Helium savings and method optimization for the analysis of Dioxin/Furans and other POPs with Magnetic Sector GC-HRMS using a helium saver solution

Heinz Mehlmann and Dirk Krumwiede , Thermo Fisher Scientific, Hanna-Kunath-Str.11, 28199 Bremen, Germany

Abstract

Purpose: Helium shortage is a global concern, affecting laboratories and operational costs. Analytical methods need to have solutions to avoid the challenges caused by helium shortage. Thermo Scientific™ DFS™ Magnetic Sector High Resolution GC/MS System is the golden standard for high sensitivity analysis of Dioxins and other POPs. Already for decades it has been proving its proficiency in this field of analysis and thus became the established analysis technique available nowadays in leading Dioxin laboratories throughout the world. Using the helium saving option, the helium consumption can be reduced drastically by substituting all GC flows by nitrogen except the flow in the analytical column is Helium.

Figure 1. Thermo Scientific DFS Magnetic Sector GC-HRMS system S coupled to two Thermo Scientific™ TRACE 1610 GCs.

Methods: For a direct comparison between a standard SSL Injector and an SSL with the Thermo Scientific™ HeSaver-H_2Safer kit, a dual GC configuration of a DFS Magnetic Sector GC-HRMS system was used. One GC using the standard SSL injector and one GC using an SSL injector with the HeSaver-H_2Safer kit option. Both columns from both GCs are connected to the Ion Source of the MS.

Results: No discrimination in terms of peak-shape or sensitivity was observed for Dioxin/Furan or PCB application when using the HeSaver-H_2Safer kit option .

Introduction

With the background of Helium shortage and increasing price, the HeSaver-H_2Safer kit is an easy way to save Helium without compromising performance or change existing Methods and Instrument parameter. The maximum saving of Helium using the HeSaver-H_2Safer kit option is in split less injection mode is calculated to approx. a factor of 8 compared to a standard SSL Injector. In split mode, the saving is calculated to approx. a factor of 3 compared to a standard SSL Injector. As low level of Dioxins are typically done in split less injection a maximum possible saving of Helium can be achieved for this type of application.

The standard SSL injector module on the TRACE 1610 GC can be easily upgraded with the HeSaver-H_2Safer option. Therefore, a small valve box will be installed at the back of the GC mainframe. The top gas connection will be connected to the carrier gas net of the GC and the lower connection to the Helium supply. The control cable is plugged into the according slot. The standard gas supply for the injector is connected to the Nitrogen supply.

Figure 2. Valve module installed at the back of a TRACE 1610 GC.

The inner injector body of the SSL injector module needs to be changed with the HeSaver-H_2Safer injector body.

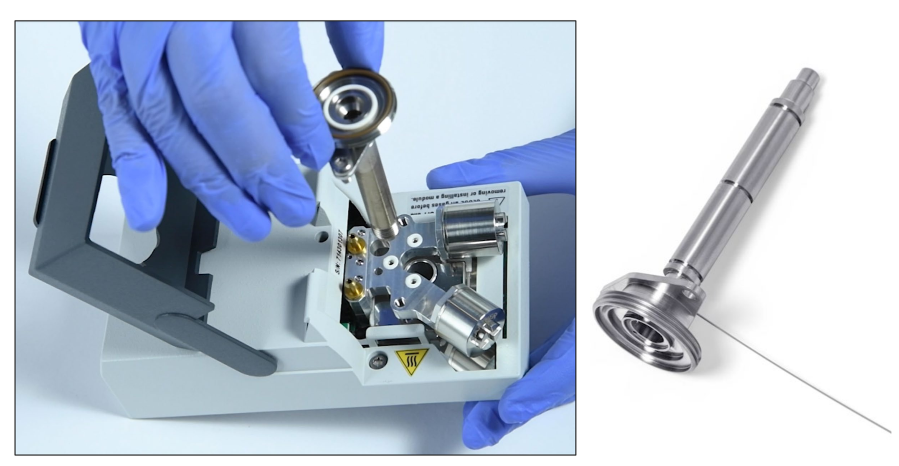

Figure 3. Exchanging the injector body of an SSL Injector module with the HeSaver-H_2Safer injector body (right side)

The Nitrogen gas supply is delivered via the gas net of the GC located underneath the GC module. The Helium gas supply enters the injector body via a thin stainless-steel capillary at the top of the injector body. The capillary is connected once the Injector module is installed The modified SSL injector module will be installed like the standard module except that the Helium capillary is connected to the carrier gas net of the GC mainframe.

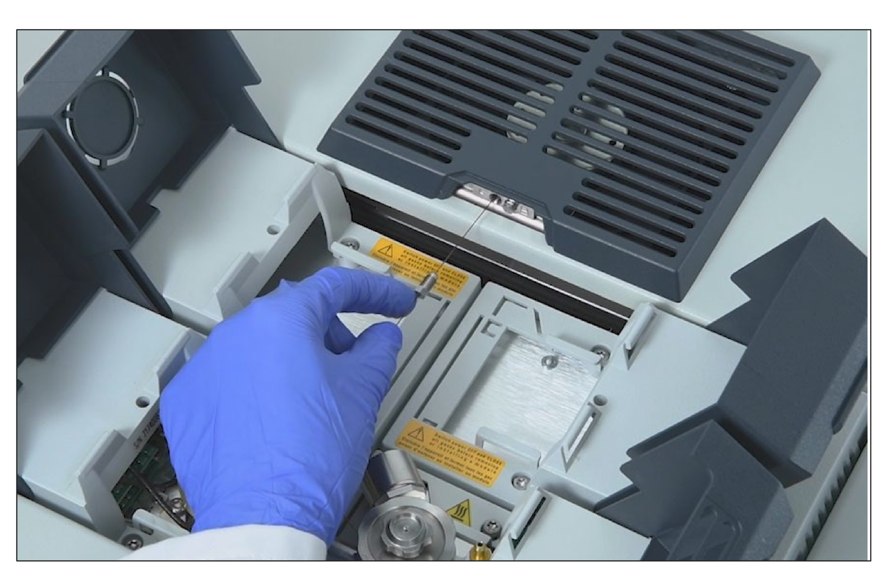

Figure 4. Installing of the SSL Module with the HeSaver-H_2Safer module and connecting the Helium gas tube.

The gas consumption of the standard SSL injector is mainly driven by the split flow and the septum purge. The amount of Helium as carrier gas in the column is minor. Typically values for the split flow is 50 ml/min and 5 ml/min for the septum purge. A standard value for the Helium carrier gas flow is 1 ml/min.

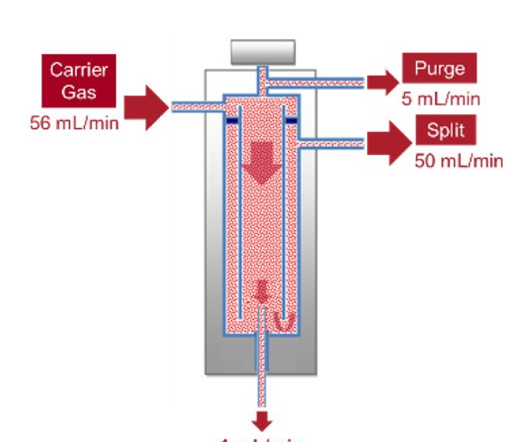

Figure 5. Example of typical values for the different gas flow rates using a standard SSL injector.

The principle of the HeSaver-H_2Safer kit option is to substitute the split and purge flow with Nitrogen. To ensure that during the measurement no Nitrogen is entering the column a special setup is used at the bottom of the injector body. The carrier gas line is connected via a T-junction with one end connected to the column capillary the other end with a restriction towards the liner. This Anti-diffusion tubing ensures that during the measurement only Helium enters the column.

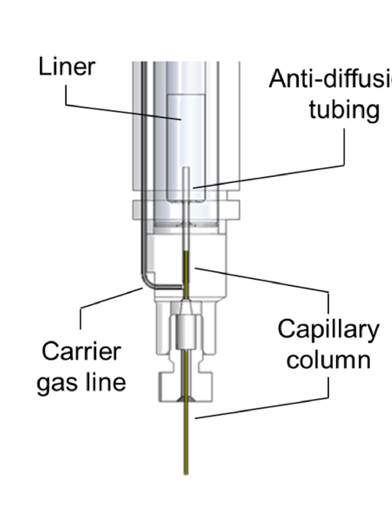

Figure 6. Schematic of the HeSaver-H_2Safer injector body at the bottom with the Anti-diffusion tubing.

During the injection phase, a sample is injected, vaporized, and transferred by the pressurizing gas flow into the column. During this phase, the valve connected to the carrier gas line is closed, and only a very small gas flow (0.1 mL/min) passes through the line to prevent contamination and possible carryover. Immediately after the injection phase, the valve opens, and 9 mL/min of Helium or Hydrogen pass through the carrier gas line. Part of this flow feeds
the column according to the flow rate set in the method, and the rest is backflushed into the injector avoiding the pressuring gas and other possible contaminants from entering the column. During the injection, the pressurization gas enters the column which leads to a Nitrogen peak in the first part of the analysis.

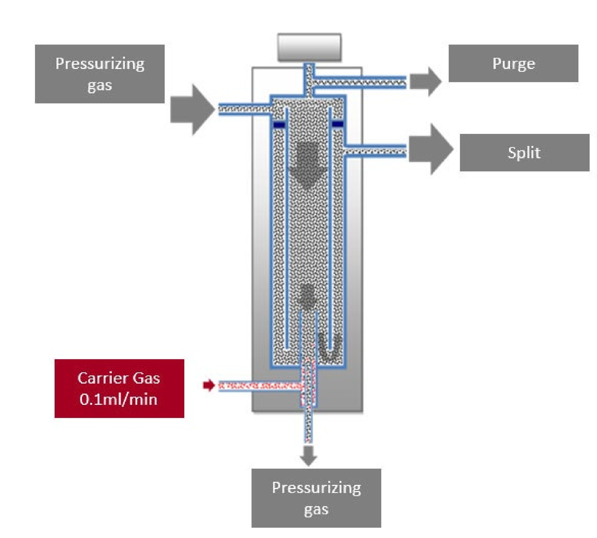

Figure 6. Schematic of the gas flows during the injection.

During the analysis the carrier gas valve opens and a total Helium flow of approx.. 9 mil/min enters the injector body where it is distributed into the anti-diffusing tube (Approx. 9mil/min) and 1 ml/min into the column.

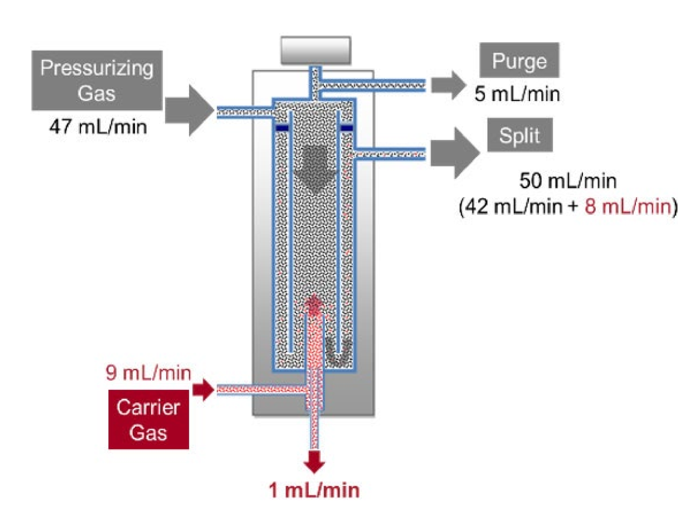

Figure 7. Schematic of the gas flows during the measurement.

Materials and methods

Materials

The configuration used in this study consists of two TRACE 1610™ GC, one GC equipped with the HeSaver-H_2Safer kit option using two columns coupled to the DFS Magnetic Sector GC-HRMS. The Thermo Scientific™ TriPlus™ RSH Autosampler with extended x-rail served both GCs from one common sample tray.

Software platform for the DFS Magnetic Sector GC-HRMS control software and the data acquisition was Thermo Scientific™ Xcalibur™ 4.2 software. TRACE 1610 GCs and TriPlus Autosampler were handled by the Thermo Scientific™ Chromeleon™ based SII Driver for Xcalibur.

Test method

The mass spectrometer was set up in a multiple ion detection mode (MID) at a resolution of 10,000 (10% valley definition). Heptacosafluortributylamin (FC43) and Perfluorkerosin (PFK) was used as a reference compound to provide the inherent lock mass and calibration masses.

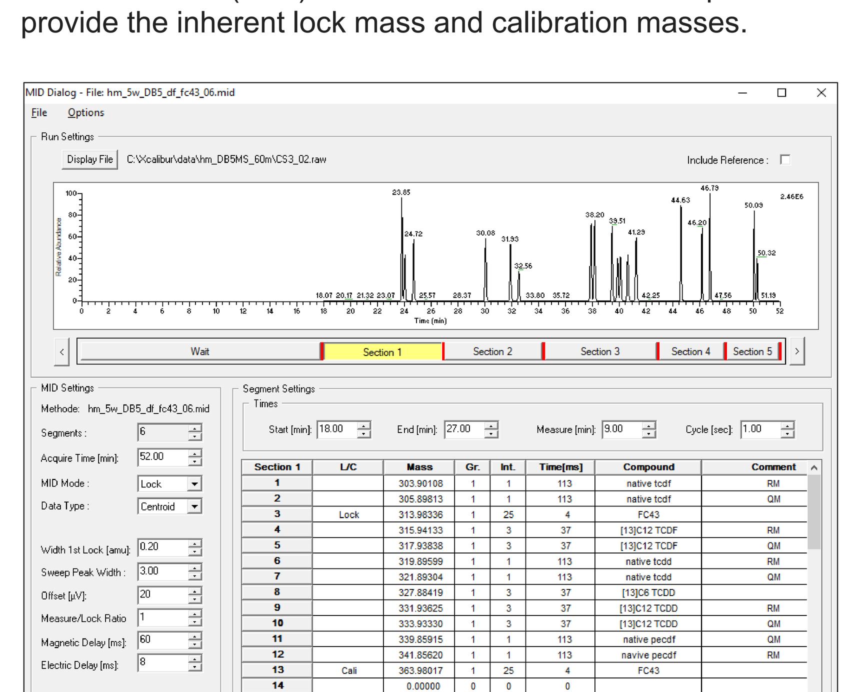

Figure 8. Example of the multi-ion detection settings for Dioxins.

One micro liter of sample was injected in split less mode on a DB5MS length: 60m x ID: 0.25mm x film: 0.25um column . A method 1613 calibration standard EPA 1613 was used as well as EPA method 1668 standards to demonstrate the chromatographic performance of the system.

Results

Chromatographical performance using the HeSaver-H_2Safer module.

A direct comparison between a standard SSL injector and the one equipped with the HeSaver-H_2Safer kit option showed no loss in performance for Dioxin and PCBs application. Difference in Peak shape, Intensity and Retention Time are within expected deviation. There was also no additional peak tailing observed.

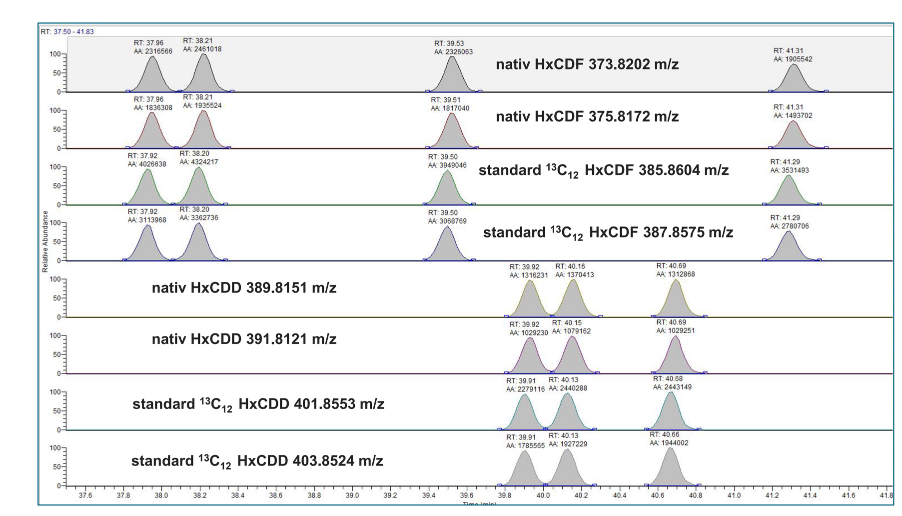

Figure 9. Example of Hexa-Furan and Dioxin of an EPA 1613 Dioxin calibration standard.

For Dioxin application a good chromatographically resolution could be archived. The example of the HxCDF/D showed a good baseline separation.

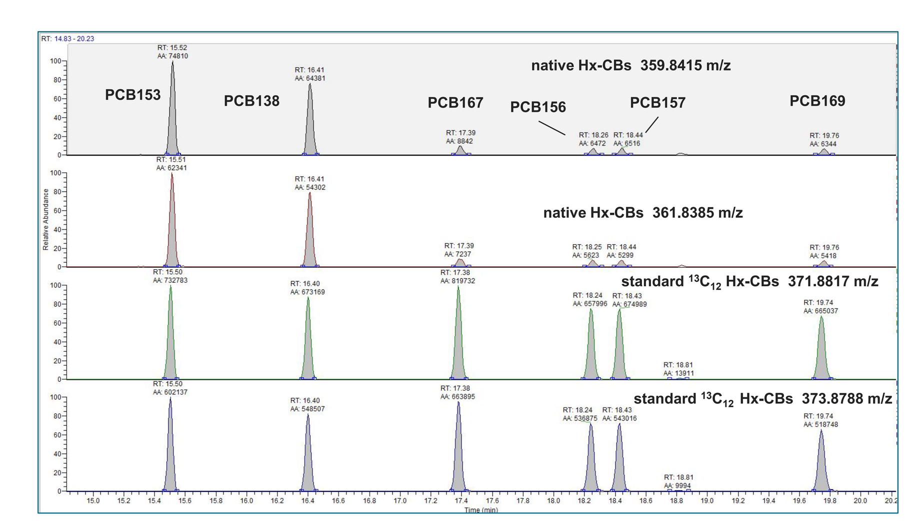

Figure 10. Example of Hexa-PCBs of an PCB calibration standard.

During the injection process the sample transfer onto the column is done by the pressurizing gas (Nitrogen). Therefore, Nitrogen enters the insource shortly after the injection. However, this has no negative influence as the solvent delay were filament and accelerating voltage are both off, is far longer than the duration of the Nitrogen peak.

Conclusions

- The HeSaver-H2Safer technology offers the advantage of reduced helium gas consumption without compromising GC-MS performance, through a smooth and simple upgrade of a standard SSL injector module.
- Existing methods can be used unchanged, with consistent analytical performance in terms of injection repeatability, analyte transfer, linearity, recovery, and robustness.
- Standard consumables such as liner, O-Rings and septa can be used.
- No discrimination in terms of peak-shape or sensitivity was observed for Dioxin/Furan or PCB application.
- The maximum Helium saving can be archived when using split less injection, which is the standard approach for low level Dioxin/Furan or PCB applications.

References (if necessary)

1. "Addressing gas conservation challenges when using helium or hydrogen as GC carrier gas "; Thermo Fisher Scientific Technical Note 001218

2. US EPA Method 1613 revision B

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Science at a scan
Scan the QR code on the right with your mobile device to find out more about the DFS Magnetic Sector GC-HRMS System.