Green energy

Reducing the helium consumption for analysis of VOCs in accordance with EPA Method 8260D without compromising performance using HeSaver-H2 Safer technology

James Pachlhofer [2], Amy Nutter[1], Terry Jeffers[1], Adam Ladak[3], Daniela Cavagnino[4], and Daniel Kutscher[5]
[1]Teledyne Tekmar, Mason, OH, USA, [2]Thermo Fisher Scientific, USA, [3]Thermo Fisher Scientific, Hemel Hempstead, UK, [4]Thermo Fisher Scientific, Milan, Italy[5]Thermo Fisher Scientific, Bremen, Germany

Abstract

Using GCMS and Purge and trap with Helium saving technology to adhere to EPA 8260D. Reduction of use of helium by 4 times to enable reduction in operation costs. Reproducibility over 240 injections with reproducibility below 10% RSDs.

Introduction

Volatile organic compounds (VOCs) have a high vapor pressure and low water solubility. Many VOCs are human-made chemicals that are used and produced in the manufacture and industry. They can also be generated as by-products of chlorination used in water treatment.[1] Many of these compounds are environmental contaminants and therefore are considered a hazard to human health when present at elevated levels. Analytical testing laboratories often perform the testing to ensure these analytes do not exceed the allowed limits in the environment globally. There are regulations in place for a number of environmental sample types, with one of the most prominent ones being United States Environment Protection Agency (U.S. EPA) method 8260D[2], that is commonly used to monitor a variety of solid waste matrices for the presence of VOCs. The analytical method of choice for the analysis of VOCs is gas chromatography coupled to single quadrupole mass spectrometry combined with purge and trap (P&T) sampling. The sample is purged with an inert gas, causing volatile compounds to be swept out of the sample. The volatile compounds are firstly retained in an adsorbent trap and then desorbed by heating the trap and transferred to the GC column for analysis.

Helium is commonly used as a carrier gas for gas chromatography thanks to its high chromatographic efficiency and inertness. Recent price rises 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-H2Safer™ carrier gas saving technology[3] 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: whereas an inexpensive gas (e.g., nitrogen or argon) is used for inlet pressurization, analyte vaporization and transfer to the analytical column, 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 to mitigate shortage issues while maintaining GC-MS performances without the need of instrument method re-optimization otherwise required in case of migration to a different carrier gas.

A Thermo Scientific™ TRACE™ 1610 gas chromatograph equipped with a Thermo Scientific™ iConnect™ split/splitless injector, upgraded to work in HeSaver-H2Safer mode, was coupled to the Thermo Scientific™ ISQ™ 7610 single quadrupole mass spectrometer and a Teledyne Tekmar Atomx XYZ Purge and Trap (P&T) and used to analyze water and soil samples according to the EPA method 8260D. To perform US EPA 8260C, all method acceptance criteria must be achieved. These criteria include calculating the mean response factor and the relative standard deviation (RSD) of the response factors for target analytes. The RSD should be <20%, with minimum response factors (RF), and MDLs for a wide range of target compounds. The analytical method must produce consistent results and be reproducible from day to day, with a continuing calibration verification (CCV) analyzed every 12 hours while samples are run. As the method covers varying matrices it is important that the performance criteria are met in all samples of interest. Details of the material and the methods used for sample preparation, as well as complete lists of reagents and consumables are reported in a previous application note.[4]

Results

One of the key benefits of the HeSaver-H_2Safer inlet is that the method transfer (for methods using the standard SSL injector) required a minimal effort and the chromatographic performance remained unaffected independent from the analyzed matrix (soil or water). Typical chromatograms for 20 µg/L (in water) and 20 µg/kg (in soil) VOC standards are shown in Figure 1.

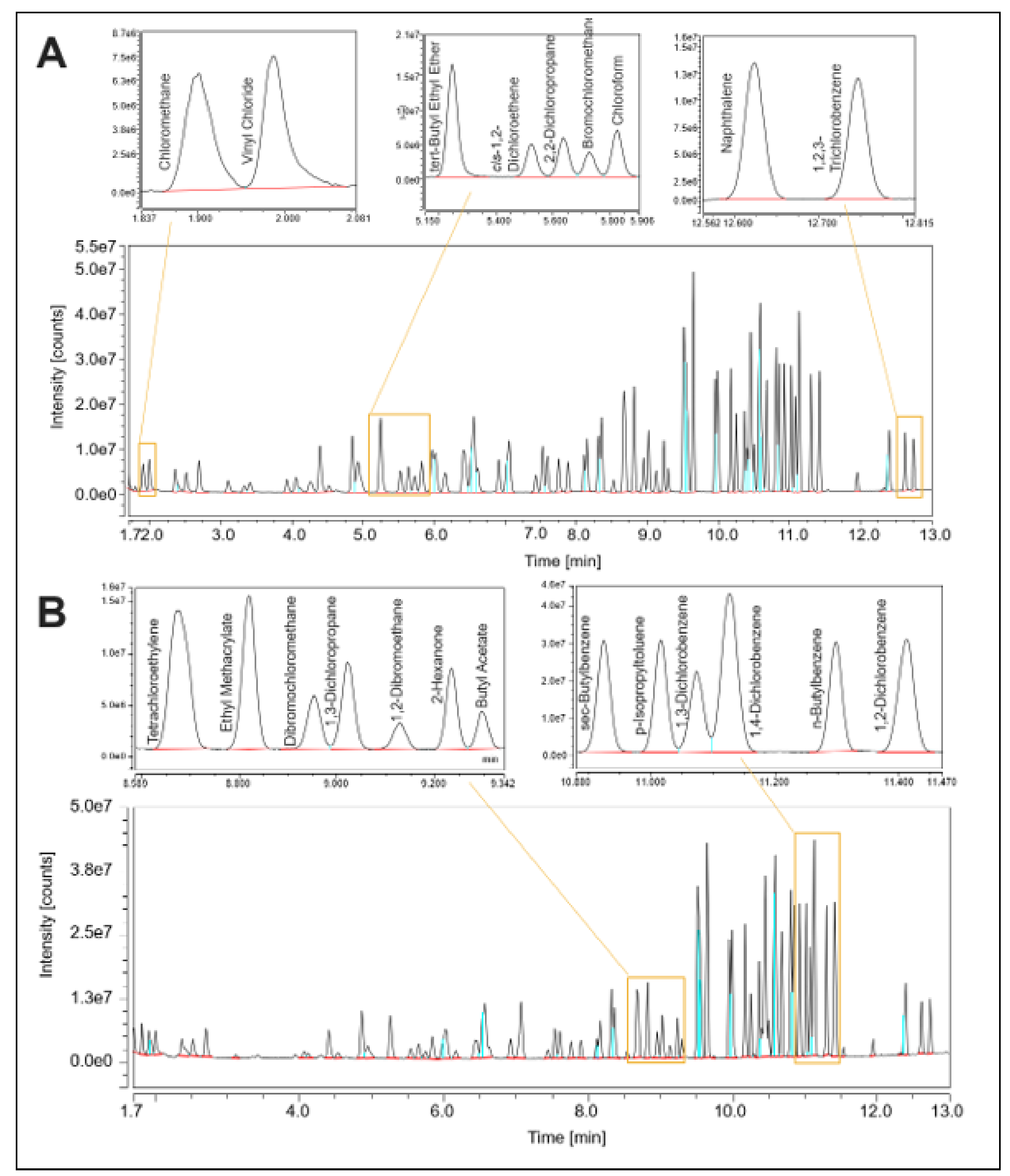

Figure 1. Chromatographic performance achieved with the HeSaver-H2Safer technology for VOC standard in water (A) and soil (B) samples spiked at 20 µg/L and 20 µg/kg respectively.

Linearity was assessed by injecting a calibration curve from 0.5 to 200 µg/L (ppb) for water, whereas for soil matrix, the calibration curve ranged from 1 to 200 µg/kg (ppb). The average response factor RSD for the calibration solutions was in compliance with the EPA method requirements with calculated values <20% for all compounds across the specified concentration range for both water and soil matrices. Examples of extracted ion chromatograms (XICs), as well as spectrum comparison with NIST library and calibration curves obtained for some selected compounds are shown in Figure 2 for water and soil samples spiked with 0.5 µg/L and 5 µg/kg VOC standard solutions respectively.

The method detection limit (MDL) and precision were assessed using n=7 replicates of a 0.5 µg/L VOC standard spiked in water sample and n=7 replicates of a 2 µg/kg VOC standard spiked in soil sample. Calculated MDLs were ≤ 0.14 µg/L and ≤ 0.42 µg/kg for water and soil, respectively, with calculated precision of less than 20% for all compounds in both the analyzed matrices

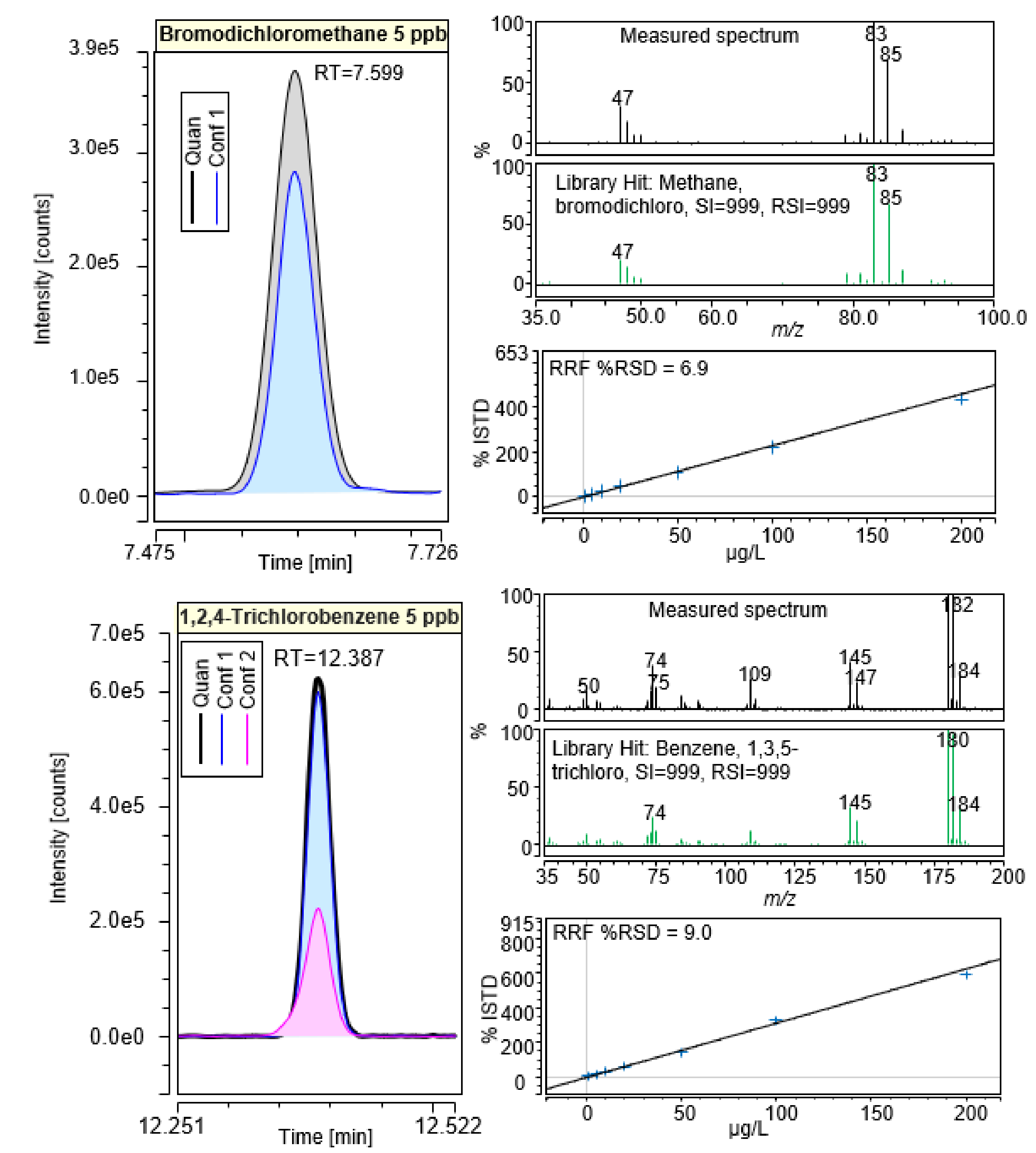

Figure 2. Examples of XICs for quantifier and qualifier ions, spectrum comparison with NIST spectral library and calibration curves

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 therefore limiting the transfer of contaminants (septum/sample matrix/by-products). This was demonstrated by running a 240-sample injection sequence over three days. To monitor system performance, n=40 quality controls (QCs), consisting of water standards at 20 µg/L, were prepared and injected at regular intervals. The water standards were acquired with no user intervention at all on the entire analytical system, including the P&T, GC, or MS system. Excellent system stability was demonstrated with QC precision within ± 30% the expected amount and accuracy ranging from 70 to 117%. The results obtained for some selected compounds covering the entire VOCs boiling point range us shown as an example in Figure 3.

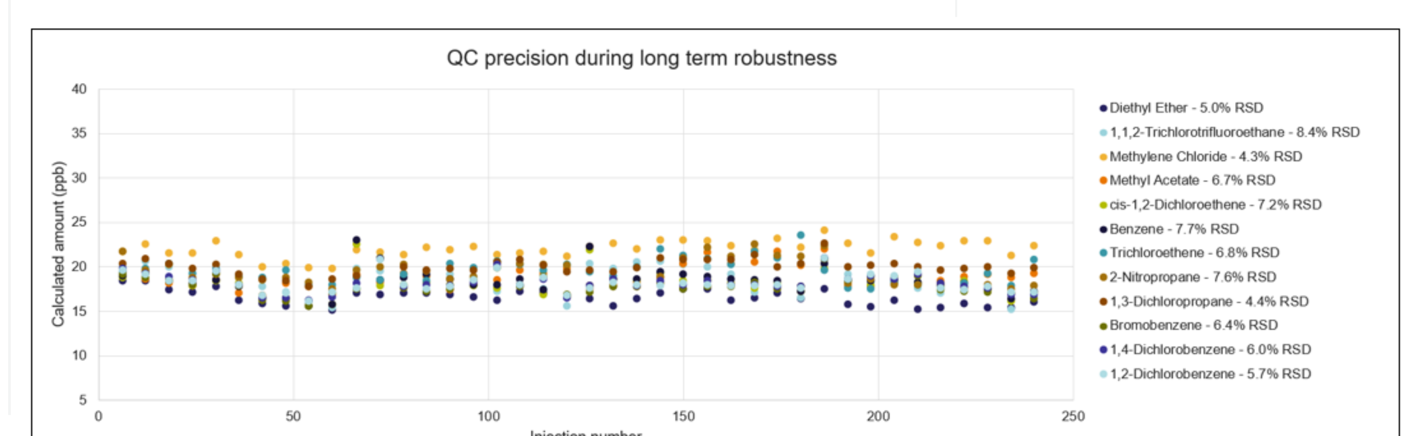

Figure 3. Precision of QC water samples spiked at 20 µg/L VOC standard assessed over n=240 consecutive injections over three days of analysis.

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[5] 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 saving. The use of the HeSaver-H_2Safer technology for the analysis of VOCs according to the U.S. EPA, Method 8260D[2] allows to use a low carrier gas flow (0.3 mL/min) compared to as standard P&T method which would require a helium flow of 0.8 mL/min when a standard SSL would be used. This would allow a helium cylinder to last about 4 times more in comparison to a standard SSL injector (Figure 4).

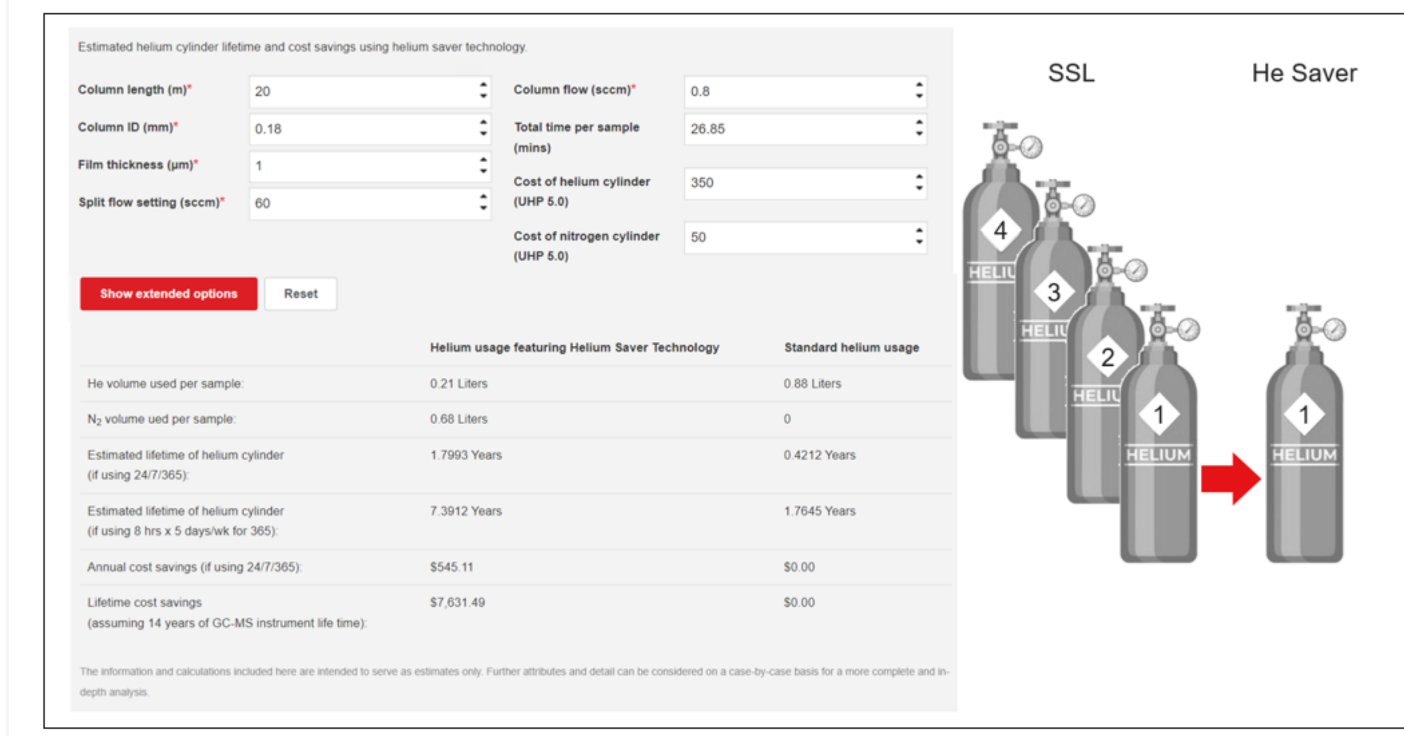

Figure 4. Gas Saver Calculator reporting the helium saving for U.S. EPA method 8260D. The cylinders cost is indicative and country dependent.

Conclusions

The HeSaver-H_2Safer technology offers the advantage of reduced helium gas consumption without compromising GC-MS performance for the analysis of VOCs in environmental samples, through a smooth and simple upgrade of a standard iConnect SSL injector module. HeSaver-H_2Safer technology provides consistent analytical performance compared to the standard SSL injector in terms of analyte transfer, linearity, precision, accuracy, and robustness in compliance with the EPA method 8260D suitability requirements.

For reference and the full application details please scan these QR

Trademarks/licensing

Learn more at thermofisher.com/ISQ7610