromatographic quality stainless steel tubing is often recommended for H_2 plumbing and is probably the best choice if available. Users may have to follow local codes or internal company guidelines.
- –We have also used new 1/8th copper that has been cleaned for GC use.
- –Dirty tubing will cause huge contamination problems, as H_2 appears to carry dirt out of metal more than He does.
- –Don't use really old copper tubing, as it becomes brittle and can break.
- –Note that MSD leak checks will not always find big outgoing leaks. Leak check when complete with electronic leak detector.
- –When plumbing a H_2 generator, start out with no traps. Only add traps if needed. Make sure the water and oxygen levels are low enough.

Choosing A Column

Determine max flow of H_2 into MS that will give source pressure of 5 x 10^{-5} torr or less source pressure. This is your max column flow.

Choose column dimensions at initial oven temp of method to give:

- –a flow ≤ max column flow for vacuum pump
- –a flow that give at least 35 cm/sec average linear velocity
- –an inlet pressure of at least 5 psig

Keeping a temperature ramp of the same number of C/void time will give similar elution order. Use the Agilent method translator for this.

These are only approximate guidelines. Sometimes you may have to deviate from them.

Method Translation Tool

Use the method translation tool to evaluate potential columns and conditions for the H_2 version.

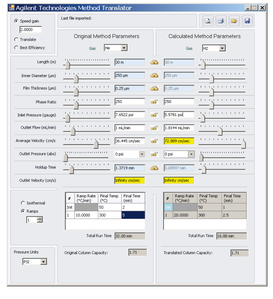
In this example, the same column is used, but has to be run 2 X faster to get a flow that gives a high enough inlet pressure.

In many cases, using a 20m X 180um X 0.18um film thickness is a good fit.

The benefit of the tool is that it gives predicatable retention times and elution order for your analytes with the new H_2 method. This saves a lot of time.

The tool is available as a free download at:

http://www.agilent.com/chem/heliumupdate



Initial Startup Problems With H_2

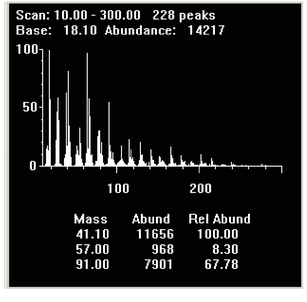
High MS background that looks like hydrocarbons ☹

Reduced signal to noise (worse MDL) from the high background☹

Significant tailing for many compounds ☹

These problems improve with time, but without the techniques presented here, could take several days to weeks to clear up ☹

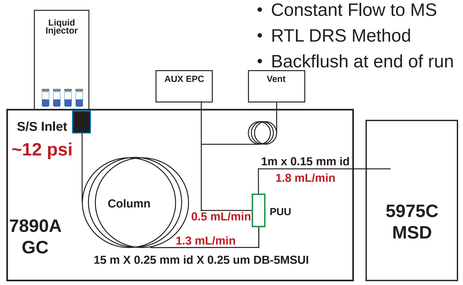
The new protocol described here greatly reduces the cleanup time ☺



Example Conversion

The example here is a forensic toxicology screening system. The setup uses post-column backflushing to reduce system maintenance. Retention time locking and mass spectral deconvolution (DRS) are used to greatly improve screening accuracy and productivity. Method translation was used for the conversion to preserve the same retention times as with helium.

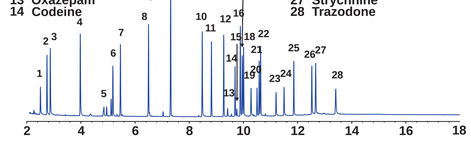
He Carrier Gas Forensic Toxicology Analyzer



- Constant Flow to MS
- RTL DRS Method
- Backflush at end of run

Forensic Tox Checkout Mix 5 ng/uL each compound

1 Amphetamine
2 Phentermine
3 Methamphetamine
4 Nicotine
5 Methylenedioxyamphetamine(MDA)
6 Methylenedioxymethamphetamine(MDMA)
7 Methylenedioxyethylamphetamine
8 Meperidine
9 Phencyclidine
10 Methadone
11 Cocaine
12 SKF-525a (RTL Compound)
13 Oxazepam
14 Codeine
15 Lorazepam
16 Diazepam
17 Hydrocodone
18 Tetrahydrocannabinol
19 Oxycodone
20 Temazepam
21 Flunitrazepam
22 Diacetylmorphine
23 Nitrazepam
24 Clonazepam
25 Alprazolam
26 Verapamil
27 Strychnine
28 Trazodone

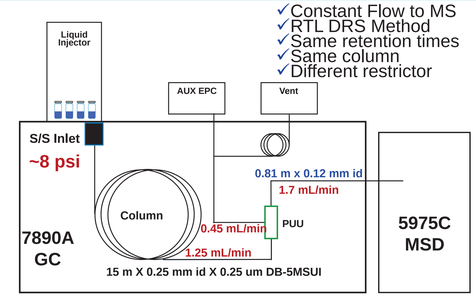


Improved H2 Carrier Protocol

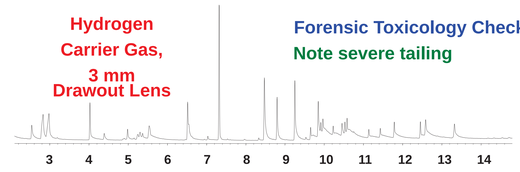
1. Have a good source of clean hydrogen like a >99.9999% purity hydrogen generator.
2. Keep H_2 flow limited to value suitable for your pumping system
3. Use optional Hydrogen draw out lens
4. After setup, purging and pump down:
   - –Set the source to max temp for your source (check to see what yours is)
   - –Reduce the EMV to 800V
   - –Switch to filament 2
   - –Leave the **FILAMENT ON** overnight. This cleans up background rapidly.
5. Peak shape will be much better and background will be much lower in the morning.
6. Lower the source temp to method value, switch back to filament 1, retune, and run some samples
7. It is a good idea to have an extra set of filaments on hand in case one burns out. This hasn't been a problem, but a good idea.

H_2 Carrier Gas Forensic Toxicology Analyzer

- ✓Constant Flow to MS
- ✓RTL DRS Method
- ✓Same retention times
- ✓Same column
- ✓Different restrictor

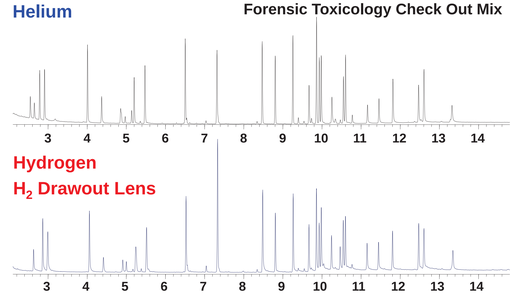


Initial Result Before New H_2 Protocol



Result Using New H_2 Protocol

- Much Faster Stabilization
- Peak Shape Much Better
- Background is Lower
- Better Signal-to-Noise



Summary And Conclusions

- •**Signal-to-noise ratio is often worse by about 2-5 x. This obviously varies from compound to compound**
- •**While most spectra remain the same, there are always exceptions. Users should check the reference spectra for important targets to make sure they have not changed**
- •**Same comment for target/qualifier ratios**
- •**Some compounds may disappear at low levels.**
- •**Good news: Source cleaning will be needed much less frequently**

- **H_2 is not an inert gas, so check for inertness problems**
- **Use the lowest inlet temp that works (to reduce reactions with H_2)**
- **Use pulsed injection, especially with small bore columns**
- **Consider using an MMI (PTV) in cold splitless mode for fragile compounds**
- **Using a deactivated S/SL weldments may help**
- **Avoid using methylene chloride as a solvent (especially wet).**
  - –**At higher inlet temps (like >280C), HCl is formed.**
  - –**If must be used, use lowest inlet temp and maybe a deactivated S/SL weldment, or Multimode inlet**
- **Use liners with a taper at bottom (like the Agilent Ultra Inert Liner)**