

Optimizing HydroInert EI Source Functionality and Longevity

Introduction

The benefits of hydrogen in gas chromatography (GC) are widely accepted, as it can be generated in the lab in a cost-effective manner, produces fast chromatography, and allows for higher sample throughput. Since hydrogen is a reactive gas, hydrogenation and dechlorination reactions can and do occur in the mass spectrometer electron ionization (EI) source. These reactions can make applying hydrogen carrier gas to many applications difficult. A specially designed extractor source for the Agilent Inert Plus GC/MSD and triple quadrupole GC/MS systems address these hydrogen-related issues and improves performance with hydrogen carrier gas. The Agilent HydroInert source (Figure 1) with H₂ carrier gas retains mass spectral fidelity and can allow users to continue to use existing helium-based mass spectral libraries.

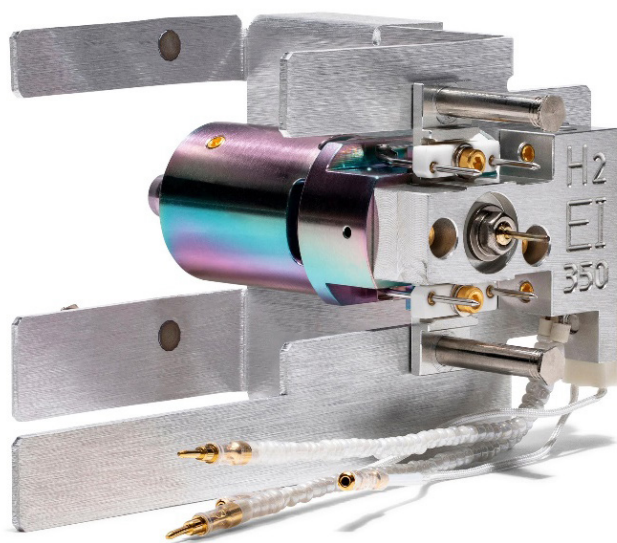


Figure 1. The Agilent HydroInert source.

When adopting hydrogen for GC/MS analysis, there are several factors to consider. First, hydrogen is a reactive gas, and it may potentially cause chemical reactions in the inlet, column, and sometimes the MS EI source, which can change analysis results. To address potential issues in the MS source while using hydrogen, the Hydrolnert source was used. Additional information can be found in the Agilent technical overview of the Hydrolnert source.¹ Second, for GC/MS applications, hardware changes in the gas chromatograph and mass spectrometer are recommended when switching to hydrogen carrier gas. The Agilent Helium to Hydrogen Carrier Gas Conversion Guide² describes in detail the steps for conversion from helium to hydrogen carrier gas. Lastly, it is recommended that when working with flammable or explosive gases, laboratories maintain proper safety in gas handling and use. Further information on the safe use of hydrogen can be found in the Agilent Hydrogen Safety Manual³ and Hydrogen Safety for the Agilent GC System Guide.^{4,5}

Reduced source cleaning

One of the advantages observed with hydrogen carrier gas is a reduced need for EI source cleaning. Less required cleaning translates to more uptime and reduced cost.⁶ This has been observed when using the Agilent JetClean self-cleaning ion source, which introduces a low flow of hydrogen into the source during or after analysis.⁷ The same reduction of required source cleaning is also observed with the Hydrolnert source.

A set of experiments was completed to investigate source lifetime using a complex soil matrix and Environmental Protection Agency (EPA) method 8270 target analytes and quality control criteria to evaluate when the GC or MS required maintenance. One set of experiments used helium carrier gas with an extractor source and 9 mm extraction lens, while the other experiment set used hydrogen carrier gas with the Hydrolnert source and 9 mm (Hydrolnert) extraction lens. The helium GC/MS system required source cleaning after an average of 365 matrix injections (581 total injections). For the Hydrolnert source, 5,200 matrix injections were completed before the source failed acceptance criteria.

Restoring Hydrolnert performance

The Agilent Hydrolnert source is constructed with materials that greatly reduce undesirable reactions in the source to maintain spectral fidelity when used with hydrogen.¹ Due to the source design, there is no way to mechanically or chemically clean Hydrolnert source parts without compromising the source. As mentioned previously, hydrogen gas in concert with electrons can clean off a vast majority of organics from matrix entering the source. This reduces the interval for source cleaning. However, over time and with extended use, parts of the Hydrolnert source will begin to show reduced functionality. Hydrogen does not have the ability to remove inorganic deposits on source parts, some of which originate from column bleed. When inorganic materials start to deposit on source parts, charge distribution across those parts can become disrupted. When functionality is reduced, it is recommended to replace the parts listed below at a suitable interval of time. The exact time to reduced functionality will be dependent on several application factors discussed in detail below. In many cases, replacing the extraction lens and repeller of the Hydrolnert source will restore the functionality of the source. However, depending on application conditions, incremental replacement of the ion body or source ceramics may also be required.

Parts list

- Repeller (part number G7078-20902)
- Repeller insulator (part number G1099-20133)
- Ion source body (part number G7078-20903)
- Transfer line tip seal (part number G3870-20542)
- Extractor insulator (part number G3870-20445)
- 9 mm Extractor lens (part number G7078-20909)

Best practices to increase HydroInert longevity

When running hydrogen carrier gas with any source, but especially with the HydroInert Ion source, good GC/MS practices cannot be ignored. While running hydrogen has been shown to clean sources, hydrogen carrier will not eliminate the issues that plague poor operation and maintenance of GC/MS systems.

Maintain leak-free and water-free carrier gas

Hydrogen carrier gas is not an inert gas. In an ion source, H_2 gas will separate into $2H^+$. H_2O in the presence of H^+ in the ion source makes H_3O^+ . N_2 in the presence of H^+ in the ion source makes N_2H^+ . Further, N_2 in the ion source significantly reduces ion creation and transmission into the quad. Additionally, as little as 50 ppm of O_2 causes damage to the column's stationary phase, which can lead to elevated column bleed.

Due to the unique redox chemistry that can occur in the ion source with hydrogen carrier in the presence of leaks, it is recommended that more time and care be taken to ensure the system is leak-free. The Agilent Gas Clean System should always be equipped on the gas lines into the GC. Gas Clean cartridges are designed to scavenge H_2O and O_2 from the carrier gas source. When first pumping the MS down, set the oven, inlet, and transfer line temperatures to < 100 °C. Once the system is confirmed by the Air and Water check to be leak-free, then heat the other components up to temperature. Temperatures below 100 °C are less likely to start the process of column bleed. Once the system has been equilibrated and is confirmed to be leak-free, it is safe to use the HydroInert source.

Use low-bleed, micro-bore GC columns within recommended operating temperatures

Hydrogen is a less viscous gas than helium. To maintain positive head pressure, a micro-bore column diameter of 180 μm is recommended with a flow between 0.6 and 0.9 mL/min. Larger diameter, narrow-bore columns of 250 μm can be used with column lengths of 40 m or longer. Larger ID columns require higher flow rates that may compromise ionization efficacy.

Excessive column bleed can contribute to reduced functionality and increased maintenance of ion sources. Column bleed cannot be completely avoided, but can be properly controlled. First, MS columns designed with low-bleed polymers can significantly reduce interfering siloxane bleed ions in mass spectrometer applications and are therefore highly recommended. The ultra-low bleed Agilent DB-5Q and HP-5Q GC columns are ideal for use with hydrogen carrier. Columns designated as "MS" are also recommended. Secondly, thin-film columns with short column lengths are also recommended to reduce the amount of stationary phase, the origin of column bleed, in the sample flow path. It is recommended to start with a DB-5Q or HP-5Q with 20 m \times 0.18 mm, 0.18 μm dimensions.

Because temperature significantly impacts the amount of column bleed, it is important to observe the thermal programming of your GC column. Every GC column has a maximum allowable operating temperature (MAOT), which will vary based on the type of column phase. This represents the highest temperature to which your column should be exposed to limit premature deterioration. Maximum temperatures are commonly stated with two numbers, for example 325/350 °C. The first, lower number is the MAOT for isothermal analysis. The second, higher number is the MAOT when using thermal gradients or oven programs. Operate your oven programs within the operating limits to optimize column lifetime and limit the impact of bleed.

For more detailed information on controlling column bleed, please see refer to the following technical overview: "How Does Bleed Impact GC/MS Data and How Can It Be Controlled?"⁸

Elevate the source temperature

Tuning the mass spectrometer should always occur with the chosen method temperature and method parameters active. This ensures the mass spectrometer is optimizing itself for the conditions meant to be run. To increase the longevity of the HydroInert, it is recommended to run the source at 275 °C or higher. For example, for PAH analysis, a source temperature of 320 °C is ideal.⁹

It should be noted that the optimum temperature for the tune compounds in PFTBA, which is infused into the source during tuning, was determined to be 230 °C. Often, this temperature will remain as a default in many applications; however, that does not mean that 230 °C is optimum for use with hydrogen carrier. It should also be noted that running the "Tune Eval" function at any source temperature other than 230 °C could result in values on the report that are not considered passing. The "Tune Eval" feature was designed to evaluate the tune at 230 °C and the values within the report are the optimum values for PFTBA at 230 °C. If the "Tune Eval" function is part of a customer SOP, it is recommended that the instrument be tuned at 230 °C, the "Tune Eval" be run, then the source temperature and methodology should be loaded, and the instrument retuned under running conditions.

Conclusion

Agilent HydroInert source longevity is dependent on application and method conditions. However, by using some of the best practices highlighted above, the longevity of HydroInert parts can be prolonged. The HydroInert source cannot be mechanically or chemically cleaned, thus when source parts show reduced functionality, replacement of the recommended parts above is required to restore functionality. Replacement intervals will be method- and application-dependent.

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DE-000077

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Printed in the USA, August 6, 2024
5994-7680EN