

Environmental

Analysis of volatile organic compounds in drinking water according to U.S. EPA Method 524.4

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Keywords

EPA, U.S. EPA 524.4, VOCs, phthalates, trace analysis, gas chromatography, single quadrupole mass spectrometry, selected ion monitoring, sensitivity, GRO, THM, volatiles, environmental lab, environmental sample analysis, analytical testing laboratories, ISQ 7610 MS, TRACE 1610 GC, GC-MS

Goal

Demonstration of an analytical method that meets the requirements outlined in U.S. EPA Method 524.4 for the quantitation of volatile organic compounds (VOCs) in drinking water, using the Teledyne LABS Tekmar Lumin Purge and Trap (P&T) concentrator paired with the AQUATEk LVA autosampler system along with a Thermo Scientific™ ISQ™ 7610 MS system coupled with a Thermo Scientific™ TRACE™ 1610 gas chromatograph (GC) and Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS). Target analyte linearity, method detection limit (MDL), minimum report level (MRL), and mid-point precision and accuracy were assessed to evaluate method performance.

Introduction

Volatile organic compounds (VOCs) are man-made contaminants in various products that pose environmental and public health risks. These compounds are regulated in drinking water, and laboratories follow regulations including U.S. EPA Method 524.2.¹ A previous application note described U.S. EPA Method 524.2,^{2,3} which is the most common method for VOC water testing in the USA. U.S. EPA Method 524.4 is an updated method that allows for nitrogen to be used as the purge gas, thus reducing helium consumption, and is now being more widely used. Targeting 75 VOCs, this method differs by offering more method parameter flexibility but stricter quality control, including the stringent calculation of the minimum reporting level for evaluating calibration checks.

Implementing U.S. EPA Method 524.4 can present multiple challenges for laboratories. Striking a balance between optimized method conditions and meeting the acceptance criteria, like detection limit requirements for various compound concentrations, is pivotal. Another major hurdle is managing the water matrix to minimize GC-MS system water introduction, which could lead to detrimental effects and delay sample reporting, thereby increasing public safety risks. Ultimately, maintaining consistency in test results to avoid sample reanalysis is crucial. Another factor for laboratories to consider is the fluctuating cost of helium, which is the analytical gas of choice for this analysis. Reducing the use of helium—a limited resource—will enable the laboratory to reduce operational costs and increase profitability.

The following evaluation describes the use of the ISQ 7610 GC-MS system coupled with a TRACE 1610 GC with the Thermo Scientific™ HeSaver-H₂Safer™ split/splitless injector and Teledyne LABS Tekmar Lumin P&T concentrator paired with the AQUATEk LVA autosampler for U.S. EPA Method 524.4.

Experimental

Sample preparation

A 50 µg/mL (parts per million or ppm) calibration working standard was prepared in P&T grade methanol (Honeywell/Burdick & Jackson, P/N 232-1L) from the following Restek™ standards: 524.3 VOA MegaMix™ (P/N 30013) and 524.3 Gas Calibration Mix (P/N 30014). In total, the standards contained 75 compounds.

The calibration curve was prepared from 0.2 µg/L to 50 µg/L (parts per billion or ppb) for all compounds. The linear response was calculated for each compound using three Restek internal standards: 1,4-difluorobenzene, chlorobenzene-d₅, and 1,4-dichlorobenzene-d₄ (P/N 30017). Surrogate standards from Restek consisted of methyl-t-butyl ether-d₃, 4-bromofluorobenzene, and 1,2-dichlorobenzene-d₄ (P/N 30017). Internal and surrogate standards were prepared in methanol from Restek standards at a concentration of 10 ppm, after which 5 µL was mixed with each 5 mL sample for a resulting concentration of 10 ppb.

Seven 0.5 ppb standards were prepared in deionized water to calculate the MDL and MRL calculations for all compounds. Also, seven 10 ppb standards were prepared to determine the accuracy and precision of recovery of each compound. All calibration, MDL, MRL, and recovery standards were analyzed with the Tekmar Lumin P&T concentrator and AQUATEk LVA autosampler conditions in Table 1.

Table 1. Teledyne LABS Tekmar Lumin P&T concentrator and AQUATEk LVA autosampler conditions

Standby	Variable
Valve oven temp.	140 °C
Transfer line temp.	140 °C
Sample mount temp.	90 °C
Purge ready temp.	35 °C
MCS purge temp.	20 °C
Standby flow	10 mL/min
Purge	Variable
Purge temp.	20 °C
Purge time	8.00 min
Purge flow	50 mL/min
Dry purge temp.	20 °C
Dry purge time	1.0 min
Dry purge flow	100 mL/min
Sparge vessel heater	Off
Desorb	Variable
Desorb preheat temp.	245 °C
Desorb temp.	250 °C
Desorb time	1.00 min
Desorb flow	300 mL/min
GC start signal	Start_Only
Bake	Variable
Bake time	3.00 min
Trap bake temp.	270 °C
MCS bake temp.	180 °C
Bake flow	200 mL/min
AQUATEk LVA	Variable
Sample loop time	0.35 min
Sample transfer time	0.35 min
Rinse loop time	0.30 min
Sweep needle time	0.30 min
Presweep time	0.25 min
Water temperature	90 °C
Bake rinse cycles	1
Bake rinse drain time	0.35 min
Trap	9
Chiller tray	On
Purge gas	Nitrogen

Since U.S. EPA Method 524.4 has strict carryover requirements, the Tekmar Lumin P&T paired with the AQUATEk LVA is recommended for this analysis. The 2-stage needle displaces the sample from the vial through the 6-valve pressurized manifold and sample loop, decreasing the liquid sample pathway, therefore reducing carryover and contamination. After the sample is desorbed to the GC-MS, the system cleans up with 90 °C water rinsing the entire liquid sample pathway up to three times. The Mass Flow Controller monitors and precisely controls the gas flow rates during this clean-up process, creating an efficient bake out of the P&T, further reducing possible carryover and contamination.

GC-MS conditions

A TRACE 1610 GC was coupled to the ISQ 7610 MS equipped with the Thermo Scientific™ NeverVent™ vacuum probe interlock (VPI) and a Thermo Scientific™ ExtractaBrite™ ion source. A Thermo Scientific™ TraceGOLD™ TG-VMS, 30 m × 0.25 mm, 1.4 µm film column (P/N 26080-3320) was used for compound separation. The HeSaver-H₂Safer injector was operated in split mode with a run time under 13 minutes. HeSaver-H₂Safer technology 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 total flow to a limited value needed to maintain the gas flow rate for the separation process into the analytical column.

The ISQ 7610 MS was operated in full scan mode, offering sufficient sensitivity to achieve the required limits of detection. Note: The instrument can also be operated in Selected Ion Monitoring (SIM) mode to increase selectivity. Expanded method parameters for the ISQ 7610 MS are displayed in Table 2.

Table 2. GC-MS conditions

TRACE 1610 GC conditions	
Column	TraceGOLD TG-VMS, 30 m × 0.25 mm, 1.4 µm film (P/N 26080-3320)
Carrier gas	Helium, 1.5 mL/min
Oven profile	35 °C, 2 min; 15 °C/min to 100 °C; 30 °C /min to 225 °C; 2 min hold; run time 12.5 min
HeSaver H ₂ Safer SSL	200 °C, 30:1 Split, purge flow 5.0 mL/min, 0.30 min helium delay
ISQ 7610 MS conditions	
Temperature	Transfer line 230 °C; ion source 280 °C
Scan range	35 amu to 260 amu
Solvent delay	1.43 min
Dwell/Scan time	0.10 s
Emission current	30 µA
Gain	3.00E+005

Instrument control and data processing

The data was acquired, processed, and reported using Chromeleon CDS software, version 7.3. This software provides control of the entire system including the GC-MS system and the Tekmar Lumin P&T concentrator and AQUATEk LVA autosampler. This allows a single software to be utilized for the full workflow, simplifying the instrument operation.

Results and discussion

Chromatography

All compounds of interest were separated using the GC conditions described in Table 2. Minimal water interference transferred from the samples was indicated by the consistent peak shapes of the compounds. This resulted in optimized chromatography that was maintained in lower concentration samples as show in Figure 1.

Linearity and sensitivity

The calibration range of 0.2 ppb to 50 ppb was assessed for all compounds. Figure 2 demonstrates the quantitation of 4-chlorotoluene at 2 ppb in a VOC standard with excellent library spectral matching and calibration curve. Appendix Table A1 displays the linear value (r^2) and the MDL for each analyte calculated by injecting n=7 injections of the 0.5 ppb standard. Also included in Table A1 is the MRL data which was calculated from injecting n=7 of a 0.5 ppb standard.

Table A1 also shows the mid-point calibration check for the method. This was determined by analyzing a 10 ppb standard seven times to ensure that the precision was under 20% variation and the accuracy was ±30%. The average precision for all compounds was 3.25% and the average accuracy was 102%. This demonstrates that the method is fit for purpose. Figure 3 shows a subset of results for 30 compounds across the analytical range.

Method robustness

When performing VOC analysis, it is essential to produce consistent results to ensure sample results are delivered on time. To assess the stability of the method, 10 ppb calibration check standards were injected at intervals 30 times over a sequence of 185 injections. This extended sequence is equivalent to three days of uninterrupted analysis. No maintenance was performed on any part of the system during this extended test. Figure 4 shows the reproducibility of 10 of the compounds over 185 injections with excellent percentage RSDs. RSDs for all compounds were under the 30% method requirements and gave an average of 6.6% RSD across the compounds. Appendix Table A2 shows the reproducibility results for all compounds over the 185-injection sequence.

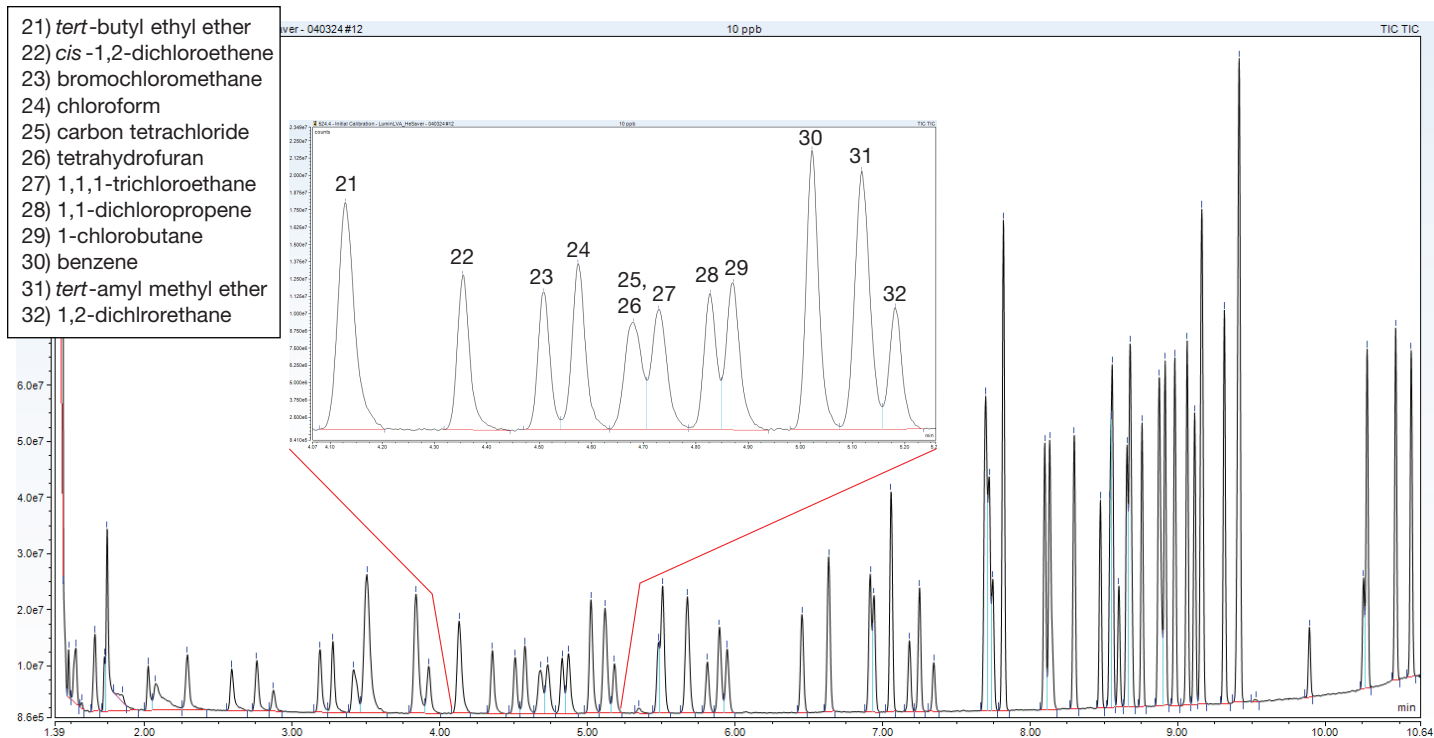


Figure 1. Total ion chromatogram (TIC) of a water method 10 ppb VOC standard with an inset indicating consistent peak shapes and separation with minimal water interference

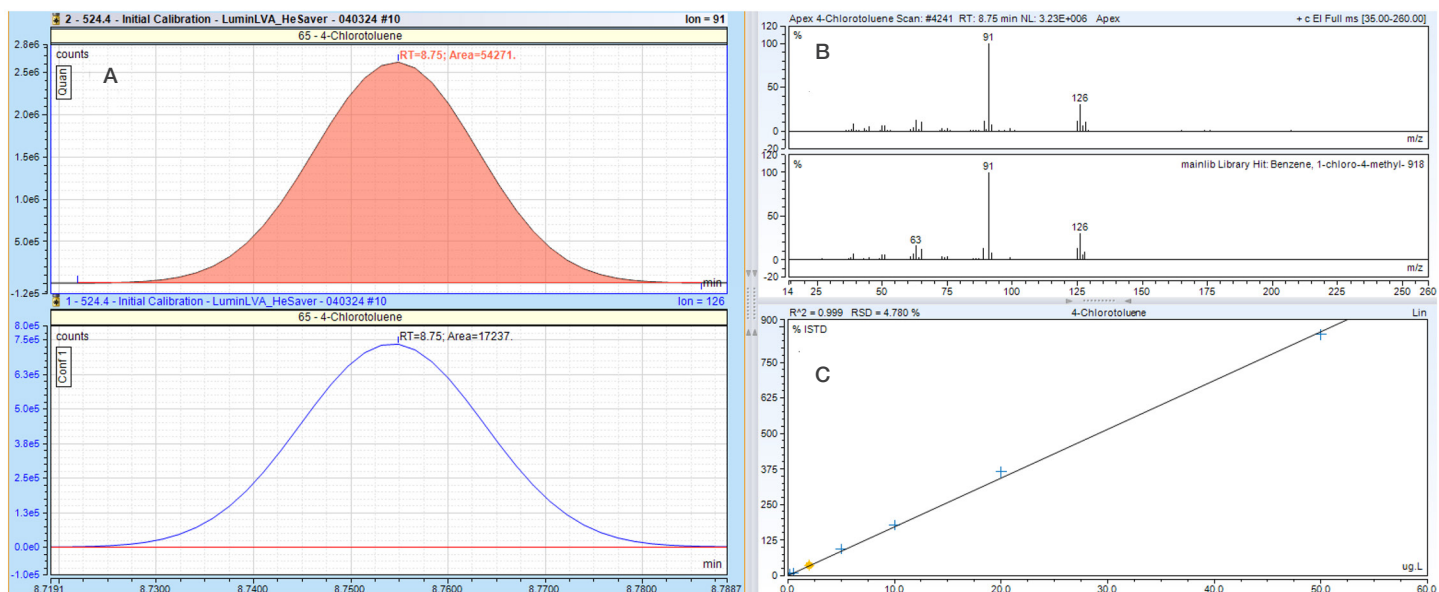


Figure 2. Chromleon CDS results browser showing extracted ion chromatograms for 4-chlorotoluene in the 2 ppb standard, quantitation ion ($m/z = 91$) and one confirming ion ($m/z = 126$) (A), a matching measured spectrum to the NIST library (B), and a linear calibration over a concentration range of 0.2 ppb to 50 ppb (C)

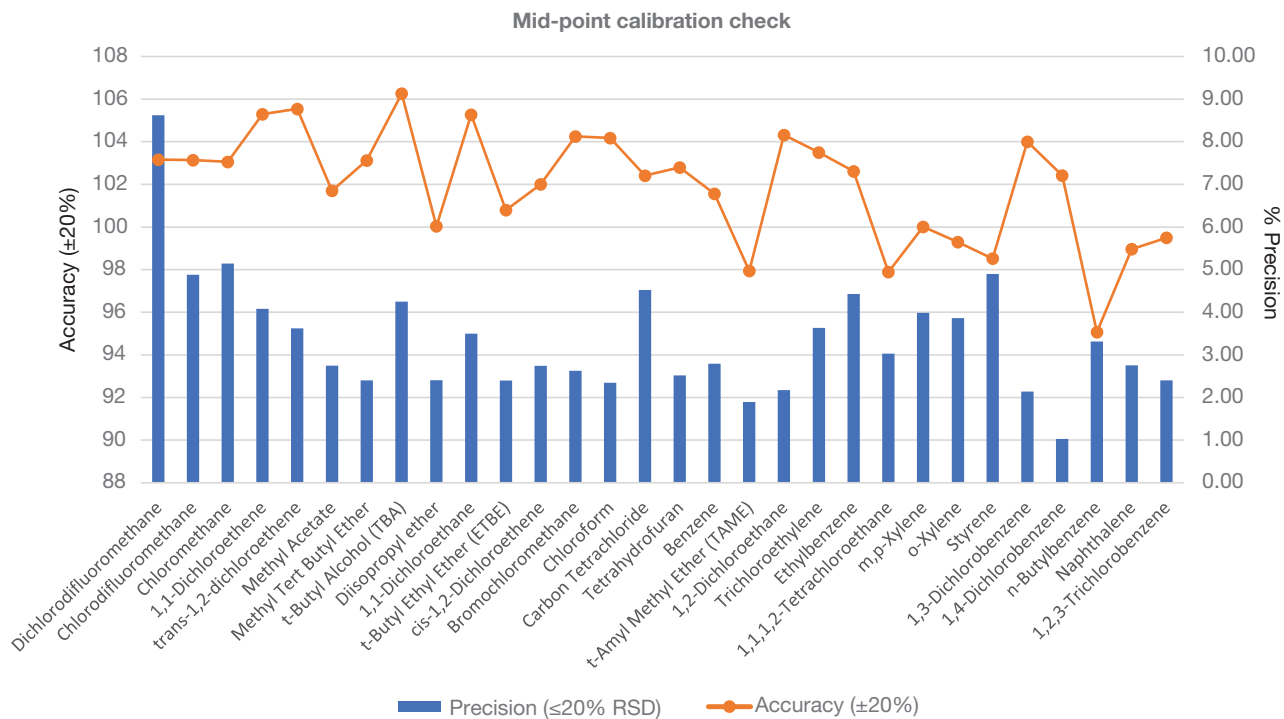


Figure 3. IDC calculations of a subset of compounds for precision and accuracy of $n=7$, 10 ppb injections

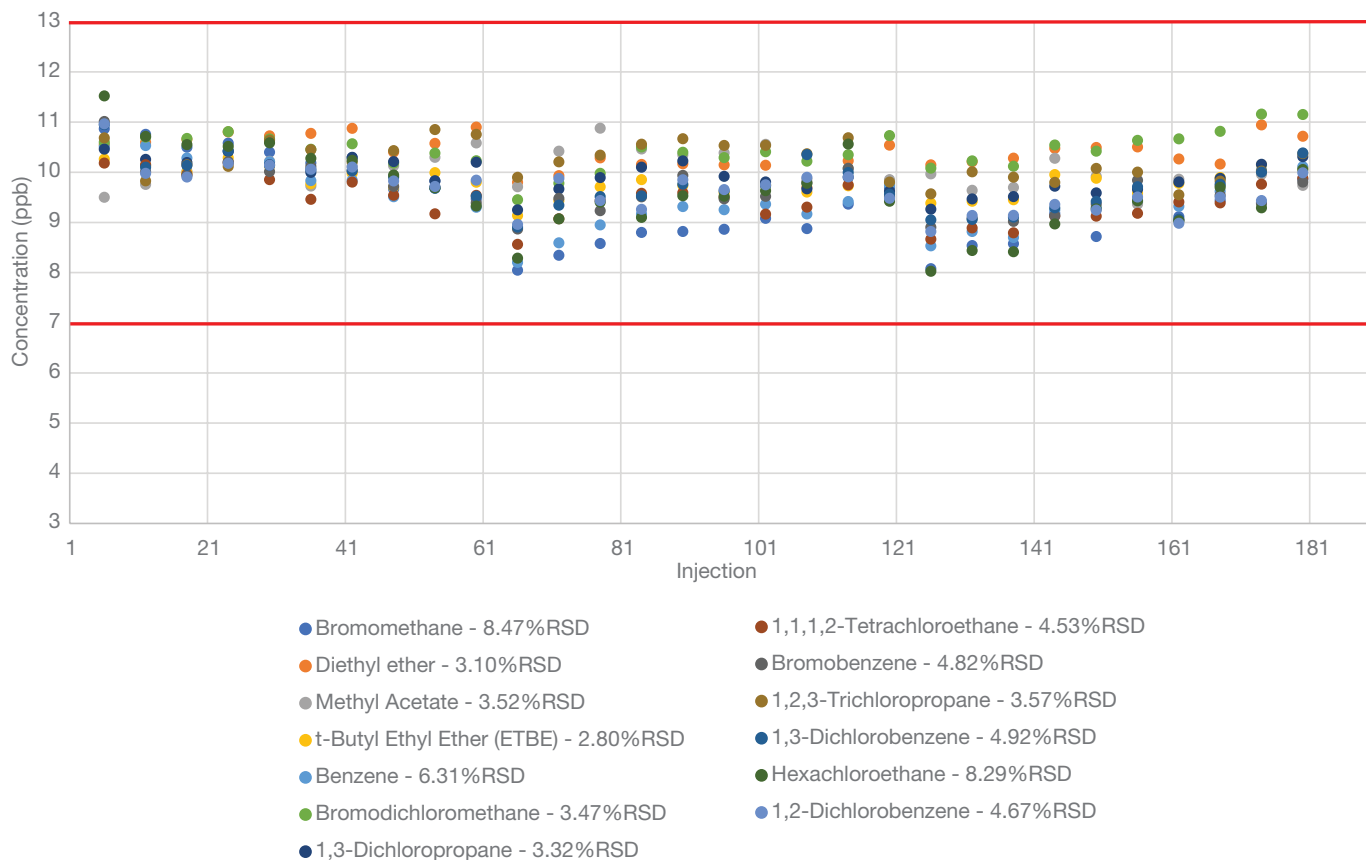


Figure 4. Repeatability of a subset of compounds of a 10 ppb VOC standard ($n=30$) (as concentration) assessed over $n=185$ consecutive injections. Red bars represent $\pm 30\%$ accuracy method requirements.

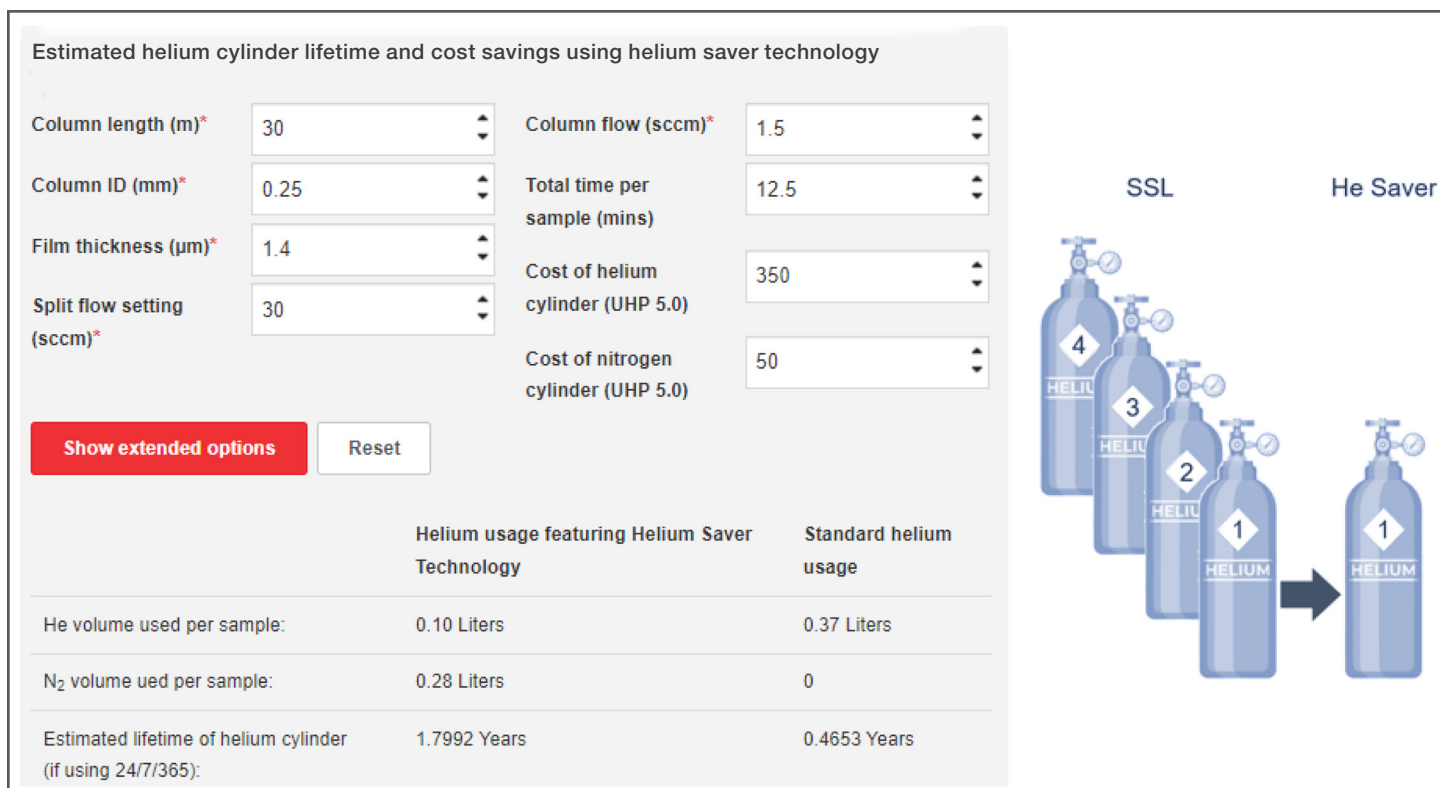


Figure 5. Helium Saver Calculator tool reporting the helium savings

Reduced helium consumption and cost savings

The HeSaver-H₂Safer technology significantly extends helium cylinder lifetime from months to years, saving gas during both idle periods and sample injection/analysis. Its impact can be estimated using the [Thermo Scientific™ Gas Saver Calculator tool](#).⁴ By simply inputting specific parameters (column geometry, carrier and split flow settings, and helium and nitrogen costs), users can determine expected helium consumption, cost impact, and cylinder lifetime. Utilizing this technology for U.S. EPA Method 524.4 analysis can potentially quadruple the helium cylinder lifespan compared to a standard SSL injector (Figure 5), making it an ideal choice for helium conservation.

Conclusion

The combined solution of the TRACE 1610 GC coupled with the ISQ 7610 system and the Tekmar Lumin P&T concentrator with the AQUATEk LVA autosampler system successfully addresses the challenges of VOC analysis and provides a robust, sensitive solution needed for ensuring maximized sample throughput and regulatory method compliance for U.S. EPA Method 524.4.

- Excellent linearity for all compounds was demonstrated with the linear regression ($r^2 \geq 0.995$) of the calibration response factors passing all method requirements.
- MDL and precision for seven 0.5 ppb standards showed no interference from excessive water and produced very reproducible results.
- The precision for n=30 samples over 245 injections displayed <30% RSD for all compounds as the method requires and an average recovery of 91%.
- Utilizing HeSaver-H₂Safer technology reduced helium consumption for the method by 4 times, which provides a significant cost savings.

References

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2. Thermo Fisher Scientific Application Note 000525: Uninterrupted analysis of volatile organic compounds in drinking water using single quadrupole GC-MS. <https://assets.thermofisher.com/TFS-Assets/CMD/Application-Notes/an-000525-gc-ms-volatile-organic-compounds-water-an000525-en.pdf>
3. Thermo Fisher Scientific Application Note 002952: Adhering to U.S. EPA Method 524.2 for the analysis of volatile organic compounds in drinking water. <https://assets.thermofisher.com/TFS-Assets/CMD/Application-Notes/an-002952-gc-ms-isq-environmental-an002952-na-en.pdf>
4. Thermo Fisher Scientific Helium Saver Calculator. <https://www.thermofisher.com/it/en/home/industrial/chromatography/chromatography-learning-center/chromatography-consumables-resources/chromatography-tools-calculators/helium-saver-calculator.html>

Appendix

Table A1 (part 1). U.S. EPA Method 524.4 calibration, accuracy, and precision data

Compound	Calibration				MDL (n=7, 0.5 ppb)		IDC (n=7, 10 ppb)			MRL confirmation (n=7, 0.5 ppb)		
	Retention time	Cal. type	Linearity ($r^2 \geq 0.995$)	Avg. RF	MDL (ppb)	Precision ($\leq 20\% \text{RSD}$)	Avg. conc.	Precision ($\leq 20\% \text{RSD}$)	Accuracy ($\pm 20\%$)	HR _{PIR}	Lower _{PIR} ($\geq 50\%$)	Upper _{PIR} ($\leq 150\%$)
Dichlorodifluoromethane	1.48	Lin	0.997	0.445	0.06	4.1	10.3	8.6	103	0.08	85	117
Chlorodifluoromethane	1.52	Lin	0.997	0.877	0.14	9.0	10.3	4.9	103	0.18	65	136
Chloromethane	1.65	Lin	0.998	0.953	0.11	6.9	10.3	5.1	103	0.14	77	135
Vinyl chloride	1.72	Lin	0.998	0.475	0.12	6.9	10.8	4.9	108	0.15	79	138
1,3-Butadiene	1.74	Lin	0.998	0.493	0.04	2.8	10.6	4.0	106	0.05	87	109
Bromomethane	2.02	Lin, WithOffset, 1/A	0.997	0.383	0.10	6.5	10.3	4.0	103	0.13	75	127
Trichlorofluoromethane	2.28	Lin	0.998	0.489	0.05	3.8	10.4	4.0	104	0.07	77	104
Diethyl ether	2.58	Lin	0.999	0.250	0.05	2.7	10.6	2.3	106	0.06	103	127
1,1-Dichloroethene	2.75	Lin	0.999	0.168	0.11	6.9	10.5	4.1	105	0.14	75	131
Carbon disulfide	2.76	Lin, WithOffset, 1/A	0.998	0.215	0.09	6.2	10.4	3.8	104	0.11	69	114
Methyl iodide	2.87	Lin, WithOffset, 1/A	0.995	0.274	0.10	7.9	9.7	2.5	97	0.13	56	108
Allyl chloride	3.18	Lin	0.998	0.138	0.12	6.8	10.4	2.9	104	0.15	84	145
Methylene chloride	3.27	Lin	0.997	0.269	0.06	3.9	10.6	1.9	106	0.08	89	122
trans-1,2-dichloroethene	3.40	Lin	0.998	0.189	0.10	5.6	10.6	3.6	106	0.13	89	139
Methyl acetate	3.43	Lin	0.999	0.320	0.14	9.4	10.2	2.7	102	0.18	61	133
Methyl- <i>t</i> -butyl ether-d3 (surr)	3.49	AvgCalFact	2.09	1.08		1.5	9.9	1.6	99	0.57	94	105
Methyl <i>tert</i> butyl ether	3.50	Lin	0.999	0.980	0.05	2.6	10.3	2.4	103	0.06	102	126
<i>t</i> -Butyl alcohol (TBA)	3.59	Lin	0.999	0.040	0.08	4.6	10.6	4.2	106	0.10	91	131
Diisopropyl ether	3.83	Lin	1.00	1.55	0.06	3.6	10.0	2.4	100	0.07	85	113
1,1-Dichloroethane	3.91	Lin	0.998	0.549	0.07	3.9	10.5	3.5	105	0.09	94	129
<i>t</i> -Butyl ethyl ether (ETBE)	4.12	Lin	1.000	1.04	0.05	3.4	10.1	2.4	101	0.07	88	115
<i>cis</i> -1,2-Dichloroethene	4.35	Lin	0.999	0.298	0.06	3.2	10.2	2.7	102	0.07	100	129
Bromochloromethane	4.50	Lin	0.999	0.137	0.07	4.0	10.4	2.6	104	0.09	93	129
Chloroform	4.57	Lin	0.998	0.574	0.06	3.4	10.4	2.3	104	0.08	99	130
Carbon tetrachloride	4.67	Lin	0.999	0.261	0.10	6.5	10.2	4.5	102	0.13	73	123
Tetrahydrofuran	4.68	Lin	0.999	0.045	0.13	7.1	10.3	2.5	103	0.16	82	146
1,1,1-Trichloroethane	4.72	Lin	0.998	0.380	0.09	5.6	10.3	3.4	103	0.11	79	125
1,1-Dichloropropene	4.82	Lin	0.998	0.098	0.10	6.1	10.4	4.1	104	0.12	76	124
1-Chlorobutane	4.86	Lin	0.999	0.436	0.06	4.0	10.4	4.3	104	0.08	86	118
Benzene	5.02	Lin	1.00	1.04	0.06	3.3	10.2	2.8	102	0.07	94	123
<i>t</i> -Amyl methyl ether (TAME)	5.11	Lin	1.00	0.905	0.06	3.8	9.8	1.9	98	0.07	82	111
1,2-Dichloroethane	5.17	Lin	0.998	0.401	0.06	3.3	10.4	2.2	104	0.07	97	126
Trichloroethylene	5.47	Lin	1.00	0.236	0.08	4.9	10.3	3.6	103	0.10	87	128
1,4-Difluorobenzene (ISTD)	5.50	AvgCalFact										
<i>t</i> -Amyl ethyl ether (TAEE)	5.67	Lin	1.00	0.808	0.04	2.3	9.9	2.0	99	0.05	91	109
Dibromomethane	5.80	Lin	0.998	0.202	0.05	2.9	10.4	2.0	104	0.06	98	124
1,2-Dichloropropane	5.88	Lin	0.999	0.365	0.04	2.4	10.4	2.3	104	0.05	100	121
Bromodichloromethane	5.94	Lin	0.999	0.448	0.08	4.6	10.0	1.8	100	0.10	85	124
<i>cis</i> -1,3-Dichloropropene	6.44	Lin	1.00	0.567	0.09	5.5	10.0	2.6	100	0.12	82	128
Toluene	6.63	Lin, WithOffset, 1/A	1.00	0.948	0.06	4.4	9.8	3.7	98	0.08	72	102
Tetrachloroethylene	6.91	Lin	0.999	0.455	0.10	6.0	11.7	6.3	117	0.13	82	133

Table A1 (part 2). U.S. EPA Method 524.4 calibration, accuracy, and precision data

Compound	Calibration				MDL (n=7, 0.5 ppb)		IDC (n=7, 10 ppb)			MRL confirmation (n=7, 0.5 ppb)		
	Retention time	Cal. type	Linearity ($r^2 \geq 0.995$)	Avg. RF	MDL (ppb)	Precision ($\leq 20\% \text{RSD}$)	Avg. conc.	Precision ($\leq 20\% \text{RSD}$)	Accuracy ($\pm 20\%$)	HR _{PIR}	Lower _{PIR} ($\geq 50\%$)	Upper _{PIR} ($\leq 150\%$)
<i>trans</i> -1,3-Dichloropropene	6.93	Lin	1.00	0.560	0.04	2.5	10.0	2.9	100	0.05	94	115
1,1,2-Trichloroethane	7.05	Lin	1.00	0.325	0.07	4.4	9.7	3.0	97	0.09	85	122
Ethyl methacrylate	7.05	Lin	1.00	0.468	0.07	4.2	9.8	2.2	98	0.08	84	118
Dibromochloromethane	7.17	Lin	1.00	0.337	0.04	2.4	9.9	2.7	99	0.05	90	109
1,3-Dichloropropane	7.24	Lin	1.00	0.608	0.07	3.9	10.1	2.8	101	0.08	91	125
1,2-Dibromoethane	7.34	Lin	0.999	0.329	0.06	3.4	10.1	2.6	101	0.07	91	119
Chlorobenzene-d5 (ISTD)	7.68	AvgCalFact										
Chlorobenzene	7.69	Lin	1.00	0.876	0.05	2.9	9.9	3.3	99	0.06	92	116
Ethylbenzene	7.71	Lin	0.998	1.44	0.06	3.7	10.3	4.4	103	0.08	93	125
1,1,1,2-Tetrachloroethane	7.73	Lin	1.00	0.305	0.04	2.5	9.8	3.0	98	0.05	87	106
<i>m,p</i> -Xylene	7.81	Lin	0.999	0.567	0.10	3.3	20.0	4.0	100	0.13	88	115
<i>o</i> -Xylene	8.09	Lin	0.999	0.607	0.05	3.3	9.9	3.9	99	0.07	89	116
Styrene	8.12	Lin	0.999	0.994	0.05	3.3	9.9	4.9	99	0.07	90	117
Bromoform	8.14	Lin	0.999	0.283	0.06	4.6	9.2	2.5	92	0.08	71	103
Isopropylbenzene	8.29	Lin	1.00	1.52	0.07	4.5	9.8	4.0	98	0.08	78	112
4-Bromofluorobenzene (surr)	8.47	AvgCalFact				2.1	9.8	1.5	98	0.83	93	110
Bromobenzene	8.53	Lin	1.00	0.694	0.05	3.0	10.4	2.0	104	0.07	97	124
<i>n</i> -Propylbenzene	8.55	Lin	0.999	2.78	0.08	4.7	10.4	3.1	104	0.10	86	125
1,1,1,2-Tetrachloroethane	8.59	Lin	0.999	0.747	0.04	2.2	10.2	2.3	102	0.05	96	115
2-Chlorotoluene	8.64	Lin	1.00	0.551	0.05	2.9	10.2	2.9	102	0.06	97	122
1,3,5-Trimethylbenzene	8.66	Lin	0.998	1.99	0.07	4.5	10.5	2.6	105	0.09	86	124
1,2,3-Trichloropropane	8.68	Lin	0.998	0.187	0.07	4.0	10.4	3.1	104	0.09	91	125
4-Chlorotoluene	8.75	Lin	0.999	1.83	0.07	3.9	10.4	2.3	104	0.08	93	127
<i>tert</i> -Butylbenzene	8.86	Lin	1.00	0.570	0.07	4.6	9.7	3.6	97	0.09	83	119
Pentachloroethane	8.88	Lin	0.999	0.243	0.13	10.0	10.2	8.7	102	0.17	52	120
1,2,4-Trimethylbenzene	8.90	Lin	0.999	2.08	0.05	3.0	10.5	2.9	105	0.06	94	119
<i>sec</i> -Butylbenzene	8.97	Lin	0.999	2.66	0.05	3.5	10.3	3.5	103	0.07	84	111
<i>p</i> -Isopropyltoluene	9.05	Lin	1.00	2.18	0.04	2.7	10.0	3.3	100	0.05	87	108
1,3-Dichlorobenzene	9.10	Lin	0.999	1.50	0.04	2.2	10.2	2.1	104	0.05	100	119
1,4-Dichlorobenzene-d4 (ISTD)	9.15	AvgCalFact										
1,4-Dichlorobenzene	9.16	Lin	0.999	1.56	0.06	3.2	10.2	1.0	102	0.07	96	124
<i>n</i> -Butylbenzene	9.30	Lin	0.999	0.749	0.07	4.6	9.5	3.3	95	0.08	75	108
Hexachloroethane	9.39	Lin	0.997	0.296	0.12	6.7	10.9	3.4	109	0.16	87	150
1,2-Dichlorobenzene (surr)	9.40	AvgCalFact				0.8	9.9	2.1	99	0.32	96	102
1,2-Dichlorobenzene	9.41	Lin	0.999	1.59	0.05	2.9	10.2	2.2	102	0.06	96	121
1,2-Dibromo-3-chloropropane	9.88	Lin	0.999	0.220	0.06	3.8	10.2	2.2	102	0.08	90	122
Hexachlorobutadiene	10.25	Lin	0.999	0.073	0.06	3.7	10.4	4.6	104	0.07	88	118
1,2,4-Trichlorobenzene	10.27	Lin	1.00	1.28	0.05	3.5	9.7	2.8	97	0.07	86	113
Naphthalene	10.46	Lin	0.999	2.89	0.04	2.7	9.9	2.8	99	0.05	90	111
1,2,3-Trichlorobenzene	10.57	Lin	0.999	1.22	0.04	2.8	9.9	2.4	99	0.06	91	114

Table A2. Repeatability of a 10 ppb VOC standard (n=40) (as absolute peak area counts) assessed over n=245 consecutive injections

Compound	Analyte recovery (10 ppb n=30, 185 injections)		Compound	Analyte recovery (10 ppb n=30, 185 injections)		Compound	Analyte recovery (10 ppb n=30, 185 injections)	
	Precision (≤20%RSD)	Accuracy (±30%)		Precision (≤20%RSD)	Accuracy (±30%)		Precision (≤20%RSD)	Accuracy (±30%)
Dichlorodifluoromethane	10.4	97	1-Chlorobutane	8.7	97	4-Bromofluorobenzene (surr)	2.1	104
Chlorodifluoromethane	10.2	102	Benzene	6.3	95	Bromobenzene	4.8	96
Chloromethane	10.1	90	<i>t</i> -Amyl methyl ether (TAME)	2.9	92	<i>n</i> -Propylbenzene	7.7	100
Vinyl chloride	11.2	96	1,2-Dichloroethane	3.3	109	1,1,2,2-Tetrachloroethane	10.1	93
1,3-Butadiene	14.2	87	Trichloroethylene	10.2	102	2-Chlorotoluene	6.2	95
Bromomethane	8.5	94	1,4-Difluorobenzene (ISTD)			1,3,5-Trimethylbenzene	6.9	98
Trichlorofluoromethane	10.2	102	<i>t</i> -Amyl ethyl ether (TAEF)	3.5	96	1,2,3-Trichloropropane	3.6	102
Diethyl ether	3.1	104	Dibromomethane	3.2	105	4-Chlorotoluene	5.3	103
1,1-Dichloroethene	9.6	94	1,2-Dichloropropane	4.0	104	<i>tert</i> -Butylbenzene	8.4	87
Carbon disulfide	14.6	77	Bromodichloromethane	3.5	104	1,2,4-Trimethylbenzene	6.3	99
Methyl iodide	9.6	87	<i>cis</i> -1,3-Dichloropropene	3.9	95	<i>sec</i> -Butylbenzene	8.6	95
Allyl chloride	7.4	96	Toluene	7.3	88	<i>p</i> -Isopropyltoluene	9.0	89
Methylene chloride	4.4	105	Tetrachloroethylene	12.4	115	1,3-Dichlorobenzene	4.9	97
<i>trans</i> -1,2-dichloroethene	8.4	95	<i>trans</i> -1,3-Dichloropropene	4.0	95	1,4-Dichlorobenzene-d4 (ISTD)		
Methyl acetate	3.5	100	1,1,2-Trichloroethane	3.7	97	1,4-Dichlorobenzene	4.8	98
Methyl- <i>t</i> -butyl ether-d3 (surr)	1.9	102	Ethyl methacrylate	4.8	86	<i>n</i> -Butylbenzene	12.3	80
Methyl <i>tert</i> butyl ether	2.2	102	Dibromochloromethane	3.7	97	Hexachloroethane	8.3	96
<i>t</i> -Butyl alcohol (TBA)	6.1	103	1,3-Dichloropropane	3.3	99	1,2-Dichlorobenzene (surr)	1.6	98
Diisopropyl ether	3.2	100	1,2-Dibromoethane	3.7	94	1,2-Dichlorobenzene	4.7	97
1,1-Dichloroethane	6.4	106	Chlorobenzene-d5 (ISTD)			1,2-Dibromo-3-chloropropane	6.1	88
<i>t</i> -Butyl ethyl ether (ETBE)	2.8	98	Chlorobenzene	5.7	93	Hexachlorobutadiene	11.0	85
<i>cis</i> -1,2-Dichloroethene	5.1	99	Ethylbenzene	7.0	97	1,2,4-Trichlorobenzene	10.0	78
Bromochloromethane	4.1	100	1,1,1,2-Tetrachloroethane	4.5	95	Naphthalene	11.6	79
Chloroform	5.1	108	<i>m,p</i> -Xylene	7.7	91	1,2,3-Trichlorobenzene	10.0	81
Tetrahydrofuran	4.1	96	<i>o</i> -Xylene	6.7	90			
Carbon Tetrachloride	9.3	101	Styrene	5.9	89			
1,1,1-Trichloroethane	8.2	102	Bromoform	4.5	83			
1,1-Dichloropropene	9.8	89	Isopropylbenzene	8.3	89			

Pentachloroethane is a reactive compound and broke down during the long-term study.

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