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Environmental

Reducing the helium consumption without compromising performance using the HeSaver-H_2Safer technology for PCBs analysis

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Introduction

Polychlorinated biphenyls (PCBs) were used in hundreds of industrial and commercial applications due to their chemical and physical properties, but their production was banned because of their persistence in the environment and their tendency to enter food chains and bioaccumulate in living organisms. Out of 209 known congeners, the 12 most toxic ones (DL-PCBs or dioxin-like PCBs) have been regulated under the Stockholm Convention for POPs since 2001 because of their high similarity to dioxins.

Helium is the most commonly used carrier gas for gas chromatography thanks to its high chromatographic efficiency and inertness. Recent price increases in helium and supply issues caused by shortages have led GC manufacturers, researchers, and analysts to investigate possible mitigation options that entail either switching to alternative carrier gases or reducing the helium consumption. The Thermo Scientific™ HeSaver-H_2Safer™ carrier gas saving technology[1] offers an innovative and smart approach to dramatically reduce carrier gas consumption, especially during GC operation. It consists of a modified SSL body connected to two gas lines; an inexpensive gas (e.g., nitrogen or argon) is used for inlet pressurization, analyte vaporization, and transfer to the analytical column, whereas the selected carrier gas (e.g., helium or hydrogen) is used only to supply the chromatographic column for the separation process, with a limited maximum flow rate. When used with helium as carrier gas, the limited consumption allows shortage issues to be mitigated while maintaining GC-MS performances, without the need of instrument method re-optimization otherwise required in case of migration to a different carrier gas.

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A Thermo Scientific™ TRACE™ 1610 gas chromatograph equipped with two Thermo Scientific™ iConnect™ split/splitless injectors, one operating as a standard SSL and the other one upgraded to work in HeSaver-H_2Safer mode, was coupled to the Thermo Scientific™ TSQ™ 9610 triple quadrupole mass spectrometer and used to run a direct comparison to demonstrate consistency in the analytical results. Chromatographic performance, linearity, instrument detection limits (IDLs), and robustness were evaluated. Details of the material and the methods used for sample preparation, as well as complete lists of reagents, consumables, and instrument settings are reported in a previous application note.[2] For this evaluation, the TSQ 9610 triple quadrupole mass spectrometer was operated using an emission current of 10 µA.

Results and discussion

One of the key benefits of the HeSaver-H_2Safer inlet is that method transfer (for methods using the standard SSL injector) was straightforward and there was no need for re-optimization, as the chromatographic performance remained unchanged with retention time (RT) consistency (deviation of ≤0.03 minutes) compared to standard SSL. Efficient transfer of analytes, including the high boiling point PCBs, is demonstrated by the Gaussian peak shape observed in the chromatogram displayed in Figure 1. Accuracy and precision of the HeSaver-H_2Safer inlet was excellent, with an average recovery of >97% for a solvent spike containing 0.2 ng/mL of a series of PCBs (Figure 1).

Compound	Retention time (min)			Recovery (%)	
	SSL	HeSaver inlet	Deviation (min)	SSL	HeSaver inlet
PCB-1	8.44	8.44	0.00	101	101
PCB-3	8.97	8.97	0.00	100	102
PCB-4	9.13	9.14	0.01	101	102
PCB-19	9.81	9.83	0.02	101	101
PCB-15	10.28	10.29	0.01	102	102
PCB-54	10.46	10.47	0.01	102	100
PCB-104	11.26	11.27	0.01	100	100
PCB-37	11.75	11.76	0.01	103	101
PCB-155	12.12	12.14	0.02	100	100
PCB-77	13.30	13.33	0.03	101	100
PCB-81	13.58	13.6	0.02	101	100
PCB-188	13.69	13.71	0.02	100	111
PCB-123	13.86	13.88	0.02	101	101
PCB-118	13.94	13.95	0.01	101	101

Compound	Retention time (min)			Recovery (%)	
	SSL	HeSaver inlet	Deviation (min)	SSL	HeSaver inlet
PCB-114	14.16	14.18	0.02	101	99
PCB-105	14.63	14.65	0.02	100	99
PCB-126	15.54	15.53	0.01	99	97
PCB-202	15.55	15.58	0.03	101	102
PCB-167	15.81	15.83	0.02	98	101
PCB-156	16.50	16.52	0.02	98	100
PCB-157	16.68	16.7	0.02	98	100
PCB-169	17.71	17.73	0.02	98	99
PCB-208	18.03	18.05	0.02	101	101
PCB-189	18.75	18.79	0.03	100	106
PCB-205	19.86	19.89	0.03	102	102
PCB-206	20.43	20.45	0.02	101	98
PCB-209	20.87	20.89	0.02	103	100

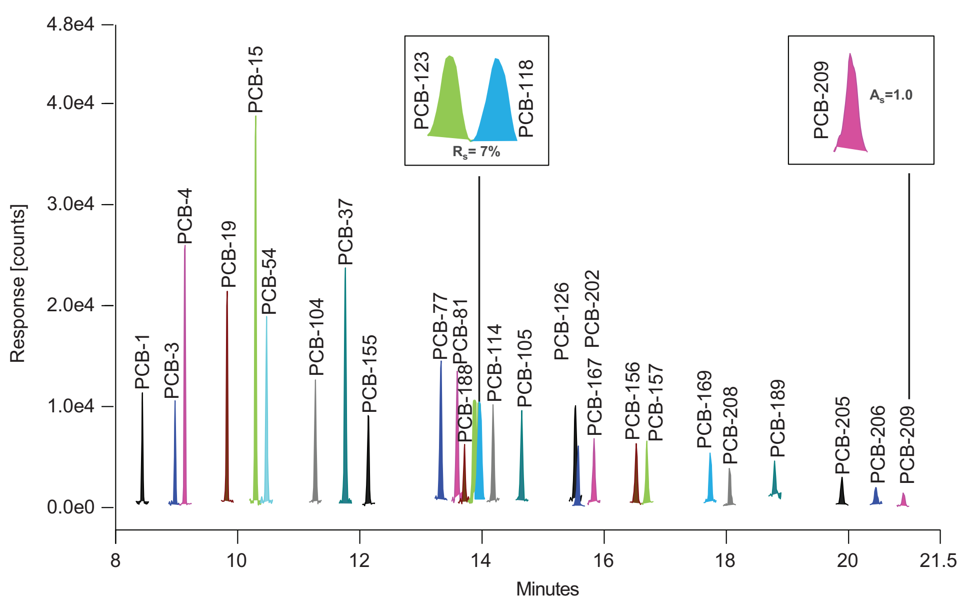


Figure 1. Chromatographic performance achieved with the HeSaver-H_2Safer technology for a solvent standard at 0.2 ng/mL. The insets report the chromatographic resolution (R_s) of the critical pair PCB-123/PCB-118 and the asymmetry factor (A_s) of PCB-209.

A five-point calibration curve ranging from 0.20 to 2,000 ng/mL was used to evaluate linearity and IDLs. Coefficient of determination (R^2), residual values (measured as % RSD of average response factors, AvCF %RSD), calculated IDLs, and absolute peak area repeatability for n=10 injections at the lowest calibration point showed similar results when compared to standard SSL injector operations (Table 1).

The HeSaver-H_2Safer concept of decoupling the inlet pressurizing gas and the carrier gas 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 therefore limiting the transfer of contaminants (septum/sample matrix/by-products). Robustness was evaluated by repeatedly injecting different environmental extracts (n=100) bracketed by quality control standards in nonane (QC) at a concentration of 1.0 ng/mL. The QCs showed stable and consistent responses in comparison to the SSL injector with absolute peak area %RSD < 10% (Figure 2A). No inlet or MS maintenance and no re-tuning was required during the whole sequence despite the injection of dirty matrix samples (Figure 2B).

Table 1. Calculated coefficient of determination (R^2), residual values (measured as % RSD of average response factors, AvCF %RSD), IDLs, and absolute peak area %RSDs at the lowest calibration point obtained using the HeSaver-H_2Safer technology and the standard SSL injector

Compound	Coefficient of determination (R^2)		AvCF %RSD		Calculated IDLs (0.2 ng/mL)		Peak area %RSD (0.2 ng/mL, n=10)	
	SSL	HeSaver inlet	SSL	HeSaver inlet	SSL	HeSaver inlet	SSL	HeSaver inlet
PCB-1	0.99999	0.99999	0.8	0.7	0.02	0.03	4.3	4.5
PCB-3	0.99996	0.99999	1.4	0.7	0.02	0.04	3.4	7.1
PCB-4	0.99999	0.99999	0.6	0.6	0.02	0.02	2.7	3.2
PCB-19	1.00000	1.00000	0.3	0.4	0.01	0.02	2.5	2.7
PCB-15	0.99997	0.99997	1.2	1.3	0.01	0.02	2.3	4.1
PCB-54	0.99998	0.99986	1.1	2.5	0.02	0.03	3.4	4.8
PCB-104	0.99992	0.99987	2.0	2.4	0.02	0.02	2.7	3.9
PCB-37	0.99998	1.00000	1.1	0.4	0.01	0.02	2.3	3.7
PCB-155	0.99999	0.99987	0.7	2.4	0.03	0.02	6.0	4.1
PCB-77	0.99996	0.99991	1.4	2.1	0.01	0.03	2.6	5.6
PCB-81	1.00000	1.00000	0.4	0.5	0.02	0.02	3.0	2.9
PCB-188	0.99982	0.99999	2.8	0.8	0.02	0.03	3.1	5.3
PCB-123	0.99999	0.99978	0.6	3.2	0.02	0.03	2.7	5.7
PCB-118	0.99997	1.00000	1.1	0.3	0.04	0.02	7.6	3.2
PCB-114	0.99999	0.99997	0.6	1.3	0.02	0.03	3.2	4.6
PCB-105	0.99997	0.99992	1.2	1.9	0.01	0.02	2.6	3.0
PCB-126	0.99994	0.99988	1.7	2.4	0.01	0.03	2.4	4.7
PCB-202	1.00000	0.99990	0.3	2.1	0.02	0.04	3.9	7.3
PCB-167	0.99996	1.00000	1.4	0.4	0.02	0.03	3.4	5.9
PCB-156	0.99997	0.99999	1.2	0.7	0.01	0.03	2.6	5.1
PCB-157	0.99999	0.99999	0.8	0.6	0.01	0.03	2.4	5.4
PCB-169	0.99999	0.99995	0.6	1.5	0.02	0.05	3.1	9.6
PCB-208	1.00000	0.99999	0.2	0.5	0.04	0.05	6.4	9.4
PCB-189	0.99999	1.00000	0.8	0.4	0.03	0.02	5.9	3.6
PCB-205	0.99998	0.99998	0.9	1.0	0.02	0.04	4.3	7.8
PCB-206	0.99998	0.99999	1.0	0.6	0.05	0.05	8.9	9.3
PCB-209	1.00000	0.99994	0.5	1.7	0.03	0.05	6.0	9.5
Average	**0.99997**	**0.99995**	**1.0**	**1.2**	**0.02**	**0.03**	**3.8**	**5.4**

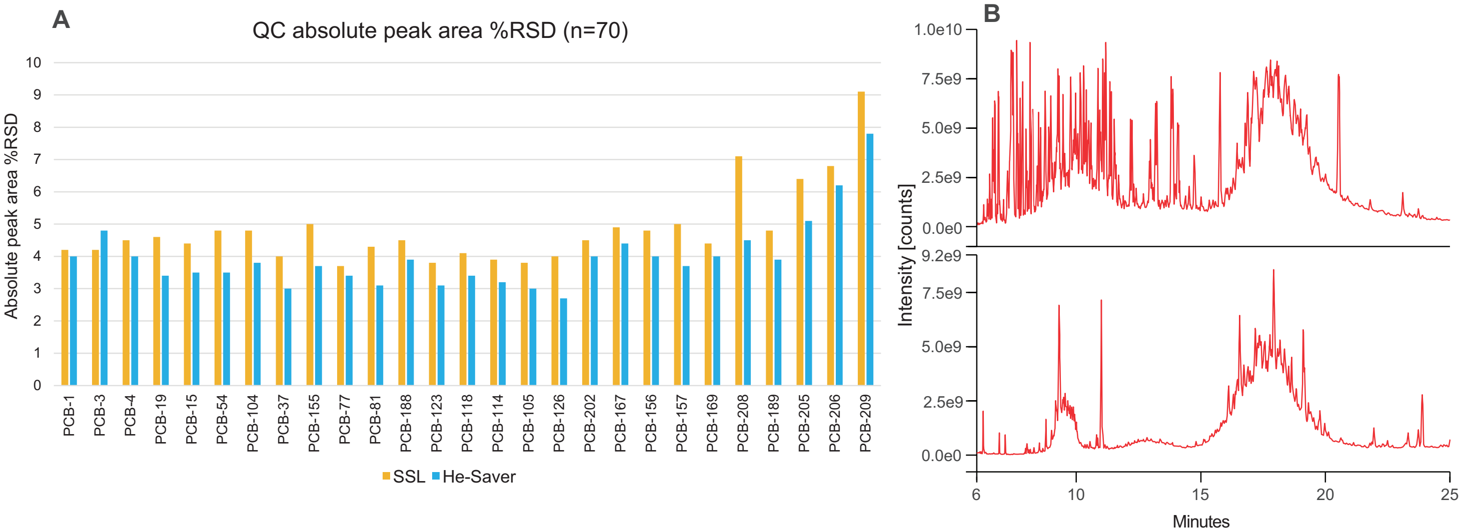
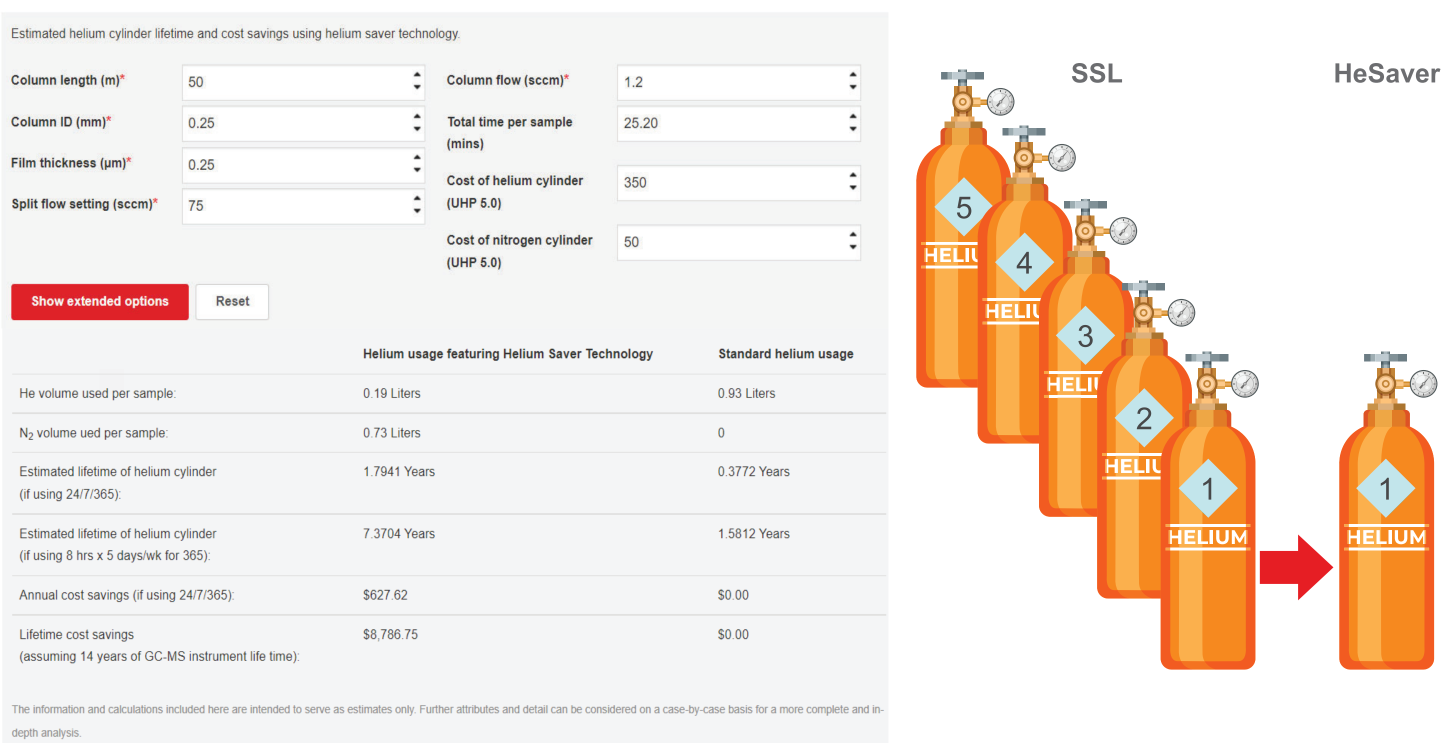


Figure 2. QC absolute peak area %RSD across a sequence of n=100 injections of various environmental extracts (A) and examples of full-scan acquisition (*m/z* 100–600) for environmental sample extracts showing the complexity of the matrix (B)

Reduced helium consumption and cost savings

The HeSaver-H_2Safer technology offers significant gas savings not only when the GC is idle, but mainly during sample injection and analysis. When used with helium as carrier gas, it translates into an extended helium cylinder lifetime from months to years, depending on the instrument method and usage, and how many GCs are connected. The Thermo Scientific™ Gas Saver Calculator tool[3] offers an easy-to-use and intuitive interface to estimate the helium consumption and cost impact. The user is only required to insert the column geometry, the carrier and split flow settings, as well as helium and nitrogen costs, and the tool provides an estimation of both the helium cylinder lifetime and the cost savings. The usage of the HeSaver-H_2Safer technology for the analysis of PCBs according to the U.S. EPA, Method 1668C[4] would allow the helium cylinder to last about 4.5 times more in comparison to the usage of a standard SSL injector (Figure 3).


Estimated helium cylinder lifetime and cost savings using helium saver technology.

Column length (m)*	50	Column flow (sccm)*	1.2
Column ID (mm)*	0.25	Total time per sample (mins)	25.20
Film thickness (µm)*	0.25	Cost of helium cylinder (UHP 5.0)	350
Split flow setting (sccm)*	75	Cost of nitrogen cylinder (UHP 5.0)	50

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	Helium usage featuring Helium Saver Technology	Standard helium usage
He volume used per sample:	0.19 Liters	0.93 Liters
N_2 volume ued per sample:	0.73 Liters	0
Estimated lifetime of helium cylinder (if using 24/7/365):	1.7941 Years	0.3772 Years
Estimated lifetime of helium cylinder (if using 8 hrs x 5 days/wk for 365):	7.3704 Years	1.5812 Years
Annual cost savings (if using 24/7/365):	$627.62	$0.00
Lifetime cost savings (assuming 14 years of GC-MS instrument life time):	$8,786.75	$0.00

The information and calculations included here are intended to serve as estimates only. Further attributes and detail can be considered on a case-by-case basis for a more complete and in-depth analysis.

Figure 3. Gas Saver Calculator reporting the helium saving for U.S. EPA Method 1668C. The cylinder cost is indicative and country dependent.

Summary

The HeSaver-H_2Safer technology offers the advantage of reduced helium gas consumption without compromising GC-MS performance for the analysis of PCBs in environmental samples, through a smooth and simple upgrade of a standard iConnect SSL injector module.

- The transition from the injection phase using an inexpensive pressurizing gas to the separation process using the best carrier gas is extremely fast (within a few milliseconds), ensuring a rapid gas replacement into the column and thus identical performance compared to the standard SSL injector.
- Existing validated methods can be used unchanged, with consistent analytical performance in terms of injection repeatability, analyte transfer, linearity, recovery, and robustness.
- Moreover, the HeSaver-H_2Safer technology provides additional advantages, such as maintaining the column flow during the inlet maintenance and limiting possible septum or liner contaminants into the analytical column.
- The Helium Saver Calculator tool allows for easy and immediate estimation of helium cylinder lifetime and cost savings when using the Helium Saver technology.

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