

Introduction

Hydrogen is widely recognized for its benefits in gas chromatography (GC), including cost-effective generation in the lab, rapid chromatography, and higher sample throughput. However, its reactivity can lead to hydrogenation and dechlorination reactions in the mass spectrometer electron ionization (EI) source, complicating its application in many scenarios. To address these issues, Agilent has developed a specially designed extractor source for the Inert Plus GC/MSD and triple quadrupole GC/MS systems, enhancing performance with hydrogen carrier gas. The Agilent Hydrolnert source maintains mass spectral fidelity and allows users to utilize existing helium-based mass spectral libraries.

Due to the design of Hydrolnert, there is no way to mechanically or chemically clean the source parts without compromising source integrity. However, over time and with extended use, parts of the Hydrolnert source will begin to show reduced functionality. Excessive column bleed can contribute to reduced functionality in all EI sources. Column bleed cannot be completely avoided but can be properly controlled. Low-bleed MS columns, such as the Agilent DB-5Q and HP-5Q GC columns, are recommended for use with the Hydrolnert Ion source. Ultra-low bleed columns are ideal for enhancing data accuracy, spectral fidelity, and baseline integration. The ultra inert performance of 5Q columns further increases sensitivity for trace-level analytes and provides balanced deactivation for multiclass analyte panels.

This study investigates how the 5Q series of columns impact the performance and durability of the Hydrolnert source tune. The study involved cycling through 10,000 oven temperature ramps without any injections to assess the effects of column bleed from the 5Q phase. Furthermore, the study examined how the combination of sample matrix and column bleed from the 5Q phase affects source longevity.

Experimental

Hydrolnert Functionality with 5Q Columns (no Injection)

An experiment was designed to investigate the effects of ultra low bleed columns (HP-5Q) on the functionality of the Hydrolnert source over time. A method simulating a large oven temperature ramp cycle (Table 1) over 12 minutes was created. The syringe was removed from the ALS tower to simulate injections, but to isolate the source of siloxanes to column bleed from the HP-5Q only. 10,000 injections were executed, and a system tune was performed every 100 injection to monitor source parameter shifts.

Experimental

The Agilent 7000E was run in SCAN mode, scanning from 50-450 m/z during each run. Air leaks were detected and immediately remedied when found to ensure the experiment was free from oxides. Liner and septum were not changed during this experiment.

Table 1. Method parameters for no injection experiment

| GC Parameter | Value |
|-------------------|-----------------------|
| Run Time | 12 mins |
| Oven Ramp | 60 to 320 @ 30 °C/min |
| Column Flow | 0.8 mL/min |
| MSD Transfer Line | 320 °C |
| Inlet Temperature | 250 °C |

Hydrolnert Functionality with 5Q Columns when Injecting a Complex Soil Matrix Extracted for PAHs

A second experiment was designed to investigate the effects of ultra low bleed columns (DB-5Q) and matrix on the functionality of the Hydrolnert source. A 10 minute, two column, method was developed to monitor samples in SCAN mode (Table 2). The septum and liner were changed every 90 injections. To keep the system clean the low-frit liner (5190-5112) was used for this application. The Agilent 7000E was run in SCAN mode, scanning from 50-450 m/z during each run. No air leaks were detected during this experiment.

Table 2. Method parameters for soil injection experiment

| GC Parameter | Value |
|-------------------|-----------------------|
| Run Time | 10 mins |
| Oven Ramp | 60 to 320 @ 35 °C/min |
| Column 1 Flow | 0.6 mL/min |
| Column 2 Flow | 0.8 mL/min |
| MSD Transfer Line | 280 °C |
| Inlet Temperature | 300 °C |



Figure 1. The Agilent Hydrolnert Source

Results and Discussion

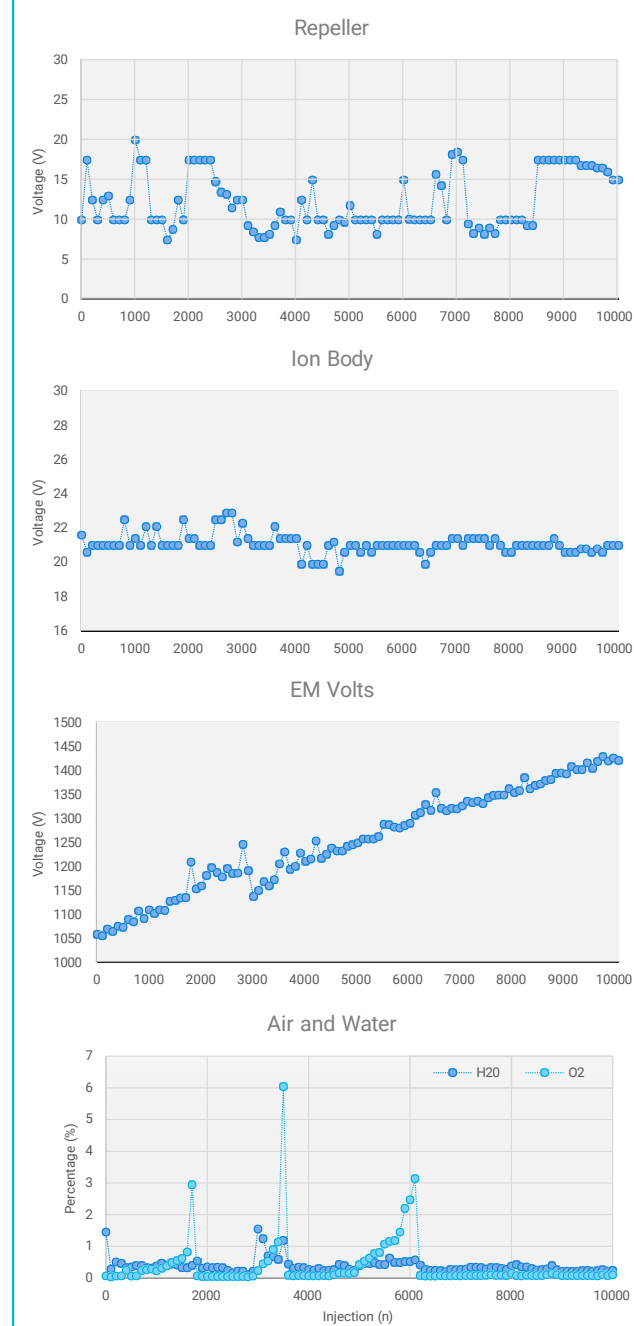


Figure 2. Repeller voltage (V), ion body voltage (V), Electron multiplier voltage (V), and air and water percentage (%) every 100 injections for 10,000 injections on an HP-5Q with no injections.

Results and Discussion

Hydrolnert Functionality with 5Q Columns (no Injection)

Results of the effects of 10,000 oven temperature cycles on the Hydrolnert source tune parameters are shown in Figure 2. Repeller and ion body voltages are set automatically by the tune algorithm to maximize the transference of ion on the 7000E. Throughout the 10,000 injections the ion body voltage remained stable. The EM voltage gain across 10,000 injections was as expected. The source was in use for 120,000 minutes during this study and data was being collected throughout the duration of the study in SCAN mode. During this study, three leaks were detected at the MS transfer line and were corrected as soon as they were detected. The leaks are reflected in the spike in O2 percentage.

A new ion body will typically have a voltage of 16-22 V on a Hydrolnert source. When an ion body begins to lose functionality the ion body voltage will begin to rise to compensate for the loss. Eventually the ion body voltage tops out at 29-30 V and the tune begins to show lower abundance of tune ions, until the source fails to tune. In the current study the ion body voltage was set by the tune algorithm between 19-24 V. This is a stable and normal operating range for the ion body. It is important to remember that a single elevated ion body tune is not cause for concern, there needs to be a trend of elevation over time.

The overall purpose of this first study was to examine what baseline changes would occur to the Hydrolnert tune parameters when using a 5Q column in leak free conditions. Water and oxygen are known to speed the liberation of column bleed, which in turn reduces the functionality of the Hydrolnert source. This study shows that under leak free conditions, with a 5Q column, the Hydrolnert source can function optimally for an extended period. This study adds to a growing body of evidence that has demonstrated one of the main factors in Hydrolnert longevity is leak state of the system.

Hydrolnert Functionality with 5Q Columns when Injecting a Complex Soil Matrix Extracted for PAHs

The results of the effects of complex matrix, while using a 5Q column, on the Hydrolnert source tune parameters are shown in Figure 3. The study was terminated at 4,500 injections when the source failed to tune. Throughout the study the repeller voltage remained stable and within expected operating ranges. The ion body voltage ranged from 20-29 V, showing an increasing trend with increasing sample injections. The EM voltage gain showed the same increasing trend as seen in Figure 2. This system was run for 40,000 minutes during the experiment where the MS was collecting data in SCAN mode.

Throughout the study, no leaks were detected, which is attributed to the frequent liner and septum changes. Another factor influencing the longevity of the Hydrolnert source is the complexity and makeup of sample matrix. Within complex matrix there will be reactive oxygen species that interact with the hydrogen carrier gas and reduce the functionality of the Hydrolnert source over time. Eventually the repeller, ion body, or extraction lens of the Hydrolnert source will need to be replaced on an as needed basis. In the current study, an extracted soil matrix was used. This sample type represents a realistic running scenario for a Hydrolnert source. The rise in the ion body voltage is as expected as is the performance over 4500 samples before the need for source maintenance.

The combination of the two studies presented demonstrates that using Q phase columns with the Hydrolnert source, under leak free conditions, can provide an extended longevity for the source. However, longevity of the source is also matrix and application specific. By using good MS technique, which includes sample cleanup, maintaining a leak free system, and regular inlet maintenance, the longevity of the Hydrolnert source can be maximized for the specific application.

Conclusions

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

More Information



Agilent Technical Overview 5994-7680EN

