An Innovative Injector Allows Helium Carrier Gas Conservation in Analytical Gas Chromatography

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Overview

Purpose: We introduce a new, innovative injector which allows conservation of helium carrier gas. While preserving the analytical GC column flow with helium, it maintains the septum purge and the split flows with another inert gas like nitrogen - even during the analytical run. The analytical performances of the injector are in line with those of the common split/splitless injector module.

Introduction

The helium supply chain crisis has negative implications on research and laboratory operations world-wide. Helium rationing, delayed deliveries and price increases still cause difficulty in production planning and uncertainty in instrument productive uptime.

Although the GC & GC-MS segment consumes less than one percent of the global helium supply usage per year, the shortages and delivery interruptions have wide-spread consequences for many industries utilizing varied analytical techniques.

There are different way to tackle this problem, one is reducing the split ratio during the split time of the injection or switch to another carrier gas such as hydrogen or switch gas only when the GC or GC/MS are not running.

All of these solutions present some downsides: reducing the split flow is not suitable for splitless injections and can affect accuracy for smaller volume injections, hydrogen presents some safety concerns and require a translation and optimization of the methods in use, the switching of gas when the instrument is not running is not applicable to routine labs and requires some time to switch back to helium before resuming operations.

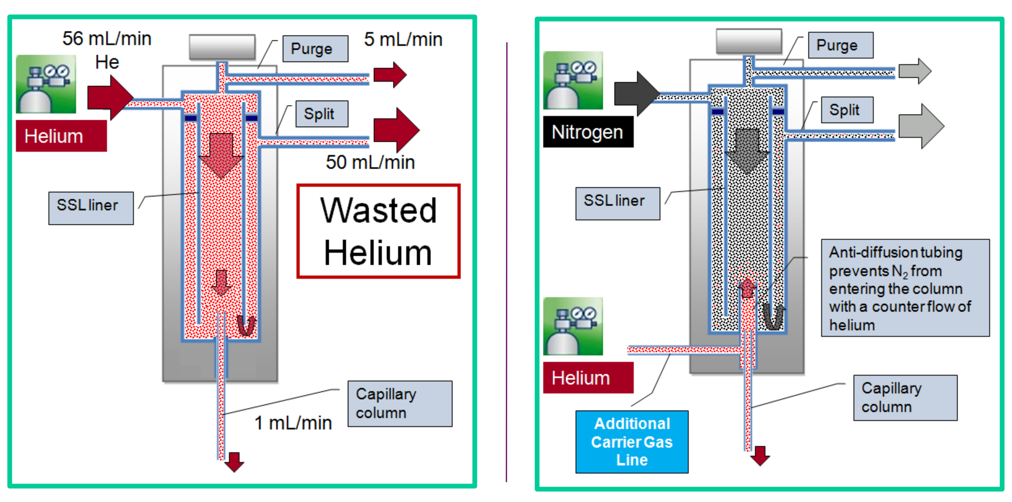
The Thermo Scientific™ Instant Connect Helium Saver Injector Module allows to massively reduce the helium consumption while working as an usual Split Splitless injector. The Helium saver module benefits from the modularity concept of all of the injectors and detectors for the Thermo Scientific™ TRACE™ 1300 Series GC and can be quickly installed and operative maximizing instrument uptime. The Helium Saver module can be mounted also on pre-existing TRACE 1300 Series GC systems without modifications.

FIGURE 1. TRACE 1300 Series GC Modularity with user-installable modules

Until now, split/splitless injectors use one single gas as carrier, for septum purge and sample split. Usually, only ~1/10th - 1/50th of the total gas flow enters the column. Purge and split flows cannot be reduced beyond a certain limit without introducing contamination into column/detector due to sample matrix and septa particles accumulated in liner and lines or without air diffusing from septa seals.

The Helium Saver injector uses two different gases: nitrogen is used for septum purge and sample split flows while helium only feeds the analytical column, so that in this way its consumption is drastically reduced.

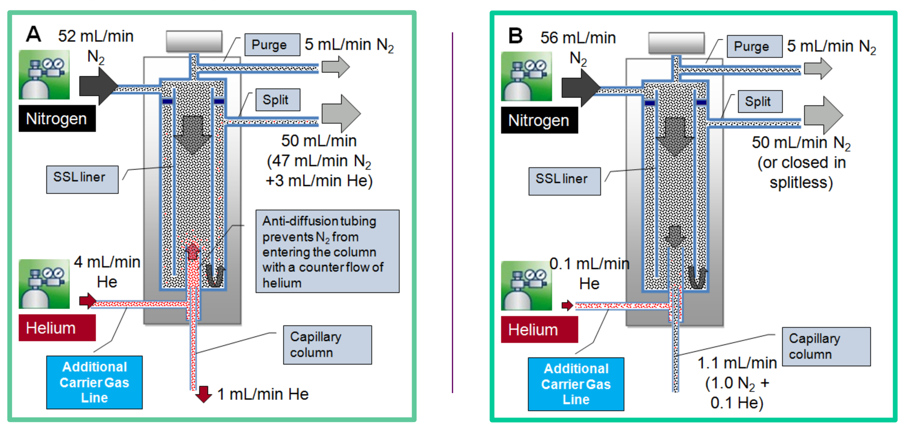
FIGURE 2. Differences between the standard SSL injector and the Helium Saver injector.



How the Helium Saver Injector Works

During all "non-injection" periods, helium is supplied with a flow slightly higher than the column flow. Helium consumption is drastically reduced compared to a standard Split/Splitless injector (Figure 3A).

FIGURE 3. Helium flow during non-injection (A) and injection phase (B).



During the injection period, nitrogen flows into the column for sample introduction, and helium is supplied at 0.1 mL/min to keep the connection swept and avoid dead volumes (Figure 3B).

When the GC is in stand-by, it can be left in this condition with zero consumption of helium. Separation is always done using helium carrier gas.

Benefits of the Helium Saver Injector

The Helium Saver injector is able to significantly increase the duration of helium gas tanks. A standard gas tank can last up to 3.5 years when continuously used 24/7 all 365 days of the year for GC-MS analysis and up to 14.6 years shutting the helium off or switching to N_2 on weekends and overnight. This means that, potentially, this could be the last and only helium cylinder that will be needed for the instrument lifetime.

The Instant Connect Helium Saver module is operated exactly like an SSL injector allowing a pain free translation of any methods that involves a Split/Splitless type injection.

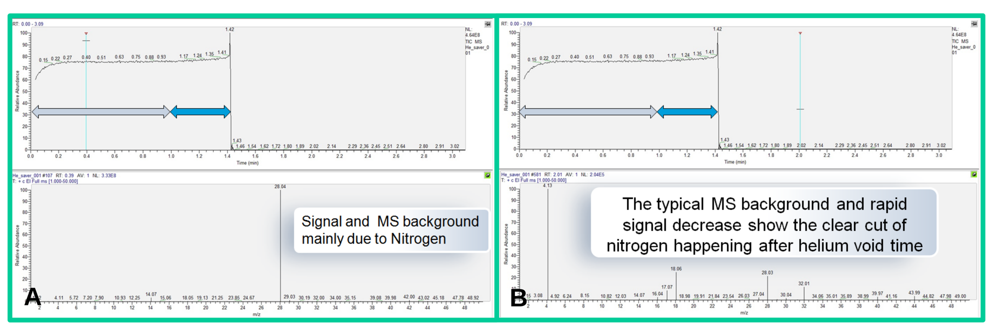
As an example, we chose the GC-MS analysis of semi-volatiles compounds performed following the guidelines of **U.S. EPA Method 8270.**

The method dictates the employ of a carrier flow of 1 mL/min with a split flow of 60 mL/min, the gas saver is turned on after 3 minutes and reduces the split flow to 20 mL/min. The GC run time is 25 minutes, and we can estimate for a routine lab, a total of 57 analyses per day for round the clock operations. The daily helium consumption calculated would be 45.56 liters, considering a helium tank with a capacity of 7300 liters, this would guarantee a tank duration of 157 days for continuous uninterrupted use. The helium consumption using the Helium Saver injector would be reduced to 5.76 liters per day, extending the tank duration to 1267 days with a significant impact on savings.

Helium Saver Module Performances

The main concern users may have about the injector may be whether its double gas configuration may negatively influence the analysis or its performances may differ from those of the regular Split/Splitless injector. The switch between the two gases is rapid and efficient, due to the minimum volume of the injector area (Figure 3).

FIGURE 4. Nitrogen/Helium Switch. Splitless injection, 1 min. Chromatogram on top.



A.) During the injection phase, only nitrogen flows into the column. The chromatographic signal (see top) and the MS spectrum reflect the large amount of nitrogen reaching the MS Quadrupole.

B.) After the 1-minute splitless, helium starts flowing into the column. We can still observe a high nitrogen signal for another 30 seconds or so. This corresponds to the column void time. Immediately after helium reaches the end of the column and the MS detector, the chromatographic signal drops and the MS spectrum of the baseline is the typical background observed in MS with helium carrier gas. The proper water-nitrogen ratio in the spectrum and the sharp signal drop clearly show how quickly the system switches from nitrogen to helium.

The injector module performance matches those of the regular SSL injector. The chromatograms obtained by this module are comparable to those obtained with SSL module in both areas and retention times.

FIGURE 5. Hydrocarbon mix chromatogram.

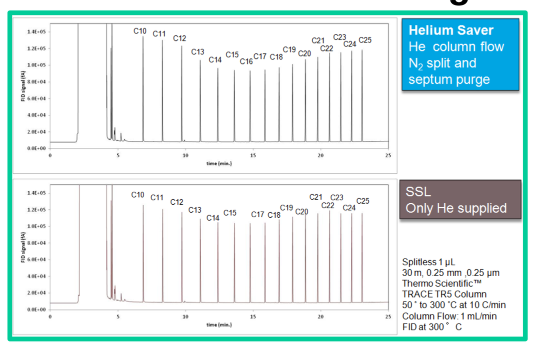


FIGURE 6. Areas and retention time comparison between SSL and Helium Saver Module.

Component	Absolute peak area (counts)			Retention times (min.)			
	Helium only	Helium Saver (N2 + He)	Diff%	Helium only	Helium Saver (N2 + He)	Diff (min.)	Diff%
nC10	1509238	1643138	+8.9	6.878	6.862	-0.017	-0.24
nC11	1501697	1631649	+8.7	8.312	8.297	-0.015	-0.18
nC12	1525277	1634623	+7.2	9.733	9.720	-0.013	-0.14
nC13	1539736	1643560	+6.7	11.100	11.092	-0.008	-0.08
nC14	1528093	1626721	+6.5	12.393	12.387	-0.007	-0.05
nC15	1535544	1631809	+6.3	13.618	13.612	-0.007	-0.05
nC16	1555112	1644967	+5.8	14.778	14.773	-0.005	-0.03
nC17	1564216	1652088	+5.6	15.877	15.873	-0.003	-0.02
nC18	1566094	1652903	+5.5	16.922	16.918	-0.003	-0.02
nC19	1573423	1658194	+5.4	17.917	17.913	-0.003	-0.02
nC20	1588978	1677003	+5.5	18.867	18.865	-0.002	-0.01
nC21	1584344	1669784	+5.4	19.773	19.772	-0.002	-0.01
nC22	1585427	1668266	+5.2	20.643	20.640	-0.003	-0.02
nC23	1582252	1661415	+5.0	21.477	21.475	-0.002	-0.01
nC24	1588109	1665599	+4.9	22.278	22.277	-0.002	-0.01
nC25	1568167	1640702	+4.6	23.047	23.047	0.000	0.00

No changes on RT (few 1/1000 of a minute) nor on peak areas

Repeatability has been tested as well, both in split and splitless mode along with the linearity of the split ratio. The results confirm the excellent analytical performance of the module

FIGURE 7. Area and retention times repeatability for splitless mode.

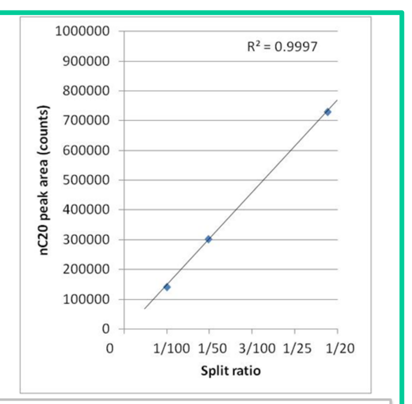
Component	Absolute peak area		Retention times		
	Average (counts)	RSD%	Average (min.)	SD (min.)	RSD%
nC10	1675299	0.48	6.863	0.001	0.01
nC11	1660541	0.59	8.299	0.001	0.01
nC12	1674407	0.66	9.722	0.001	0.01
nC13	1679538	0.53	11.092	0.001	0.01
nC14	1662749	0.54	12.389	0.001	0.01
nC15	1663737	0.71	13.615	0.001	0.01
nC16	1672853	0.48	14.775	0.001	0.01
nC17	1679632	0.50	15.875	0.001	0.01
nC18	1679468	0.45	16.920	0.002	0.01
nC19	1685406	0.50	17.916	0.002	0.01
nC20	1702906	0.54	18.866	0.002	0.01
nC21	1697257	0.56	19.774	0.002	0.01
nC22	1695464	0.56	20.644	0.001	0.01
nC23	1689021	0.61	21.478	0.001	0.01
nC24	1696156	0.65	22.279	0.001	0.01
nC25	1671095	0.64	23.049	0.001	0.00

RSD% below 1% on absolute peak areas and SD in the range of 1/1000 of a minute on retention time

Precision is not affected by the Helium Saver Module

FIGURE 8. Area and retention times repeatability for split mode, and split ratio linearity.

Component	Absolute peak area*	
	Average (counts)	RSD%
nC10	739872	1.01
nC11	732835	0.81
nC12	736868	0.96
nC13	737309	0.97
nC14	730281	0.87
nC15	729157	0.99
nC16	733663	0.84
nC17	732963	0.94
nC18	731400	0.89
nC19	727888	0.76
nC20	732675	0.78
nC21	728136	0.86
nC22	724306	0.70
nC23	717839	0.72
nC24	711406	0.70
nC25	693043	0.77

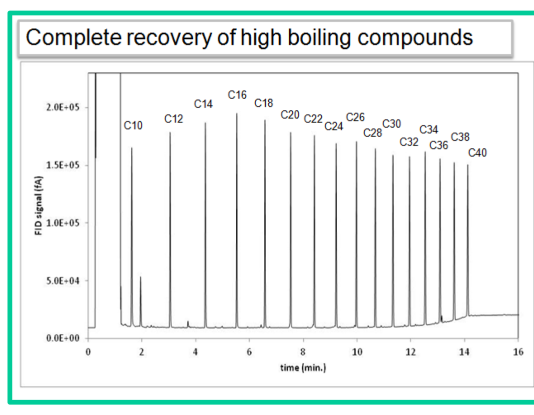


RSD% around 1%. Good linearity of split ratio.
Precision and split ratio accuracy is not affected by the Helium Saver mode

The injector module has been tested for high boiling compounds discrimination and activity towards labile compounds, always with excellent results.

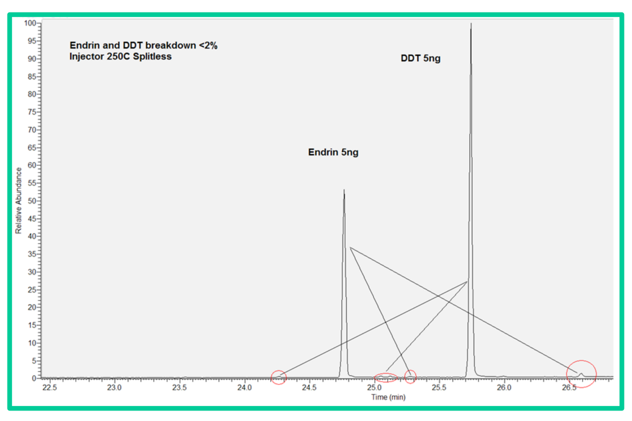
FIGURE 9. Discrimination results on a C10-C40 hydrocarbon mix.

Complete recovery of high boiling compounds



Component	Absolute Peak Areas (counts)	Recovery (%) *
nC10	2498258	100.3
nC12	2457224	98.7
nC14	2550661	102.4
nC16	2504229	100.6
nC18	2484007	99.8
nC20	2489876	100.0
nC22	2482628	99.7
nC24	2455417	98.6
nC26	2409871	96.8
nC28	2438442	97.9
nC30	2469831	99.2
nC32	2412554	96.9
nC34	2472760	99.3
nC36	2422105	97.3
nC38	2394975	96.2
nC40	2349780	94.4

FIGURE 10. Endrin and DDT breakdown.



Conclusion

The Instant Connect Helium Saver Module is an innovative and exclusively featured injector whose revolutionary concept benefits the user with significant and continuous helium gas savings without influencing the operator routine. It also guarantees analytical performances that are comparable to those of the most widespread Split/Splitless injector. The injector is compatible with all the TRACE 1300 Series GC systems and doesn't require the intervention of a service engineer for its installation allowing immediate savings and productivity.

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