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Helium Conservation and Alternate Carrier Gas Strategies for GC and GC-MS Analysis in Conjunction with GERSTEL Instrumentation

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This GERSTEL presentation provides critical information for the safe use of hydrogen as a carrier gas with its products. All guidelines presented here must be followed.

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Summary of Steps for Helium Conservation*

1. Perform a helium use audit
2. Institute helium conservation methods to reduce use by 95%
3. Use nitrogen as carrier gas – GC only
4. Use hydrogen for carrier gas – GC and GC-MS

*Helium is the preferred carrier gas for use with GC and GC-MS. The best strategy is to continue to use helium to avoid having to revalidate methods and/or deal with the many issues that need to be addressed when using hydrogen.

Step 1 - Check Entire Laboratory for Leaks/Unnecessary Helium Usage

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Develop a spreadsheet with the following information:

- Each system that uses helium
- Determine the total flow used by each system by doing the following:
  - Determine inlet and column flows (split flow, septum purge, column flow)
  - Include helium being used as detector make-up gas
  - Estimate flow for any other components using helium (valved systems, special options, etc.)
  - If you have a large number of systems use 100 mL/min as a rough estimate
- Check each system and helium delivery infrastructure (manifolds, regulators, etc.) for leaks
- Compare actual usage to theoretical usage. If close, leaks are under control.

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Step 2 - Implement He Conservation Measures

He conservation procedures in order of difficulty and expense:

- Optimize split ratios (lowest practical flow)
- Activate "Gas Saver" in GC Methods
- Use nitrogen as detector make-up gas
- Switching to nitrogen as carrier gas when not in active use. Agilent's He Conservation Module (Option 303/G3529A) does this automatically, or a manual 3-way Swagelok valve can be used.

Helium Conservation Measures Results*

- Optimizing split ratios, activating “Gas Saver” mode and using nitrogen as detector make-up gas can reduce helium usage by 85%
- Doing all the above and switching to nitrogen as carrier gas when not in active use can reduce helium usage by 95%
- Agilent provides tools for helium conservation. Examples can be found at links below:
- https://www.agilent.com/en/products/gas-chromatography/gc-systems/handle-the-helium-shortage
- https://www.agilent.com/en/product/gas-chromatography/gc-systems/helium-conservation-cost-savings-calculator

*From Agilent He conservation presentation

Conserving Helium with GERSTEL ePneumatics

For TDS, TDU 2, TD 3.5+, and ALEX systems, helium can be further conserved by using a GERSTEL ePneumatic

- ePneumatic allows Maestro to control the TD and CIS flows directly
- Thus, when switching tubes (TD) or liners (ALEX), Maestro can reduce or stop the carrier flow during the switch
- Maestro can also reduce the flows when the GC is not in use, by means of a ‘standby’ method
- Although a small amount of air may enter the GC column if the carrier is shut off completely, the column is always cool at this point so neither the column phase or the MSD will be harmed

Carrier Gas Decision Tree*

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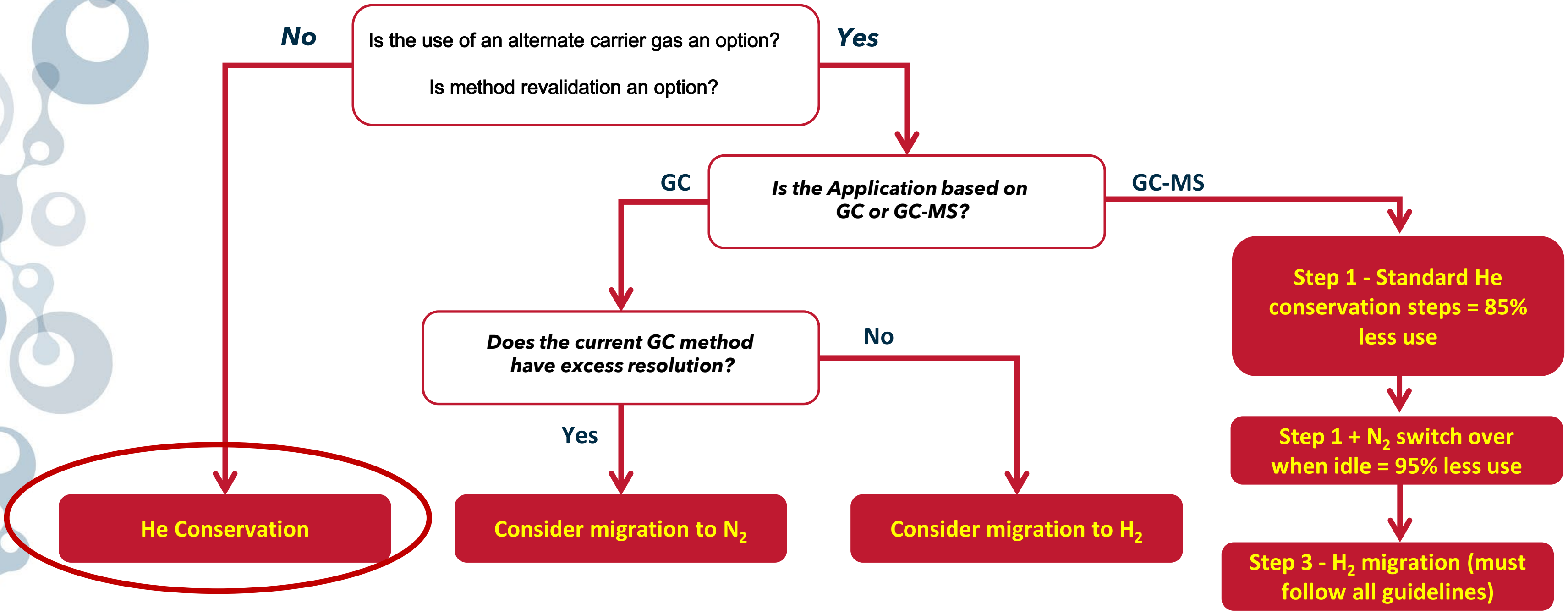

Method revalidation not required!

*Adapted from Agilent He conservation presentation

If He Conservation Measures are not Enough

- If resolution for your GC method is high enough, consider switching to nitrogen. Nitrogen is not suitable for use with GC-MS.

- For GC-MS consider switching to hydrogen as carrier gas, but there are many factors to consider – some key factors are:
  - All safety measures recommended by the GC manufacturer must be followed
  - H_2 is reactive and methods may need to re-validated. Non-targeted analyses are risky since key compounds can react/disappear
  - Libraries such as NIST are based on He as carrier gas, so search results when using H_2 can be inaccurate
  - Pumping capacity of H_2 is ½ that of He so a turbo pump is recommended, and column dimensions need to be optimized for H_2.
  - Detection limits are poorer when using H_2 with MS

Agilent 5977C GC-MSD System

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- Agilent had introduced a new series of MSDs that have been optimized for use with H_2 as carrier gas. The main difference is a more inert source (HydroInert Source) that largely overcomes the problems seen with the standard inert source. This source can be installed on appropriate 5977B MSDs,
  - The HydroInert source cannot guarantee that the system will perform as well or the same as with He, so the user must be aware that all issues seen when using H_2 and the standard source may still be present, even if at a lesser degree.
    - Methods may need to re-validated. Non-targeted analyses are risky since key compounds can react/disappear
    - Libraries such as NIST are based on He as carrier gas, so search results when using H_2 can be inaccurate
    - Pumping capacity of H_2 is ½ that of He so a turbo pump is recommended, and column dimensions need to be optimized for H_2.
    - Detection limits are poorer when using H_2 with MS

Pros & Cons for using Hydrogen as Carrier Gas

Pros	***Cons***
Less expensive, readily available carrier gas	Sensitivity decreases up to 30% (MSD); New sensitivity specs for H_2 use show this
Ionization source requires less maintenance	NIST database is not designed to work with hydrogen as carrier gas
Average 1.5x faster analysis due to higher optimal flow velocity with constant partition height and pressure	More extensive safety precautions and knowledge required from user
No retention shift if constant linear velocity is used	MS has to be tuned after changing the carrier gas; some target tunes (DFTPP, etc.) may not work at all with H_2
Use of gas generators eliminates the need to handle and resupply H_2 gas cylinders	**Increased compound degradation due to H_2 reactivity**

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General Information on Hydrogen

Hydrogen can produce an explosive mixture with air in a range from 4-74.2 % (v./v.). Self ignition can occur at 630 °C, or an open flame or electronic spark can ignite the mixture. Hydrogen can also spontaneously ignite when released from high pressure to low pressure.

Calculation of the potentially explosive atmosphere

Room Size: Area 10 m² -> Volume 20 m³

Flow: 200 mL/min -> To reach an explosive atmosphere of 4% in space, a GERSTEL thermal desorption system (for example) would have to be open for 66.66 hours with no air exchange (e.g., the room would have to be sealed).

Hydrogen Safety Instructions

- The GC manufacturer's safety instructions for using hydrogen as a carrier gas must be followed at all times.
- The pertinent Agilent guidelines and manuals are provided below.

- https://www.agilent.com/cs/library/usermanuals/public/user-guide-coverting-ei-gcms-instruments-5994-2312en-agilent.pdf
- https://www.agilent.com/cs/library/usermanuals/public/user-manual-gcms-hydrogen-safety-g7003-90053-en-agilent.pdf
- https://www.agilent.com/cs/library/usermanuals/public/usermanual-gc-safety-8890-g3540-90010-en-agilent.pdf
- https://www.agilent.com/cs/library/usermanuals/public/usermanual-gc-operation-8890-g3540-90014-en-agilent.pdf

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Agilent Technologies

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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 Delonno
Product Safety Engineer

Excerpt from the document

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

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Special consideration is necessary when operating these products due to open flow paths during tube or liner exchange:

- TDU, TDU 2, TD 3.5+
- TDS, TDS 2, TDS 3, TDS 3C
- ALEX

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Safety Precautions for hydrogen as carrier gas in combination with Thermal Desorption and ALEX

To prevent H_2 concentrations above 4% these precautions should be followed:

- General conditions when using Agilent EPC:
  - TD desorption flow 50 mL/min
  - Connect the split vents to an exhaust system
- GERSTEL ePneumatics is recommended
  - Carrier gas is switched off when transport adapters are exchanged, and the TD inlet is open
  - Make sure that standby flow is as low as possible

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- Fume Hood/Exhaust Vent
  - To prevent local hydrogen accumulation, there should always be a local fume hood or an overhead exhaust vent to assist in room air exchange.
- Hydrogen sensor in the lab
- Hydrogen generator
  - The hydrogen generator shuts off the gas supply in the event of a leak when consumption exceeds the delivery capacity (typically 200-300 mL/min).
  - The generator also shuts down if the lab loses power, which prevents H_2 accumulation if the lab air handling system also loses power.

Using Hydrogen with TD: Additional Considerations

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- No sorbent manufacturer guarantees their sorbents performance or robustness when used with hydrogen
- Analytes may be altered during tube desorption, particularly alkenes and alkynes. The sorbent and/or the metal tube surface may act as a catalyst for hydrogenation at the surface.
- Some sorbents my degrade faster when hydrogen is used [porous polymers (Tenax TA, GR) and glassy carbons (Carbopack X, etc.)].
- Hydrogen should not be used for non-targeted work with TD due to the changes that could occur. This goes for tubes packed with sorbents, Twister/TF-SPME, or thermal extraction experiments.
- TD methods that were developed under helium will need to be re-developed and re-validated under hydrogen.

These GERSTEL products are not supported with hydrogen as carrier gas

- CIS 6 – Self ignition hazard due to operating temperature above 630 °C
- PEGS - Depends on configuration
- PYRO - Pyrolysis per definition requires an inert atmosphere

GERSTEL products that can be used at your own risk with hydrogen as carrier gas

- ODP 3, ODP 4

  When used as sniff ports, inhalation of hydrogen occurs [conc. in the range of low mL/L (v/v), assuming 1 mL/min carrier gas flow, 5-50 mL/min make-up gas flow and approximately 4-8 L/min inhalation]; although there is no known toxicity data for hydrogen exposure to humans, users should refer to local laws and consider contacting an industrial hygienist for advice regarding hydrogen inhalation and its possible physiological impact.