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Gas chromatography

Addressing gas conservation challenges when using helium or hydrogen as GC carrier gas

Authors

Giuseppe Scollo[1], Ian Parry[2], Daniela Cavagnino[1]

[1]Thermo Fisher Scientific, Italy

[2]Thermo Fisher Scientific, UK

Keywords

Helium Saver, Hydrogen Safer, helium consumption, helium shortage, TRACE 1600 Series GC, iConnect SSL

Scope

The aim of this Technical Note is to illustrate how the Thermo Scientific™ TRACE 1600 Series GC approaches carrier gas conservation challenges through the new Thermo Scientific™ HeSaver-H_2Safer™ technology for iConnect Split/Splitless (SSL) injector.

Introduction

Helium is the most commonly used carrier gas for gas chromatography thanks to its high chromatographic efficiency and inertness. This is particularly important when considering GC-MS, where these characteristics ensure the best performance in terms of sensitivity. This has resulted in well-established, validated, and regulated GC and GC-MS methods using helium as carrier gas.

On the other hand, a conscientious gas chromatographer knows that helium is a limited and non-renewable natural resource.[1] Helium price increases and supply issues caused by shortages led GC manufacturers, researchers, and analysts to investigate possible mitigation options, easily summarized in two practical approaches:

- Switch to alternative carrier gases
- Reduce the consumption of helium

A possible alternative carrier gas in a capillary GC system is hydrogen due to its diffusivity comparable to helium. Hydrogen is even preferable to helium because its linear viscosity is about half, allowing lower inlet pressure for a given gas velocity, and it permits maintaining the optimum separation efficiency at higher flow rates, offering shorter analysis time. Additionally, the availability of hydrogen generators in laboratories offers a safe, steady, and renewable supply of hydrogen.

Therefore, using hydrogen instead of helium is undoubtedly an attractive solution because of its analytical and practical advantages; however, there are still important considerations for the use of hydrogen as carrier gas:

- Safety: being a flammable and explosive gas, it requires the use of a hydrogen sensor in the GC oven, capable of turning off the heating and the gas supply in case of detected leaks.
- Validated methods: many existing validated methods are based on helium and must be re-optimized and re-validated in the case of carrier gas conversion.
- GC-MS: hydrogen is a light and reactive gas. Its use as carrier with mass spectrometer detectors affects the vacuum conditions in the ion source and therefore the ionization process, with consequent decrease of the response.

Despite these shortcomings, many applications are successfully run by using hydrogen, with clear advantages in terms of speed of analysis. However, the possibility to use helium as carrier gas with a significant reduction of its consumption is attractive to mitigate cost and supply issues while maintaining existing validated methods and optimum GC-MS performance.

The Gas Saver mode, available within standard SSL injector control parameters, is a well-established approach to reduce carrier gas consumption through the split line. The split flow is typically reduced to 10–20 mL/min at a specified time after the sample transfer onto the column. However, even using the Gas Saver option, the split flow remains the major source of carrier gas consumption.

The Thermo Scientific™ Helium Saver technology has been exclusively available for many years on the Thermo Scientific™ TRACE™ 1300 Series GC,[2,3] offering an innovative and smart approach to dramatically reduce helium consumption by using a cheaper gas like nitrogen to pressurize the SSL injector and split the sample during the transfer onto the column.

With the introduction of the new TRACE 1600 Series GC, this technology has been further improved:

- Usability: a standard iConnect SSL injector can be easily self-upgraded to HeSaver-H_2Safer functionality.[5] Up to two adapted SSL injectors can be accommodated on the same GC.
- Hydrogen Safer mode: this technology has been extended to the use of hydrogen as carrier, limiting its maximum flow rate and removing the associated safety risks, eliminating the need to install a sensor in the GC oven. The limited maximum flow rate of hydrogen does not affect the maximum permissible split ratio, since the sample split is achieved with an auxiliary gas, as explained in the Principle of operation section.

This technical note illustrates the new HeSaver-H_2Safer technology, how it works, and the benefits delivered to GC users.

Principle of operation

In a split/splitless injector, the carrier gas consumption for the chromatographic separation into a capillary column (0.25–0.32 mm i.d.) is typically 1–2 mL/min, whereas 3–5 mL/min are used for septum purge. Most of the carrier gas is consumed through the split line with flow rates up to hundreds of mL/min depending on the required split ratio. Therefore, in a standard SSL operation, most of the supplied gas is discharged while only 1–2 mL/min are effectively used for chromatographic separation (Figure 1).

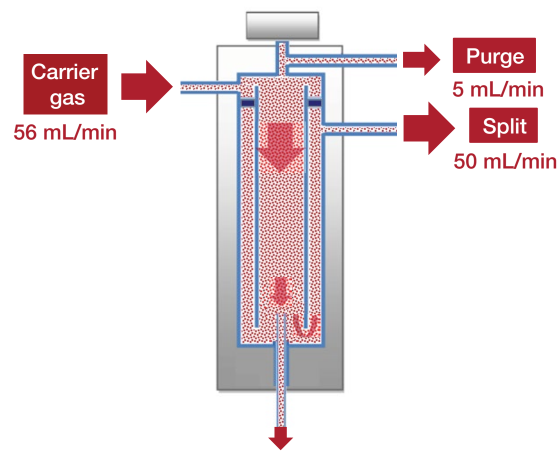


Figure 1. Carrier gas flows through a standard SSL injector

The HeSaver-H_2Safer works by decoupling the gas used for the chromatographic separation from the gas used to pressurize the inlet and maintain split and purge flows. This allows reduction of the carrier gas (helium or hydrogen) total flow to a limited value needed to maintain the gas flow rate for the separation process into the analytical column.

Two operative gas modes are available:

- The **HeSaver mode** ensures a significant reduction of helium consumption during GC operation, relieving helium shortage concerns and reducing running costs.
- The **H_2Safer mode** allows limiting of the maximum hydrogen carrier flow rate for a safer use without the need to install a hydrogen sensor in the GC oven.

The core of the HeSaver-H_2Safer technology is the modified SSL body, now featuring two feeding gas lines:

- The **pressurizing gas line** on top of the inlet (as the carrier gas line in a standard SSL) supplies gas for the required inlet pressure, the septum purge, and split flow. A low-cost inert gas, like nitrogen, is suggested as the pressurizing gas.
- The **carrier gas line** at the base of the injector body delivers the carrier gas to the column— **helium** or **hydrogen,** depending on the configuration and the gas mode selected. A short segment of tubing acts as a back diffusion barrier preventing the pressurizing gas from entering the column during the chromatographic run (Figure 2).

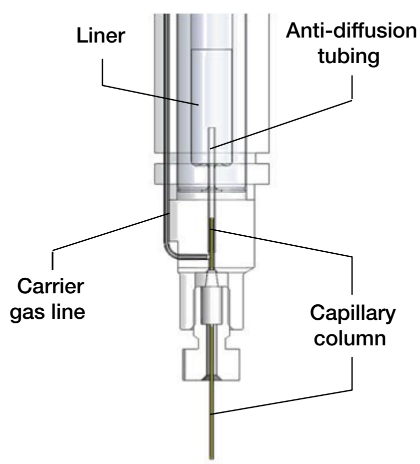


Figure 2. HeSaver-H_2Safer bottom view of the body

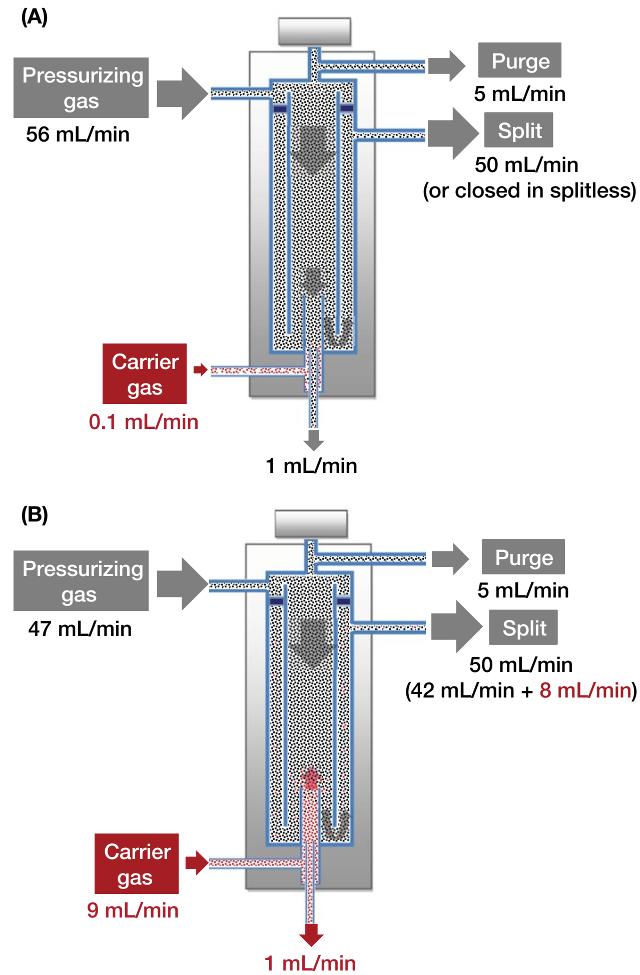


Figure 3. Inlet conditions with the carrier gas flow set at 0.1 mL/min (A) and 9 mL/min (B)

A dedicated valve automatically controls the carrier gas supply by setting two different flows according to inlet conditions:

- Injection phase – This step is achieved with the inlet in the condition represented in Figure 3A. The sample is injected, vaporized, and transferred—in split or splitless mode—by the pressurizing gas flow into the column. The low flow of carrier gas (0.1 mL/min) keeps the carrier gas line free from sample contamination preventing possible carryover.
- Separation process – Once the sample transfer is completed (at the end of the splitless time in splitless mode or after few seconds in split mode), the inlet rapidly switches to the condition represented in Figure 3B. The carrier gas is supplied at higher flow rate (9 mL/min), filling the analytical column and providing a barrier to pressurizing gas diffusion into the column.

Therefore, the chromatographic separation of the analytes is accomplished by the carrier gas at the set flow rate controlled by the inlet pressure (pressurizing gas), ensuring analytical performances equivalent to a standard SSL inlet but with limited consumption of carrier gas.

The transition from the injection phase and the separation process is extremely fast (few milliseconds), ensuring a rapid gas replacement into the column. This was verified by monitoring the pressurizing gas (nitrogen) and the carrier gas (helium) in the column effluent using a mass spectrometer. An acquisition range of 1–50 *m/z* was selected to detect the presence of nitrogen and helium before and after the gas swapping occurred after 1 min of the splitless time. As displayed in Figure 4, the first part of the chromatographic profile shows the intense background of the nitrogen gas reaching the MS, confirmed by the mass spectra showing the molecular ion *m/z* 28. Its duration corresponds to the sum of splitless time (1 min) + column void time (30 m, 0.25 mm, 0.25 µm). The end of the nitrogen plug is represented by the sharp drop of the baseline, corresponding to replacement of nitrogen with helium now reaching the MS. The mass spectrum confirms that no diffusion of nitrogen happens during the separation phase, when only helium is supplied to the column with no interferences from the pressuring gas. This process is rapid, efficient, and very repeatable, as shown in Figure 5 with the baseline profile overlay of three replicates.

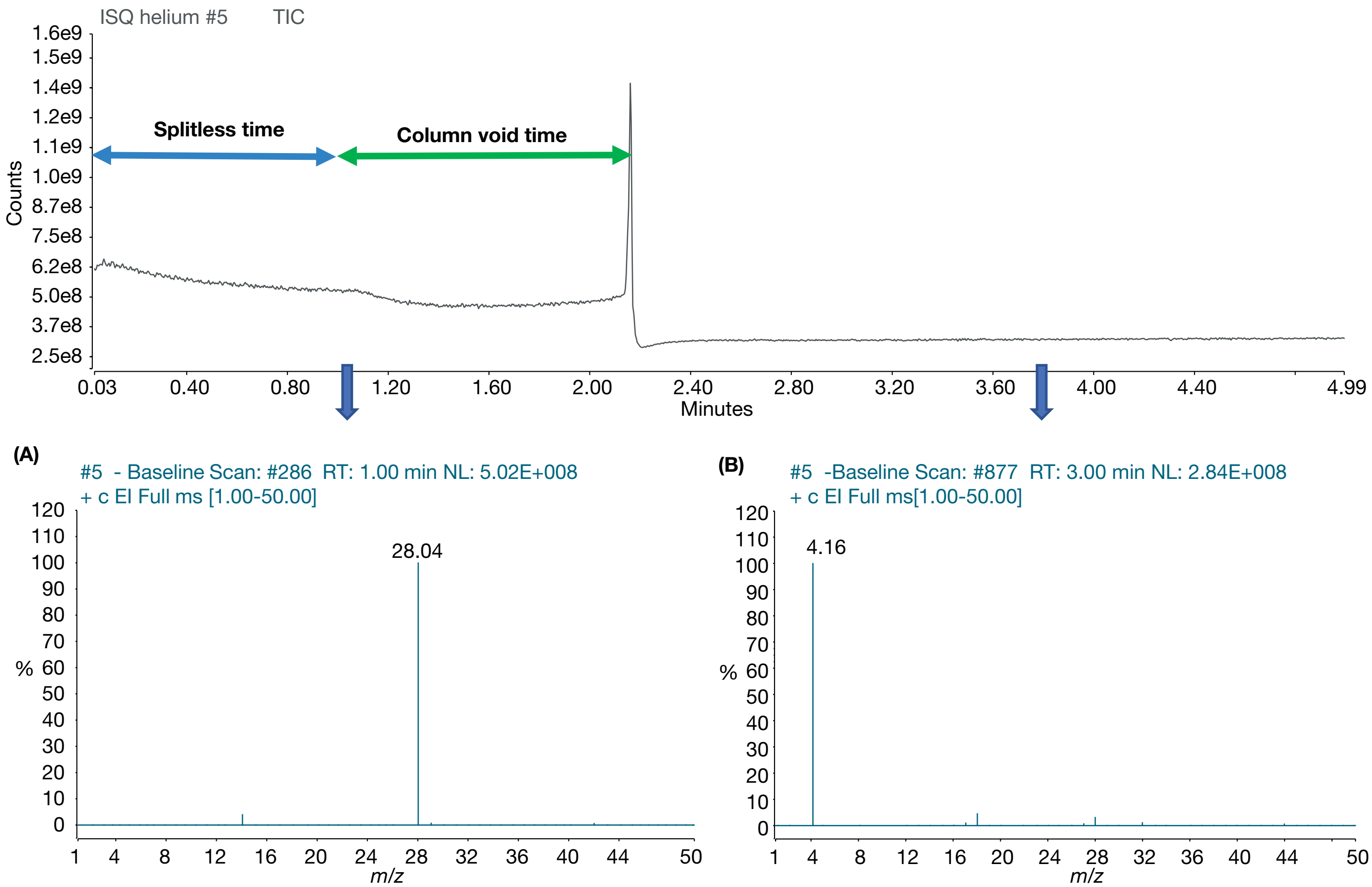


Figure 4. MS baseline profile in the HeSaver mode with nitrogen as pressurizing gas. The mass signal before swapping the gas shows nitrogen ion *m/z* 28 (A) and the mass signal after the gas swapping shows a typical clean MS background in helium (B) with no diffusion of nitrogen during separation.

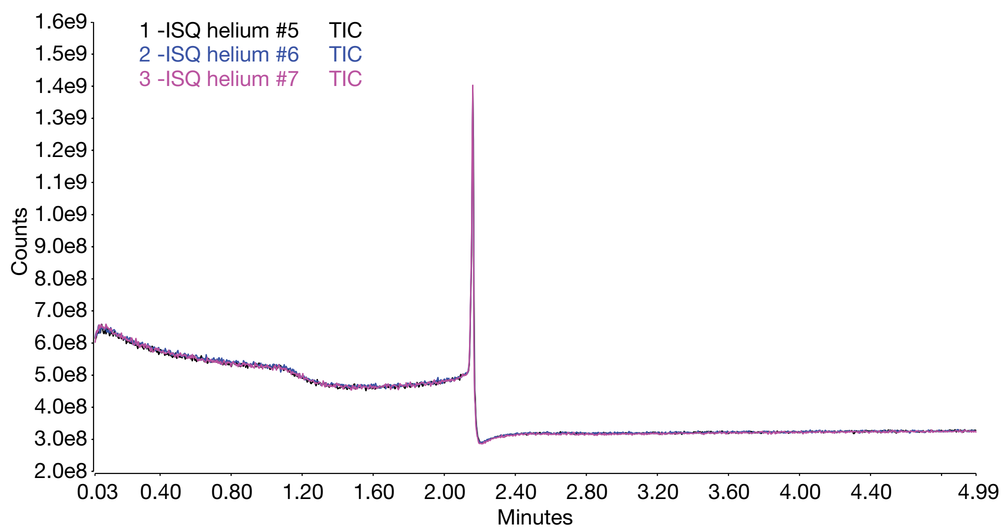


Figure 5. Three overlaid replicates of MS baseline profile in HeSaver mode with nitrogen as pressurizing gas

Benefits of working in HeSaver mode

When using helium as the carrier gas, the HeSaver-H_2Safer technology offers a significant gas savings not only when the GC is idle but also during sample injection and analysis. This leads to an extended helium cylinder lifetime from months to years, depending on the instrument method and usage and how many GCs are served.

An example of the gas savings calculation is reported in Table 2, based on typical GC method settings as listed in Table 1.

Considering a helium cylinder volume of 50 L at 200 bar serving one GC instrument running the method reported in Table 1 on a 24/7 basis, the bottle lifetime is extended about 3.5 times with around-the-clock use, reducing costs and relieving helium shortage issues like delayed deliveries. The higher the split ratio used in the GC method, the higher will be the impact on gas saving.

Table 1. Example of GC method setting considered for gas saving calculation

Parameter	Value
Carrier gas	Helium at 1 mL/min
Split flow	60 mL/min (split ratio 60:1)
Gas Saver flow (reduced split)	20 mL/min
Gas Saver on	3 min
Purge Flow	5 mL/min
Run cycle time	25 min
Runs /day (24 h operation)	57
Helium volume in a cylinder	10,000 L (at atmospheric pressure)

Table 2. Helium gas savings based on GC method settings in Table 1

	Helium consumption with standard SSL	Helium consumption with HeSaver SSL	Gas savings
Daily He usage	44.0 L	12.5 L	-72%
He cylinder life	~7.5 months	~2 years	

The carrier gas savings will be even higher if the system is not operated 24/7. When the system is idle, it can be set to the low helium flow level with almost no consumption, still maintaining the column flushed with the pressurizing gas.

Benefits of working in H_2Safer mode

When hydrogen is used as carrier gas, the HeSaver-H_2Safer technology ensures safe operating conditions.

In a standard SSL, hydrogen is supplied at high pressure with hundreds of mL/min. In case of column breakage or leaking at the inlet connection, the oven is rapidly filled by the explosive gas; the hydrogen sensor is then a mandatory safety requirement to use hydrogen as carrier with a standard SSL.

The H_2Safer mode works by limiting the maximum flow supplied to the inlet and removing any safety concerns, even in case of leaks. In addition, since the inlet is supplied with a much higher pressure of inert pressurizing gas, in case of leaks the pressurizing gas dilutes the hydrogen and helps to purge it out of the GC oven.

Combining the limited hydrogen flow and the dilution with inert gas, it becomes impossible to reach a hazardous concentration of hydrogen in the GC oven.

Another key benefit is offered as a consequence of the limited and constant consumption of hydrogen with the H_2Safer SSL: in case hydrogen generators are used in the laboratory, the limited consumption allows for the supply of more GC systems with a single hydrogen generator. Additionally, the fixed hydrogen flow rate, no longer linked to the method settings, makes the choice of the suitable gas generator capacity much easier.

Smart inlet design for additional advantages

The HeSaver-H_2Safer concept of decoupling the inlet pressurizing gas and the carrier gas provides additional analytical benefits:

- Saves time for inlet maintenance: the modified SSL injector can be opened by simply closing the pressurizing gas and maintaining the carrier gas flowing into the column, removing the need of cooling the GC oven.
- Reduces contamination from the injector into the column: the pressuring gas flushing the liner and the injector is discharged only through the split line for most of the time, entering the column just for the limited time of the injection phase and limiting the transfer of possible contaminants (septum/sample matrix/by-products).
- Removes solvent peak tail in splitless mode: the back flow of the carrier gas preventing the diffusion of the pressurizing gas into the column is effective to remove residual vapors of solvent after splitless injection, improving separation and integration of early eluting compounds typically detected on the solvent tail.
- Ensures a highly inert environment in a hot injector for samples when using hydrogen as carrier gas: when working in the H_2Safer mode, the injection is made in an inert gas, removing any possible reactions between the sample/solvent and hydrogen at high temperature.

As in the standard iConnect SSL injector, the HeSaver-H_2Safer SSL modified body can be easily extracted if deeper cleaning is required. A step-wise video instruction[6] is available through the Thermo Scientific™ TRACE™ 1610 GC touch screen user interface.

Maintained analytical performances

A TRACE 1610 GC configured with an iConnect SSL injector (upgraded to work in HeSaver-H_2Safer modes), an iConnect FID detector, and equipped with a Thermo Scientific™ AI 1610 liquid autosampler, was used to verify the injection performance in the HeSaver-H_2Safer modes in terms of precision and recovery.

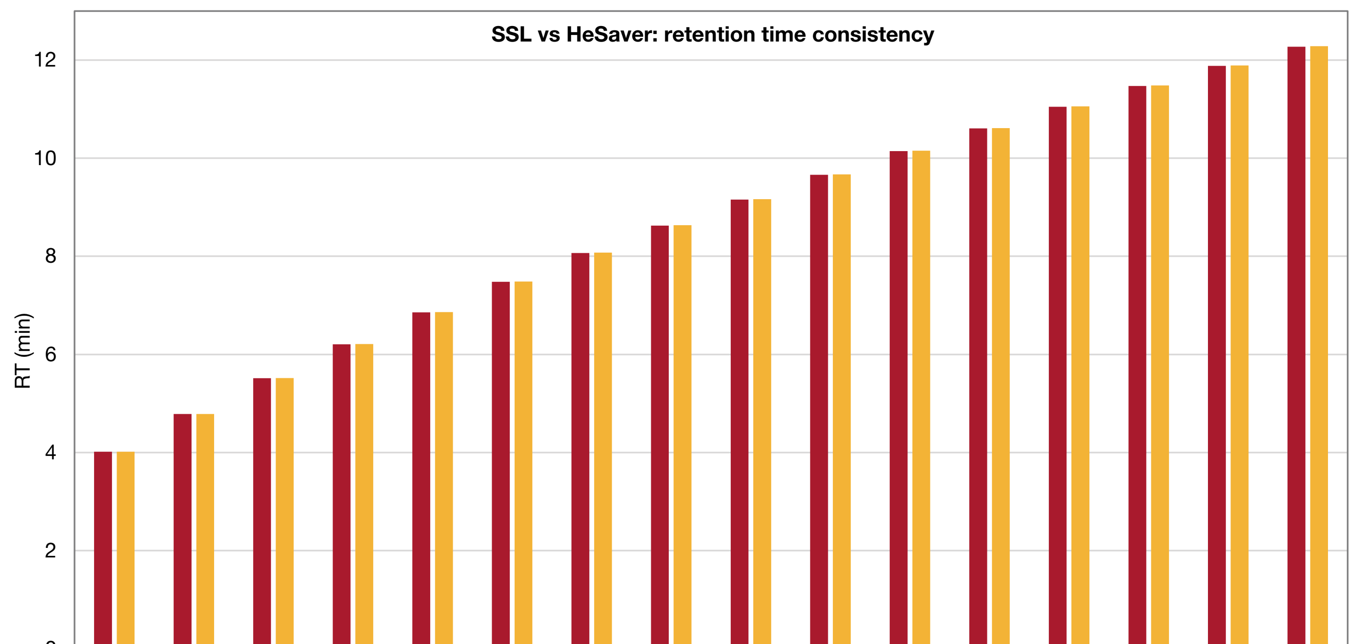
Retention times consistency between standard and HeSaver-H_2Safer SSL

One benefit of the HeSaver-H_2Safer mode is that methods developed on a standard SSL can be maintained since carrier gas flow rate and retention times remain unchanged. A standard solution of n-alkanes C_{10}–C_{25} approximatively at 10 ng/µL in hexane was used to compare retention times by injecting with a standard SSL and a HeSaver-H_2Safer SSL working with nitrogen as pressurizing gas. Both helium and hydrogen were used as carrier gas, in the conditions listed in Table 3.

The plot in Figure 6 reports the retention times comparison determined by injecting the hydrocarbon mixture in the standard SSL using helium as carrier gas and in the HeSaver-H_2Safer SSL working in HeSaver mode (nitrogen as pressurizing gas). The same comparison is shown in Figure 7 between the standard SSL with hydrogen as carrier gas and HeSaver-H_2Safer SSL in H_2Safer mode (nitrogen as pressurizing gas).

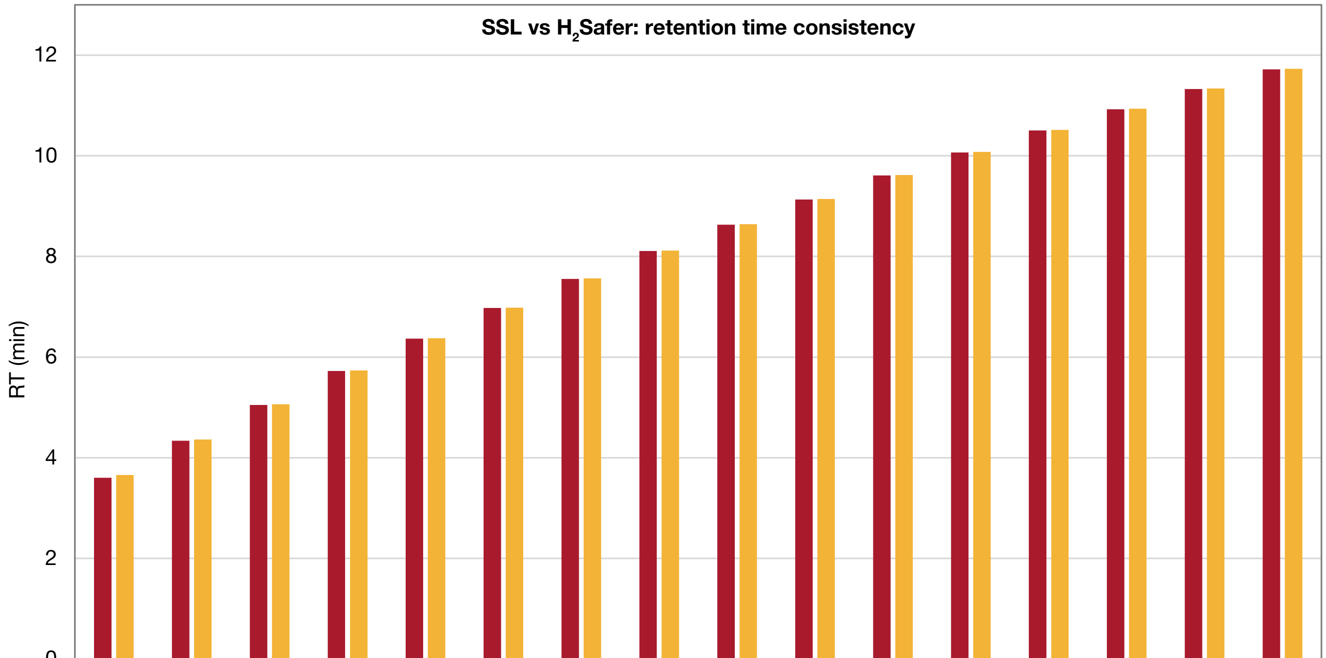
Table 3. Operating GC parameters

Parameter	Value
GC oven	
Column	Thermo Scientific™ TraceGOLD™ 15 m, 0.25 mm, 0.25 µm (P/N 26098-1300)
Temperature program	40 °C (0.5 min), 20 °C/min, 300 °C (5 min)
iConnect SSL	
Helium carrier gas flow rate	1 mL/min
Hydrogen carrier gas flow rate	1.25 mL/min
SSL temperature	300 °C
Injection mode	Splitless
Injection volume	1 µL
Liner	Splitless Single tapered with glass wool (P/N 453A1925)
Splitless time	0.8 min
iConnect FID	
FID temperature	300 °C
H_2 flow	35 mL/min
Air flow	350 mL/min
Make up (N_2) flow	40 mL/min
Acquisition rate	10 Hz



	C10	C11	C12	C13	C14	C15	C16	C17	C18	C19	C20	C21	C22	C23	C24	C25
SSL He (min)	4.016	4.784	5.516	6.207	6.859	7.479	8.066	8.624	9.155	9.66	10.143	10.606	11.048	11.473	11.881	12.273
HeSaver (min)	4.016	4.784	5.518	6.211	6.865	7.485	8.073	8.632	9.163	9.668	10.152	10.614	11.057	11.483	11.891	12.283
Δ min	0	0	0.002	0.004	0.006	0.006	0.007	0.008	0.008	0.008	0.009	0.008	0.009	0.010	0.010	0.010

Figure 6. Retention time consistency using helium as carrier gas, between standard SSL and HeSaver SSL. 1 µL splitless injection of a C_{10}–C_{25} hydrocarbon mixture at 10 ng/µL



	C10	C11	C12	C13	C14	C15	C16	C17	C18	C19	C20	C21	C22	C23	C24	C25
SSL H_2 (min)	3.604	4.339	5.05	5.727	6.369	6.977	7.556	8.107	8.631	9.131	9.608	10.066	10.503	10.924	11.327	11.717
H_2Safer (min)	3.659	4.364	5.064	5.736	6.376	6.985	7.566	8.117	8.641	9.141	9.618	10.076	10.514	10.935	11.339	11.728
Δ min	0.055	0.025	0.014	0.009	0.007	0.008	0.010	0.010	0.010	0.010	0.010	0.010	0.011	0.011	0.012	0.011

Figure 7. Retention time consistency using hydrogen as carrier gas, between standard SSL and H_2Safer SSL. 1 µL splitless injection of a C_{10}–C_{25} hydrocarbon mixture at 10 ng/µL

Repeatability

Peak area and retention time (RT) repeatability were tested in both HeSaver and H_2Safer mode using nitrogen as pressurizing gas. Both splitless and split injections were evaluated over 10 injections of the C_{10}–C_{25} normal alkanes solution in hexane (approximatively 10 ng/µL each for splitless injection and 50 ng/µL each for split injection). The results reported in Tables 4 and 5 confirm the expected repeatability comparable with a standard SSL.

Table 4. Standard deviation (SD) of retention times and repeatability (%RSD) of absolute peak areas in HeSaver mode (n=10)

	HeSaver			
	Splitless		Split	
	RT SD (min)	Absolute peak area RSD (%)	RT SD (min)	Absolute peak area RSD (%)
C10	0.001	0.74	0.001	0.73
C11	0.000	0.71	0.001	0.69
C12	0.000	0.70	0.001	0.70
C13	0.001	0.67	0.001	0.64
C14	0.001	0.67	0.001	0.71
C15	0.001	0.74	0.002	0.64
C16	0.001	0.78	0.001	0.65
C17	0.001	0.64	0.001	0.61
C18	0.001	0.87	0.001	0.67
C19	0.001	0.58	0.001	0.62
C20	0.001	0.63	0.001	0.64
C21	0.001	0.67	0.001	0.66
C22	0.001	0.61	0.001	0.65
C23	0.001	0.58	0.001	0.68
C24	0.001	0.60	0.001	0.71
C25	0.001	0.67	0.001	0.74

Table 5. Standard deviation (SD) of retention times and repeatability (%RSD) of absolute peak areas in H_2Safer mode (n=10)

	H_2Safer			
	Splitless		Split	
	RT SD (min)	Absolute peak area RSD (%)	RT SD (min)	Absolute peak area RSD (%)
C10	0.001	0.78	0.001	0.52
C11	0.001	0.77	0.001	0.50
C12	0.001	0.71	0.001	0.47
C13	0.001	0.81	0.001	0.48
C14	0.001	0.76	0.001	0.42
C15	0.001	0.82	0.001	0.45
C16	0.001	0.67	0.001	0.39
C17	0.001	0.69	0.001	0.39
C18	0.001	0.86	0.001	0.47
C19	0.001	0.71	0.001	0.44
C20	0.001	0.80	0.001	0.40
C21	0.001	0.72	0.001	0.42
C22	0.001	0.67	0.001	0.47
C23	0.001	0.66	0.001	0.46
C24	0.001	0.70	0.001	0.53
C25	0.001	0.85	0.001	0.75

Recovery

High-boiling compound recovery was tested in both HeSaver and H_2Safer modes by injecting a standard solution of n-alkanes C_{10}–C_{40} approximatively at 6 ng/µL each in isooctane.

As reported in Table 6 and Figures 8–9, the behavior of the HeSaver-H_2Safer mode in terms of recovery of high boiling compounds is excellent, with a C_{40}/C_{20} ratio > 0.9.

Table 6. Recovery (%) C_x/C_{20} for the HeSaver-H_2Safer SSL, with helium and hydrogen as carrier gas. Values are averaged on 8 replicates.

	HeSaver		H_2Safer	
	Area (pA*min)	Recovery C_x/C_{20} (%)	Area (pA*min)	Recovery C_x/C_{20} (%)
C10	3.138	97.2	3.113	96.2
C12	3.148	97.5	3.154	97.5
C14	3.170	98.2	3.180	98.2
C16	3.208	99.4	3.192	98.6
C18	3.306	102.4	3.323	102.7
C20	3.228	100.0	3.237	100.0
C22	3.212	99.5	3.227	99.7
C24	3.262	101.0	3.250	100.4
C26	3.255	100.9	3.256	100.6
C28	3.262	101.1	3.254	100.6
C30	3.231	100.1	3.227	99.7
C32	3.199	99.1	3.179	98.2
C34	3.153	97.7	3.139	97.0
C36	3.032	93.9	2.985	92.2
C38	2.928	90.7	2.921	90.2
C40	2.965	91.9	2.941	90.9

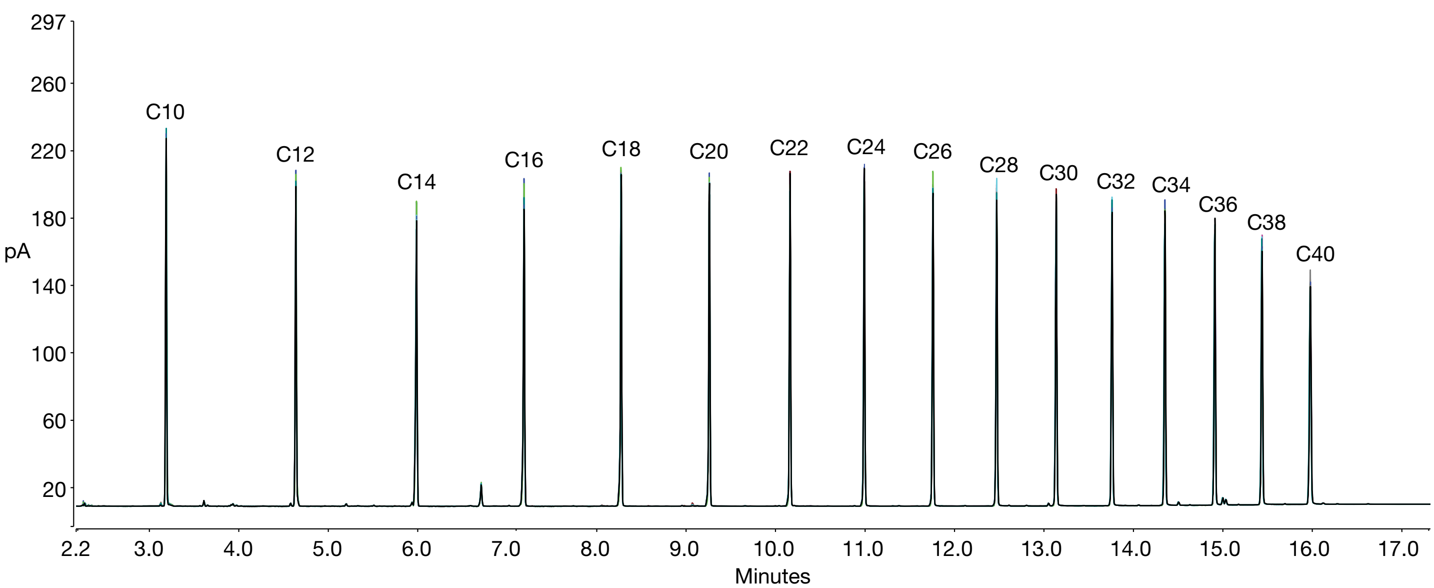


Figure 8. Overlaid chromatograms of hydrocarbon mixture C_{10}–C_{40} (n=8) with the SSL HeSaver mode, showing the recovery up to C_{40}

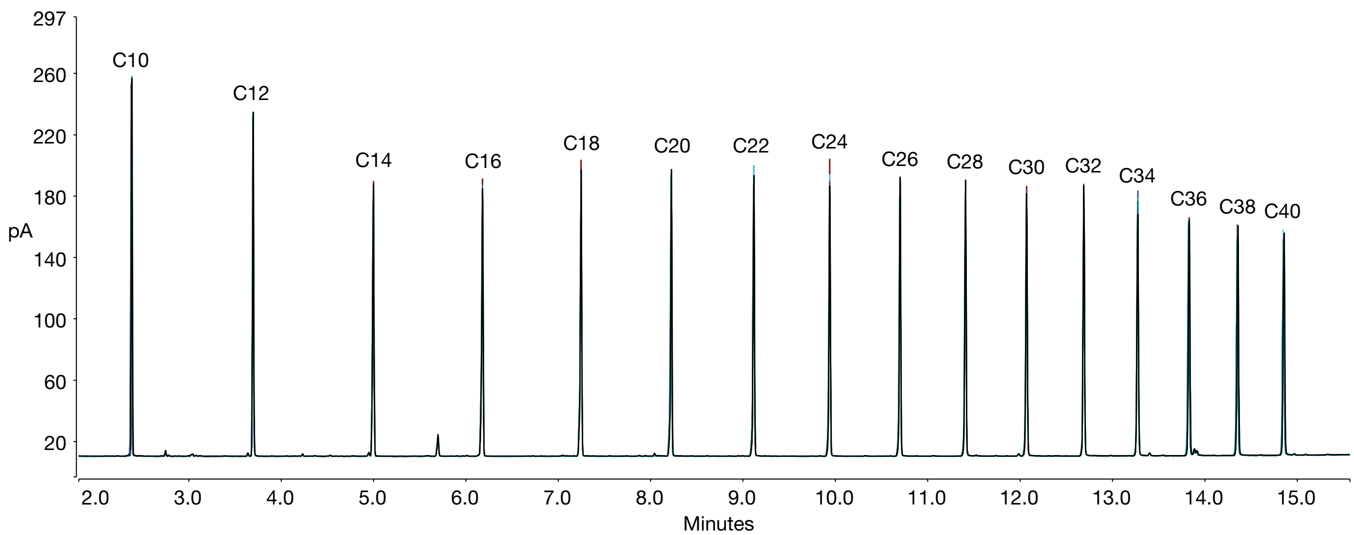


Figure 9. Overlaid chromatograms of hydrocarbon mixture C_{10}–C_{40} (n=8) with the SSL H_2Safer mode, showing the recovery up to C_{40}

Conclusion

HeSaver-H_2Safer technology is a simple, user-installable option for carrier gas saving to be used with the iConnect SSL injector module on the TRACE 1600 Series GC. Whether using helium or hydrogen as carrier gas, HeSaver-H_2Safer technology reduces the consumption by limiting the total flow rate of the carrier gas to what is necessary to feed the column and carry on the chromatographic separation.

- For helium users, this means significantly extended cylinder usage, relieving helium shortage concerns and reducing cost of operations.
- For hydrogen users, this means working in safer conditions, minimizing possible reactions between the sample and hydrogen in a hot SSL injector, and serving more GC systems in case of hydrogen generators in the laboratory.
- In both cases, the HeSaver-H_2Safer technology offers additional advantages, such as maintaining the column flow during SSL maintenance, protecting the column from possible contaminants, and removing the solvent peak tailing in splitless injection with better separation of early eluting compounds.
- Existing validated methods can be used unchanged, with consistent analytical performance in terms of injection repeatability and recovery.

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