How to Combat the Helium Shortage: Making the Switch from Helium to Hydrogen or Nitrogen Carrier Gas

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Application Engineer
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Market Situation

The world of He supply is not reliable, prices are increasing and customers are seeking alternative carrier gases

Researchers have a need to find suitable alternatives to either eliminate or reduce He consumption
Industries That Use Helium

Healthcare
Magnetic Resonance Imaging (MRI)

Research
Nuclear Magnetic Resonance spectroscopy (NMR)
Gas chromatography

Helium
Boiling point
\(-269 \, ^\circ C/4.2 \, K\)
Webinar Outline

Use of Gas Saver

Carrier gas decision tree
– Decision making guide to fit your carrier gas requirements

Helium conservation
– Smarter helium use with new hardware/software tools
– No need to revalidate existing GC methods

Migrating existing helium GC methods to $H_2$ or $N_2$
– Best practices to obtain the same results and minimize method revalidation

Cautions about making the switch to Hydrogen for MSD systems
Have You Ever Noticed the Gas Saver Section of the Inlet Method Editor?
Why Would I Want to Use Gas Saver?

- When enabled, GC automatically runs Gas Saver
  - Beneficial when using split mode

- Gas Saver mode turns **on** after injection
  - Turns **off** during the prep run and injection duration

Lowers carrier gas use to save helium (or other carrier gas) and cut costs

- **Suggested parameters**
  - Flow – No lower than 15 mL/min
    - Recommended: ~20 mL/min
  - Time – ~2 to 5 minutes
Carrier Gas Decision Tree
Continue using helium, but in a smarter way

- Is the chemist willing to convert to alternative gases?
  - No: He conservation
  - Yes: Consider migration to H₂

- Is the application based on GC or GC/MS?
  - GC: Does the current GC method have excess resolution?
    - No: Consider migration to N₂
    - Yes: GC/MS specific H₂ considerations
  - GC/MS: Method revalidation not required
Reducing Helium Use With Conservation

Programmable helium conservation module (available for Agilent 7890B, 8860, 8890 GC systems)

- Automatically switches carrier gas supply to $N_2$ standby during idle time
- Integrates into the Sleep and Wake function of the GC
- Combined with Helium Gas Saver to greatly reduce helium consumption
- Better alternative to just “shutting off the GC”
  - No system contamination with ambient air exposure
  - Faster restart of heated zones
Helium Conservation Module
Seamlessly integrated onto GC hardware and software

- Built on 5th generation EPC
- Fully controlled by Agilent data systems
- Purge channel prevents cross contamination of gases
- Precise pressure control between tank and GC
- Switch between gases within 15 to 30 min for most detectors
How Does It Work?

Normal operation mode (helium carrier or wake mode)

- **AUX EPC 1**
  - nitrogen
  - 0 psig

- **AUX EPC 3**
  - purge
  - 10 psig

- **AUX EPC 2**
  - helium
  - 80 psig

1 mL/min (out)

Bridge block

24.2 mL/min He

25.2 mL/min He

To GC inlet

EPC

Helium on at 80 psig, nitrogen off

GC/FID wake method: 15 to 30 min
GC/MS wake method: 15 to 30 min
Some other detectors may need longer

(< 0.2 mL/min) N₂
How Does It Work?
Helium savings mode (nitrogen carrier, or sleep mode)

Helium off, nitrogen on at 70 psig
How It Works: Configuring Sleep/Wake Operation

Simple, straight forward setup

![Configuration Interface](image-url)

### Instrument Schedule

Select a schedule that best matches how you use this instrument:

- **Custom**

<table>
<thead>
<tr>
<th>Day</th>
<th>Set Wake Method</th>
<th>Wake Time</th>
<th>Set Sleep Method</th>
<th>Sleep Time</th>
</tr>
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<tbody>
<tr>
<td>Sunday</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Monday</td>
<td></td>
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<td>Wednesday</td>
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<td>Thursday</td>
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<td>Friday</td>
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<td></td>
</tr>
<tr>
<td>Saturday</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Wake Method:**
- Edit Wake Method

**Sleep Method:**
- Edit Sleep Method

- Wake to last active method before sleep
- Perform a conditioning run on Wake

[Image of Configuration Interface]
Performance: GC/FID Analysis

Day 1 - Original He carrier gas run

Day 2 – First He carrier gas run after overnight N₂ Sleep.M method

Day 3 – First He carrier gas run after overnight N₂ Sleep.M method

No change in chromatography after N₂ carrier Sleep method.
Performance: MS Tune

Passes Within 15min After Switching From N₂ to He As Carrier. GC/MSD

<table>
<thead>
<tr>
<th>Time (min)</th>
<th>5 mL/min He</th>
<th>Relative to Saturation</th>
<th>2 mL/min He</th>
<th>Relative to Saturation</th>
</tr>
</thead>
<tbody>
<tr>
<td>3</td>
<td>1735168</td>
<td>20.69%</td>
<td>8388096</td>
<td>100.00%</td>
</tr>
<tr>
<td>4</td>
<td>1033280</td>
<td>12.32%</td>
<td>4959232</td>
<td>59.12%</td>
</tr>
<tr>
<td>5</td>
<td>590080</td>
<td>7.03%</td>
<td>1618944</td>
<td>19.30%</td>
</tr>
<tr>
<td>6</td>
<td>354112</td>
<td>4.22%</td>
<td>722944</td>
<td>8.62%</td>
</tr>
<tr>
<td>7</td>
<td>228480</td>
<td>2.72%</td>
<td>333696</td>
<td>3.98%</td>
</tr>
<tr>
<td>10</td>
<td>56984</td>
<td>0.68%</td>
<td>102576</td>
<td>1.22%</td>
</tr>
<tr>
<td>15</td>
<td>9052</td>
<td>0.11%</td>
<td>17080</td>
<td>0.20%</td>
</tr>
</tbody>
</table>
Helium Savings—Single GC Channel

Extend helium supply and lower cost using conservation techniques

Example

• ASTM Method D4815
  – Widely used to measure ethanol in gasoline
  – Helium cylinder last two months under normal operation

• Helium conservation
  – Helium cylinder life extended to 12 months
  – 4x yearly gas costs per year

<table>
<thead>
<tr>
<th>Parameter</th>
<th>No Conservation</th>
<th>Helium Conservation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Daily He Usage (L)</td>
<td>112</td>
<td>21</td>
</tr>
<tr>
<td>He Cylinder Life (days)</td>
<td>71</td>
<td>376</td>
</tr>
<tr>
<td>Daily N₂ Usage (L)</td>
<td>0</td>
<td>24</td>
</tr>
<tr>
<td>N₂ Cylinder Life (days)</td>
<td>0</td>
<td>340</td>
</tr>
<tr>
<td>Yearly He Cost ($)</td>
<td>$1,537</td>
<td>$292</td>
</tr>
<tr>
<td>Yearly N₂ Cost ($)</td>
<td>$0</td>
<td>$64</td>
</tr>
</tbody>
</table>

Yearly Total Gas Cost

$1,537

$356
Alternative for Older Systems (or Non-Agilent Systems)

- Use of a 3-way stream selection valve to manually switch between $N_2$/He
  - Plumb $N_2$ to one input and He to the other and switch valve as needed
  - Not automated/integrated
Carrier Gas Decision Tree
Migrating GC methods to nitrogen and hydrogen

Is the customer willing to convert to alternative gasses?

Yes

Is the application based on GC or GC/MS?

GC

No

Is the current GC method have more than enough resolution?

No

He Conservation

Yes

Consider migration to N₂

Consider migration to H₂

GC/MS

GC/MS specific H₂ considerations
Safety Considerations for Hydrogen Migration

GC and GC/MS: Both offer H₂ enabled features

Read Hydrogen safety guide!!

Safety Shutdown
When gas pressure set points are not met, the valve and heater are shut off to prevent explosion

Flow Limiting Frit
If valve fails in open position, inlet frit limits the flow

Oven ON/OFF Sequence
Fan purges the oven before turning on heater to remove any collected H₂

Explosion “ready”
GC and MS designed to contain parts in case of explosion
  i.e. Spring in GC door

H₂ sensor available
Considerations for Hydrogen Gas Sources

**H₂ generator – preferred**
- Very clean H₂, >99.9999% available
- Consistent purity
- Built-in safety features
- Make sure to buy a good generator with a low spec for water and oxygen
- Parker’s H-MD are used at Agilent sites

**H₂ cylinder**
- Consider Gas Clean filter
- Possible to add safety device
Considerations for Hydrogen Gas Plumbing

**Tubing**
- Use chromatographic quality stainless steel tubing (recommended)
- Do not use old tubing (H₂ is known as scrubbing agent)
- Especially don’t use old copper tubing (brittleness is a safety concern)

**Venting**
- Connect split vent and septum purge vent to exhaust

**Leak checking**
- Recommend G3388B leak detector
Use N₂ As Carrier Gas

Many HPI methods suited to nitrogen
- Readily available and less expensive gas
- No safety concerns
- Suitable for simple routine analysis (with sufficient resolution)
- More inert than H₂, especially with PLOT/micropacked columns
  - Some compounds catalytically reduced in H₂
- 2-D GC ideally suited to nitrogen
  - Resolution issues solved using two different columns

Potential issues
- Reduced chromatographic resolution at higher flows
- Not suitable for GC/MSD and certain GC detector applications
Helium Carrier Gas Alternatives

Important theoretical considerations relating to peak efficiency

Sharp, narrow peaks in a chromatogram is an indication of a high efficiency GC column.

- Remember that efficiency is represented mathematically by the symbol “N” called Theoretical Plates, and that the larger N is, the better the resolving power of the column (i.e., higher resolution).
- Resolution is described mathematically by the symbol \( R_s \) and its numeric value tells how well two adjacent peaks are separated from each other.

\[
R_s = \left( \frac{N}{4} \right) \left( \frac{k+1}{\alpha+1} \right) \left( \frac{\alpha-1}{\alpha} \right)
\]

A resolution value of 1.5 tells us that two peaks are baseline separated. The greater (higher) the \( R_s \) value, the more separation that has been achieved.

Calculating efficiency

We would like to know the actual time the component spends in the stationary phase.

\[
t_R' = t_R - t_m
\]

\[
t_R = \text{corrected retention time.}
\]

\[
n = \frac{5.545 \left( \frac{k}{N} \right)^2}{L}
\]

\[
n = \text{effective theoretical plates.}
\]

Thus, the more efficient the column, the bigger the "N" the smaller the "HETP".

Efficiency and carrier gas linear velocity

Efficiency is a function of the carrier gas linear velocity or flow rate.

The minimum of the curve represents the smallest HETP (or largest plates per meter) and thus the best efficiency. "A" term is not present for capillary columns.

- Plot of HETP versus linear velocity is know as the Van Deemter plot.
- The linear velocity value at the minimum of the curve is the optimum value for achieving the best efficiency.
Helium Carrier Gas Alternatives
Let’s make this easy

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- Resolution is described mathematically by the symbol $R_s$ and its numeric value tells how well two adjacent peaks are separated from each other.

$$R_s = \sqrt{\frac{N}{k+1}} \left[ \frac{\sigma + \sigma}{\sigma} \right]$$

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Calculating efficiency

We would like to know the actual time the component spends in the stationary phase.

$$n = 5.545 \left( \frac{t_R - t_m}{t_m} \right)$$

$n$ = corrected retention time.

$n$ = effective theoretical plates.

Thus, the more efficient the column, the bigger the "N" the smaller the "HETP".

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Van Deemter

Why nitrogen gets a bad reputation for capillary GC

- $\text{N}_2$ actually provides the best efficiency, but at a slower speed
- Most helium methods have too much resolution
  - Lower $\text{N}_2$ efficiency at higher flows can still provide “good enough” resolution
- Most GC methods now use constant flow
  - $\text{N}_2$ efficiency losses with temperature programming are not as severe
Helium Carrier Gas Alternatives
Let’s make this easy

• Goal: Change carrier gas while keeping other method conditions the same
  – Use the same column
  – Use the same oven program
  – Adjust column flow or holdup time to:
    • Maintain same peak elution order
    • Maintain same peak retention times (or as close as possible)

• Easier method revalidation using this approach
  – Minimal changes to timed integration events
  – Minimal changes to peak identification table

• For N₂, test resolution of key components
  – Adjust GC conditions (temperature, flow) if needed
Many Helium GC Have Excess Resolution
EN14103 – GC analysis of FAME content in biodiesel

HP-INNOWax, 30 m x 0.25 mm id x 0.25 µm

Helium at 1 mL/min constant flow (25.4 cm/s)

Nitrogen at 1 mL/min constant flow (25.8 cm/s)
Configure Inlet for Carrier Gas in ChemStation

Select H₂ or N₂
How Do I Build a New Method for Use with H$_2$ or N$_2$ Carrier?
New Windows 7 Method Translation Calculator
Another useful tool for carrier gas calculations

• Flexible tool helps convert existing helium methods to alternative carrier
• Built into the new OpenLab CDS software
• Can also run as Windows 7 program
• Download from the Agilent Helium Update page:
  www.agilent.com/chem/heliumupdate
Same Column, Hydrogen Carrier Gas

- Speed gain
- New pressure/flow/velocity
- New temperature program
Method Translation Software

Switch from He to H₂ or N₂ carrier gas
Same column and gas type but faster velocities
Different column dimensions
Combination of all of the above

Link to software download:
Helium Carrier Gas Alternative
Test Case: ASTM D6584 for free and total glycerin in biodiesel

COC inlet: Oven track mode
Precolumn: Ultimetal 2 m x 0.53 mm id
Column: Ultimetal DB5HT, 15 m x 0.32 mm id x 0.1 df
Column flow: Helium at 3.0 mL/min (50 °C)
Column pressure: 7.63 psi constant pressure mode
Initial column temp: 50 °C for 1 min
Oven ramp 1: 15 °C/min to 180 °C
Oven ramp 2: 7 °C/min to 230 °C
Oven ramp 3: 30 °C/min to 380 °C, hold 10 min
Detector: FID with 25 mL/min N₂ makeup

1. Glycerol
2. Monoglycerides
3. Diglycerides
4. Triglycerides
Wider Retention Time Variation Using the Same Flow as the Original Helium Method

Helium
Flow: 3.00 mL/min
P: 7.63 psi
T_r: 0.472 min
m: 52.97 cm/s

23.818 min

Hydrogen
Flow: 3.00 mL/min
P: 3.85 psi
T_r: 0.420 min
m: 59.50 cm/s

23.469 min

Nitrogen
Flow: 3.00 mL/min
P: 7.09 psi
T_r: 0.464 min
m: 53.84 cm/s

23.705 min
Set the Control Mode: Flow or Holdup Time

Try the same flow or holdup time of the original helium method
Same Holdup Time ($T_r$) Gives Consistent Retention Times Compared to Original Helium Method

- **Helium**
  - Flow: 3.00 mL/min
  - $P$: 7.63 psi
  - $T_r$: 0.472 min
  - $m$: 52.97 cm/s
  - Retention Time: 23.818 min

- **Hydrogen**
  - Flow: 2.64 mL/min
  - $P$: 3.43 psi
  - $T_r$: 0.472 min
  - $m$: 52.97 cm/s
  - Retention Time: 23.862 min

- **Nitrogen**
  - Flow: 2.94 mL/min
  - $P$: 6.98 psi
  - $T_r$: 0.472 min
  - $m$: 52.97 cm/s
  - Retention Time: 23.776 min
Monoglyceride Resolution “Good Enough” Using Nitrogen Carrier

All monoglycerides are summed for final reporting

High resolution of isomers is therefore not required

Helium
Flow: 3.00 mL/min
$T_r$: 0.472 min

Hydrogen
Flow: 2.64 mL/min
$T_r$: 0.472 min

Nitrogen
Flow: 2.94 mL/min
$T_r$: 0.472 min
ASTM D6584 - Quantitative Results for Alternative Carrier Gas

Carrier gas has no effect on reported results

<table>
<thead>
<tr>
<th></th>
<th>Weight Percent</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Helium</td>
</tr>
<tr>
<td>Glycerin</td>
<td>0.015</td>
</tr>
<tr>
<td>Monoglycerides</td>
<td>0.226</td>
</tr>
<tr>
<td>Total glycerin</td>
<td>0.097</td>
</tr>
</tbody>
</table>
Analysis of Oxygenates and Aromatics in Gasoline Using 2-D Gas Chromatography

ASTM Method D4815 – Oxygenated additives
- Ethers and alcohols from 0.1 wt% to 15 wt%
- Usually only one or two additives in a sample

Preliminary separation removes light hydrocarbons from sample
- Polar TCEP micropacked columns retain ethers and alcohols
- Back flush TCEP* column to nonpolar capillary column (HP-1) to complete analysis

* TCEP = 1,2,3-tris(2-cyanoethoxy)propane
Configuration and Operation for D4815 and D5580

- SST
- EPC
- PCM
- EPC
- TCEP column
- HP-1 capillary column
- Split/splitless inlet
- Primary flow
- Secondary flow
- Variable restrictor
- Split/splitless inlet
- FID
- TCD
- Primary flow
- Secondary flow
## Instrument Conditions

Use nitrogen carrier gas with original ASTM GC flow conditions

<table>
<thead>
<tr>
<th>Method D4815</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Carrier gas</td>
<td>Nitrogen</td>
</tr>
<tr>
<td>Inlet</td>
<td>Split/splitless</td>
</tr>
<tr>
<td>Inlet Temperature</td>
<td>200 °C</td>
</tr>
<tr>
<td>Inlet pressure</td>
<td>9 PSI (constant P)</td>
</tr>
<tr>
<td>TCEP column flow</td>
<td>5 mL/min</td>
</tr>
<tr>
<td>Split ratio</td>
<td>15 : 1</td>
</tr>
<tr>
<td>Split flow</td>
<td>70 mL/min</td>
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<tr>
<td>PCM pressure program</td>
<td>13 PSI for 14 min 99 PSI/min to 40 PSI</td>
</tr>
<tr>
<td>HP-1 column flow</td>
<td>3 mL/min</td>
</tr>
<tr>
<td>FID Temperature</td>
<td>250 °C</td>
</tr>
<tr>
<td>Oven Temperature</td>
<td>80 °C Isothermal</td>
</tr>
<tr>
<td>Run time</td>
<td>16 minutes</td>
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</table>
Analysis of MtBE and Ethanol in Gasoline Using $N_2$ Carrier Gas
## ASTM Precision Specifications

### D4815 precision measures

<table>
<thead>
<tr>
<th>Compound</th>
<th>Mass %</th>
<th>Repeatability Spec</th>
<th>Observed</th>
<th>Reproducibility Spec</th>
<th>Observed</th>
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</thead>
<tbody>
<tr>
<td>Ethanol</td>
<td>0.99</td>
<td>0.06</td>
<td>0.01</td>
<td>0.23</td>
<td>0.01</td>
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<tr>
<td>Ethanol</td>
<td>6.63</td>
<td>0.19</td>
<td>0.03</td>
<td>0.68</td>
<td>0.04</td>
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<tr>
<td>MtBE</td>
<td>2.10</td>
<td>0.08</td>
<td>0.01</td>
<td>0.20</td>
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<tr>
<td>MtBE</td>
<td>11.29</td>
<td>0.19</td>
<td>0.05</td>
<td>0.61</td>
<td>0.08</td>
</tr>
</tbody>
</table>

### Accuracy evaluation

<table>
<thead>
<tr>
<th>Sample</th>
<th>MtBE mass %</th>
</tr>
</thead>
<tbody>
<tr>
<td>known</td>
<td>found</td>
</tr>
<tr>
<td>SRM2294 #1</td>
<td>10.97</td>
</tr>
<tr>
<td>SRM2294 #2</td>
<td>10.97</td>
</tr>
<tr>
<td>AccuStd Check</td>
<td>12.00</td>
</tr>
</tbody>
</table>
Carrier Gas Decision Tree
Migrating GC methods to nitrogen and hydrogen

Is the customer willing to convert to alternative gasses?

Is the application based on GC or GC/MS?

Does the current GC method have more than enough resolution?

He Conservation
Consider migration to N₂
Consider migration to H₂

GC/MS specific H₂ considerations
MSD systems: Do not Switch from He to $H_2$ unless absolutely necessary

- Hydrogen is a very reactive gas!
- You **WILL** experience a noisy/elevated background that can be persistent (days/weeks)
- Chemical reactions happen in the inlet, column, and sometimes the source that can change your results.
- EVERY analyte in EVERY matrix in EVERY method will need to be validated using hydrogen to make sure there are no chemical reaction problems.
- Looking for untargeted unknowns is problematic due to the possibility of reactivity.
- Library search match quality will be impacted.
- Tuning results will be different than with helium. Some tunes, notably BFB and DFTPP, may not pass.
- First, try all helium conservation measures instead of switching to H2.

*There are no published performance specifications for any current Agilent GCMS system using hydrogen carrier gas.*
MSD: Converting from He to \( \text{H}_2 \) Carrier Gas

- Many GC/MS users are considering changing from helium to hydrogen carrier gas due to price/availability problems with helium.
- Read Chemical and Engineering News - July 16, 2012 (Page 32-34)
- It is important to recognize the differences with using hydrogen carrier. Time should be allotted for adapting the method, optimization, and resolving potential problems. Areas that will need attention include:
  - choice of supply of \( \text{H}_2 \)
  - GC/MSD hardware changes
  - choosing new chromatographic conditions
  - potential reduction in signal-to-noise ratio (2-5x or more) due to higher noise
  - changes in spectra and abundance ratios for some compounds
  - activity and reactivity with some analytes

*There are no published performance specifications for any current Agilent GCMS system using hydrogen carrier gas.*
GC/MS Migration to H₂ Carrier Gas
Recent C&EN webinar discussion points

• Read Hydrogen safety guide before proceeding!

• System setup
  – H₂ safety, H₂ source, gas connection, system clean up

• Method migration
  – Method transfer SW, method migration consideration, revalidation

• GC/MS analytical performance expectation
  – Sensitivity impact, MS spectrum impact, analyte compatibility

• For more details
  – C&EN webinar on October 9, 2012
  – Recorder session: http://cen.acs.org/media/webinar/agilent_100912.html
Summary: Helium Conservation Benefits

• Seamless integration
  No need to revalidate existing GC methods
  Fully integrated with Agilent 7890B and CDS (OpenLab, Mustang, MassHunter)
  Carrier gas ID and setpoints are a part of the method for compliance and transfer
  Easily implemented using new Agilent Sleep/Wake functions

• Greater reliability
  Based on proven 5th generation AUX EPC
  Agilent 7890/8890 provides warning if setpoints are not reached
  For hydrogen users, nitrogen substitution when not running GC

• Greater performance
  Purge channel prevents cross contamination of gases
  Delivers more stable gas pressure control from the tank regulator to the inlet
  EPC module
  Acts as an intermediate pressure regulator from the tank to inlet EPC to ensure
  greater analytical precision
Summary – Migration to H₂ and N₂

• Don’t forget about Gas Saver
• Be especially cautious when migrating to H₂ with an MSD system
• For resolution critical methods, H₂ offer the best alternative
  – Agilent GC and GC/MS systems have many built-in safety features

• For many GC applications, N₂ offers a cheap, easy alternative without any safety worries
  – Many existing helium methods have too much resolution
  – N₂ can be used without changing any of the existing GC conditions
    • Keep the holdup time the same as the original method
  – 2-D methods have high resolution built in, so N₂ is ideally suited as a carrier gas
    • Valve-based or Deans switch (not GC x GC flow modulation)

• For more information on Helium Carrier Gas
  www.agilent.com/chem/heliumupdate
Contact Agilent Chemistries and Supplies Technical Support

1-800-227-9770 option 3, option 3:

Option 1 for GC and GC/MS columns and supplies
Option 2 for LC and LC/MS columns and supplies
Option 3 for sample preparation products, filtration, and QuEChERS
Option 4 for spectroscopy supplies

Available in the USA 8-5 all time zones

gc-column-support@agilent.com
lc-column-support@agilent.com
spp-support@agilent.com
spectro-supplies-support@agilent.com