APPLICATION NOTE

Analysis of Volatile Organic Compounds (VOCs) in Water using HS-GC-MS with EPA Method 8260: Static vs Dynamic Headspace

AN188v1; March 2026, SCION Instruments

Introduction

Volatile organic compounds (VOCs) are a group of chemicals, e.g. xylene, which readily volatilize at low temperatures from certain liquids and solids.[1] VOCs can be released from an extensive number of sources as organic chemicals are used in a wide variety of everyday products such as paints, fuels and household cleaners. VOCs are known to lead to both short term and long term health effects from eye, nose and throat irritation to causing damage to kidneys, liver and the central nervous system.[2]

EPA Method 8260 is a versatile method for the determination of VOC content in a variety of samples matrices such as water and soil by gas chromatography-mass spectrometry (GC-MS). A large number of VOCs have been outlined in this method with guidance on appropriate sampling techniques, sample preparation and analysis of these compounds. In this method it states that headspace may be used for the introduction of VOCs from water samples into a GCMS system with guidance from EPA Method 5021.[3] See our technical note on EPA Method 5021 for more information.

Headspace is a sample preparation technique which can be used to extract VOCs from both solid and liquid matrices without the need for extensive sample preparation.

In this application note we will validate a method for VOC analysis in water samples with guidance from EPA method 8260 using the SCION Instruments HT3 headspace sampler combined with the SCION 8300 GC and SQ 8700 MS.

The HT3 headspace sampler can be used in static mode using a loop method or be converted into dynamic mode and use a trap method. In the application note, we will show results for the analysis of VOCs in water using the HT3 in static and also dynamic mode to draw a comparison.

Experimental

An internal standard (IS) was implemented in this application to improve the precision of the results. The internal standard selected for this application was fluorobenzene which was added at the same concentration to all samples. For more information about the importance of using an internal standard, see our technical note. A working standard was prepared at a concentration of 10 µg/ mL in methanol.

Table 1 Instrument parameters for GC-MS

GC Part	Settings
Injector (SSL)	200 °C Split 20:1
Liner	Narrow Bore Straight Through
Column	SCION 624-MS 30 m x 0.25 mm x 1.4 µm
Carrier Gas	Helium 1.5 mL/min
Oven Program	40 °C, hold 5 min 8 °C/ min to 180 °C, hold 0.17 min 30 °C/ min to 250 °C
Run Time	25.0 min
Software	MSWS
MS Part	**Settings**
MS transfer line temp.	230 °C
Ion source temp.	250 °C
MS mode	Electron ionization
Delay collection time	5.0 min
Scan mode	SIM

Commercially available VOC standards were purchased for this application which contained a range of VOC compounds, with a concentration of 2000 µg/ mL, including those reported in Table 4. Due to the number of compounds present in the standard, not all have been mentioned in the Results section. If necessary, the full validation report is accessible by request.

To prepare the linearity samples a stock standard was prepared with a concentration of 1 µg/ mL in water. This was used to prepare linearity samples at 5 concentration levels: 1, 5, 10, 30 and 100 ppb in water. All linearity samples contained 100 ppb of IS. 1 mL of each solution was added to individual headspace vials, ready for analysis.

Water samples were prepared with IS (100 ppb). QC spiked water samples were prepared with IS (100 ppb) and by spiking with the VOC standard (30 ppb). To individual headspace vials, 1 mL of sample was added, ready for analysis.

Into headspace vials, 1 mL water was added for solvent blanks.

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Table 2 HT3 static headspace (Loop) settings

Variable	Value
Constant Heat Time	ON
GC Cycle Time	35.00 min
Valve Oven Temp.	120 °C
Transfer Line Temp.	120 °C
Standby Flow Rate	30 mL/ min
Platen/ Sample Temp.	70 °C
Platen Temp Equil. Time	1.00 min
Sample Equil. Time	1.00 min
Mixer	ON
Mixing Level	Level 5
Mixing Time	10.00 min
Mixer Stabilize Time	0.50 min
Pressurize	5 PSIG
Pressurize Time	0.15 min
Pressure Equil. Time	0.20 min
Loop Fill Pressure	3 PSIG
Loop Fill Time	1.00 min
Inject Time	0.50 min
Software	HT3 TekLink

Table 3 HT3 dynamic headspace (trap) settings

Variable	Value
Trap	Trap K Vocarb 3000
Valve Oven Temp.	140 °C
Transfer Line Temp.	140 °C
Standby Flow Rate	30 mL/min
Trap Standby Temp.	30 °C
Trap Sweep Temp.	30 °C
Platen/ Sample Temp.	70 °C
Sample Preheat Time	0.00 min
Preheat Mixer	ON
Preheat Mixing Level	Level 5
Preheat Mixing Time	10.00 min
Preheat Mixer Stabilize Time	0.50 min
Sweep Flow Rate	40 mL/ min
Sweep Flow Time	3.00 min
Dry Purge Time	0.50 min
Dry Purge Flow	50 mL/min
Dry Purge Temp.	30 °C
Desorb Preheat	270 °C
Desorb Temp.	275 °C
Desorb Time	5.00 min
Trap Bake Temp.	300 °C
Trap Bake Time	5.00 min
Trap Bake Flow	50 mL/ min
Software	HT3 Teklink

Instrument parameters for the GC-MS can be found in Table 1. Table 2 and Table 3 show the headspace sampler parameters for the HT3 in static mode (loop) and dynamic mode (trap), respectively. The system showed excellent specificity with compounds resolving well from one another and exhibiting good peak shape.

To improve system sensitivity, Selective Ion Monitoring (SIM) was employed. Each VOC had a single quantifier ion and two qualifier ions selected (Table 4). The quantifier ion was chosen by selecting the most abundant ion for each VOC. Qualifier ions are important in analyte confirmation and used to prevent false identification of compounds. Qualifier ions are selected due to being the 2nd and 3rd most abundant ions for each compound. See Figure 1 for an example from our MS software, MSWS, of the quantifier and qualifier ions selected for 4-Isopropyltoluene.

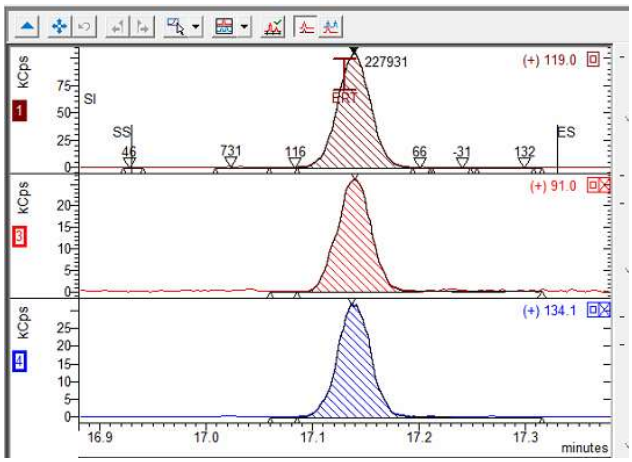

Figure 1 Example mass spectra of 4-Isopropyltoluene from linearity sample (30 ppb) ran using dynamic headspace sampling showing quantifier (top) and qualifier plots (middle and bottom).

Results

Table 4 shows the correlation coefficient (r^2) and the RSD for the system precision (n=6) for the selected VOCs using static headspace. The r^2 values found using static headspace were all >0.996 with the majority >0.998 which demonstrates excellent linearity as the EPA method 8260 states that the initial calibration should achieve a $r^2 \geq 0.99$.[3]

Table 5 shows the correlation coefficient (r^2) and the RSD for the system precision (n=5) for the selected VOCs using dynamic headspace. The r^2 values found using dynamic headspace were all >0.98 with the majority >0.993. EPA method 8260 states that due to the large number of compounds which can be analyzed using this method, it is common that some compounds will not reach the acceptance criteria and this method does allow for 10% of compounds to exceed this correlation coefficient value.

As per EPA 8260, calibration models may use an alternate fit such as forcing through the origin when it is appropriate for your project but it is deemed inappropriate to make changes once

Table 4 Correlation coefficient and system precision results for VOCs using static headspace

VOC	Correlation coefficient (r^2)	RSD (%)	SIM Ions (Quantifier and qualifiers)
2-Chlorotoluene	0.9988	2.98	91, 89,126
4-Isopropyltoluene	0.9982	5.20	119, 91, 134
Benzene	0.9985	0.83	78, 77, 51
1,3,5-trimethylbenzene	0.9986	5.29	105, 120, 77
tert-Butylbenzene	0.9984	4.07	119, 91, 134
Ethylbenzene	0.9982	2.82	91, 106, 65
Isopropylbenzene	0.9987	3.72	105, 120, 79
m-Xylene & p-Xylene	0.9983	2.96	91, 106, 105
n-Propylbenzene	0.9987	3.81	91, 120, 65
o-Xylene	0.9982	2.35	91, 106, 105
sec-Butylbenzene	0.9990	4.89	105, 134, 91

Table 5 Correlation coefficient and system precision results for VOCs using dynamic headspace

VOC	Correlation coefficient (r^2)	RSD (%)	SIM Ions (Quantifier and qualifiers)
2-Chlorotoluene	0.9953	9.14	91, 89,126
4-Isopropyltoluene	0.9966	10.06	119, 91, 134
Benzene	0.9984	17.39	78, 77, 51
1,3,5-trimethylbenzene	0.9963	9.55	105, 120, 77
tert-Butylbenzene	0.9964	10.04	119, 91, 134
Ethylbenzene	0.9972	9.20	91, 106, 65
Isopropylbenzene	0.9963	8.56	105, 120, 79
m-Xylene & p-Xylene	0.9967	7.39	91, 106, 105
n-Propylbenzene	0.9965	8.46	91, 120, 65
o-Xylene	0.9957	8.07	91, 106, 105
sec-Butylbenzene	0.9961	9.89	105, 134, 91

the calibration has been finalized, just to pass the QC criteria. In this application, using dynamic headspace it was found 29.2 % of compounds analyzed had an r^2 value <0.99. The method does state that data for compounds which do not meet the criteria and are not critical to the specific project can be used as qualified data or estimated values for screening purposes.[3] Examples of calibration curves calculated for 4-Isopropyltoluene can be seen in Figures 2 and 3 for static and dynamic headspace respectively.

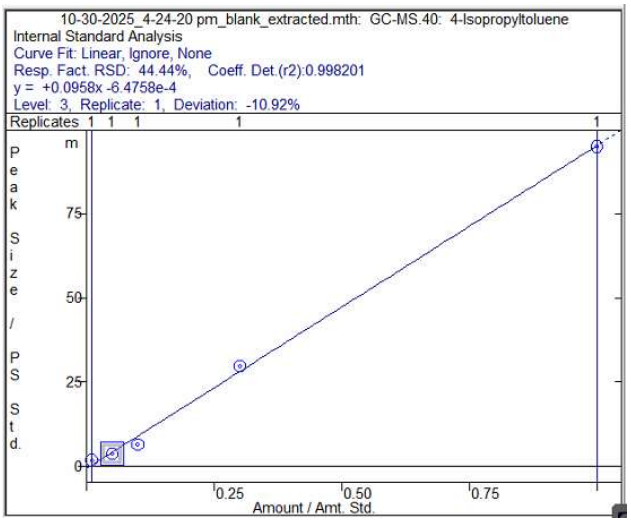

Figure 2 Example from linearity ran using static headspace sampling for 4-Isopropyltoluene, showing correlation coefficient (r^2) from data review in MSWS.

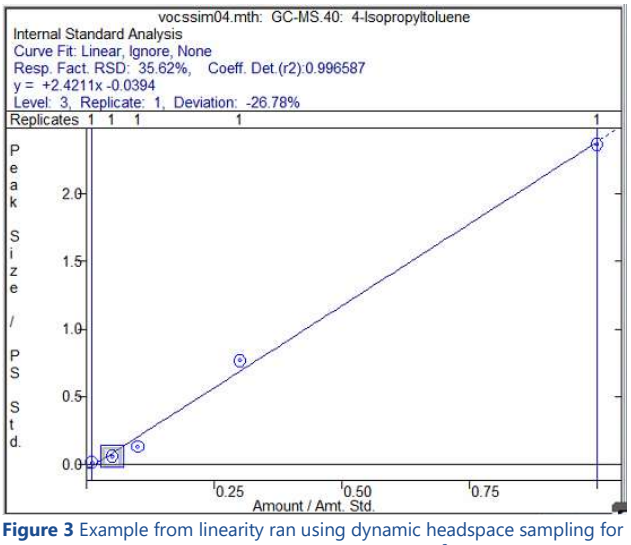

Figure 3 Example from linearity ran using dynamic headspace sampling for 4-Isopropyltoluene, showing correlation coefficient (r^2) from data review in MSWS.

Table 6 Recovery and precision results from spiked sample using static headspace

VOC	Recovery (%)	RSD (%)
2-Chlorotoluene	99.25	2.92
4-Isopropyltoluene	91.86	4.74
Benzene	108.06	0.96
1,3,5-trimethylbenzene	94.20	4.97
tert-Butylbenzene	95.33	3.73
Ethylbenzene	101.45	2.67
Isopropylbenzene	98.39	3.52
m-Xylene & p-Xylene	104.58	2.98
n-Propylbenzene	96.23	3.97
o-Xylene	101.84	2.57
sec-Butylbenzene	93.37	5.50

Table 7 Recovery and precision results from spiked sample using dynamic headspace

VOC	Recovery (%)	RSD (%)
2-Chlorotoluene	74.97	9.10
4-Isopropyltoluene	70.79	10.04
Benzene	124.26	17.44
1,3,5-trimethylbenzene	71.04	9.53
tert-Butylbenzene	70.17	9.99
Ethylbenzene	74.14	9.22
Isopropylbenzene	70.50	8.53
m-Xylene & p-Xylene	74.79	7.42
n-Propylbenzene	72.89	8.43
o-Xylene	69.34	8.11
sec-Butylbenzene	72.35	9.89

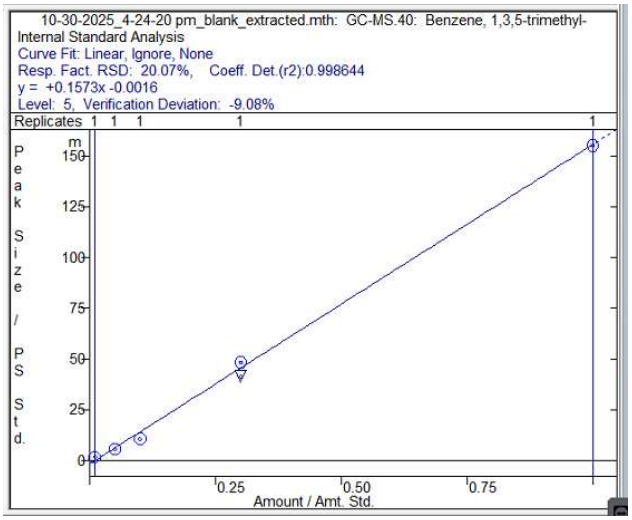

Figure 4 Example QC spiked sample ran using static headspace sampling for 1,3,5-trimethylbenzene from data review in MSWS, showing recovery of 90.92%.

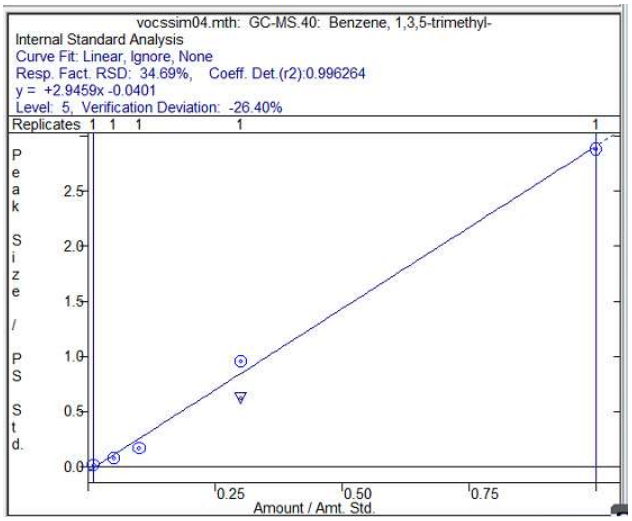

Figure 5 Example QC spiked sample ran using dynamic headspace sampling for 1,3,5-trimethylbenzene from data review in MSWS, showing recovery of 73.60%.

The system precision of each target analyte using static headspace was ≤ 6.8%. Using dynamic headspace the RSD of VOCs analyzed was ≤12.8%. EPA method 8260 states that the RSD should be ≤20% for each target analyte so using either static or dynamic headspace, this criteria was achieved.[3]

Table 6 shows the recovery and precision results for the selected compounds' QC spiked samples sampled using the static headspace (n=6). Recovery was determined to be between 87.79-108.06% with precision ≤5.5% across all VOCs analyzed.

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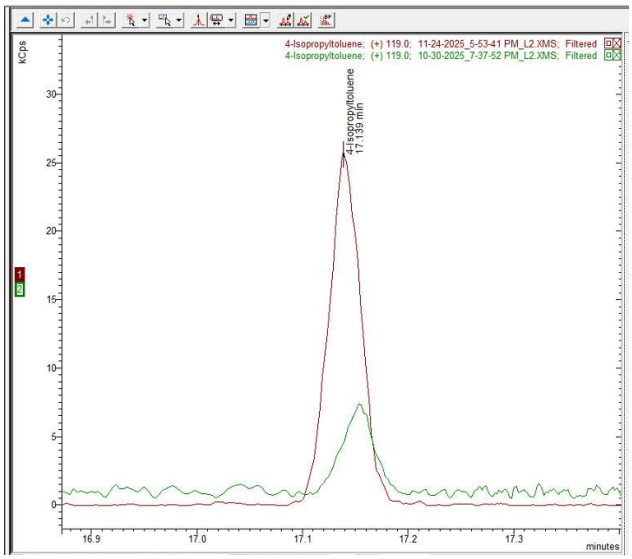

Figure 6 Example expanded TIC overlaid linearity sample (1 ppb) ran using dynamic headspace (red) and linearity sample (1 ppb) (green) ran using static headspace sampling for 4-Isopropyltoluene.

Table 7 shows the recovery and precision results for the selected compounds' QC spiked samples sampled using the dynamic headspace (n=5). Recovery was determined to be between 69.34-124.26% but almost all of analytes achieved a recovery in the 70-80% range. A precision of ≤17.44% was determined across all VOCs analyzed with the majority <11%.

Using either static or dynamic headspace the precision results for the QC spiked samples had an RSD ≤20% as specified in the method. In EPA method 8260, the suggested acceptance criteria for the recovery of the target analytes is 70-130%.[3] The QC spiked samples sampled using static headspace comfortably sit within this range. The QC spiked samples sampled using dynamic headspace are just outside this criteria on the lower end of the range with o-Xylene having a recovery of 69.34%.

Spiking the sample allows the performance of the analytical method to be evaluated to ensure that the method produces accurate and valid results. By spiking your sample you increase the concentration of the target analytes by a known amount and therefore will be able to determine if the added analytes are recovered. It is key to spike your sample at a concentration within your linearity range and sample volume is not increased. This allows calculations to be consistent and avoids introducing unknown effects. See our technical note on Recovery Spiked Sample.

Table 8 Signal comparison between static and dynamic headspace @1ppb

VOC	Signal Dynamic (KCps)	Signal Static (KCps)	Ratio Dynamic to Static
2-Chlorotoluene	17228.25	5330.09	3.12
4-Isopropyltoluene	25632.66	6600.69	3.88
Benzene	8205.87	4923.46	1.67
1,3,5-trimethylbenzene	29412.80	6526.78	4.51
tert-Butylbenzene	12469.76	2289.09	5.45
Ethylbenzene	24291.15	6370.08	3.81
Isopropylbenzene	16124.56	4106.94	3.93
m-Xylene & p-Xylene	58587.88	7865.19	7.45
n-Propylbenzene	21945.28	3948.41	5.56
o-Xylene	1996.31	7039.99	2.84
sec-Butylbenzene	14787.04	2634.52	5.61

Table 9 Signal to Noise comparison between static and dynamic headspace @1ppb

VOC	S/N Dynamic (KCps)	S/N Static (KCps)	Ratio Dynamic to Static
2-Chlorotoluene	257.04	23.98	10.72
4-Isopropyltoluene	2221.78	65.06	34.15
Benzene	108.56	28.32	3.83
1,3,5-trimethylbenzene	721.43	50.27	14.35
tert-Butylbenzene	1088.02	37.10	29.33
Ethylbenzene	522.56	24.04	21.73
Isopropylbenzene	856.78	58.66	14.61
m-Xylene & p-Xylene	1143.36	39.54	28.92
n-Propylbenzene	323.17	14.09	22.94
o-Xylene	478.04	32.23	14.83
sec-Butylbenzene	451.93	16.54	27.32

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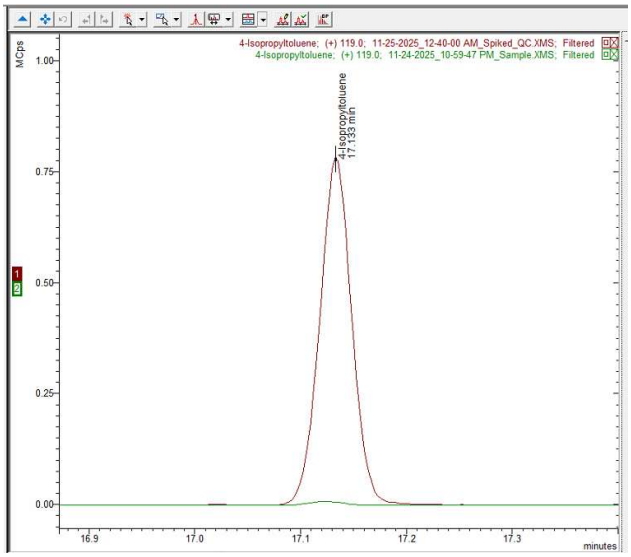

Figure 7 Example overlaid QC spiked sample (red) and water sample (green) ran using dynamic headspace sampling for 4-Isopropyltoluene.

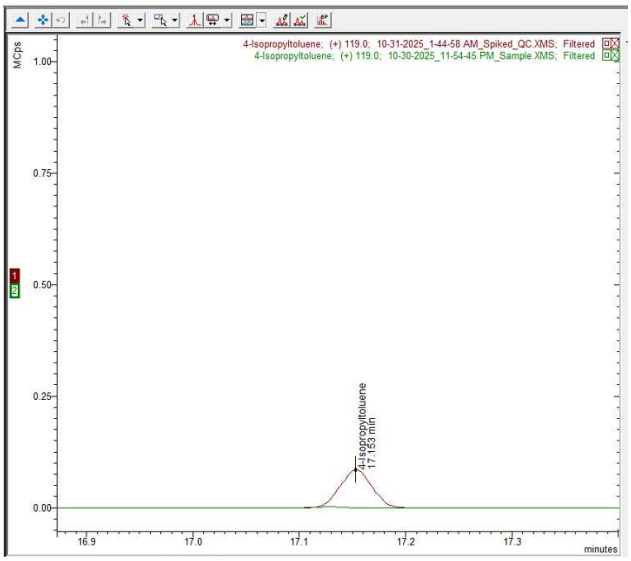

Figure 8 Example overlaid QC spiked sample (red) and water sample (green) ran using static headspace sampling for 4-Isopropyltoluene.

Figures 4 and 5 show an example of the data review in MSWS for 1,3,5-trimethylbenzene in a QC spiked sample using static and dynamic headspace respectively. The recovery is calculated using the verification deviation and then an average is calculated across the samples analyzed.

Figure 6 shows an example of a sample containing 1 ppb 4-Isopropyltoluene sampled using dynamic headspace (red) and static headspace (green) overlaid. The resulting peak for 4-Isopropyltoluene sampled using dynamic headspace is significantly larger.

Table 8 demonstrates the difference in signal between the static and dynamic sampling for the selected compounds. It is clear that the dynamic sampling results is a larger signal compared to static with the ratio of dynamic to static results ranging from 1.67-7.78 greater. Table 9 conveys the results of the signal to noise comparison between static and dynamic sampling for the selected compounds. Results show that the ratio between dynamic to static is 7.87-58.06 greater.

Figures 7 and 8 are examples of an overlaid QC spiked sample and a water sample for 4-Isopropyltoluene using dynamic and static headspace respectively. From these figures its can be seen that the signal for the QC spiked sample is much greater when using dynamic headspace. Using both static and dynamic headspace sampling, no VOCs were identified ≥LOQ.

Conclusion

A method was validated for VOC analysis in water samples with guidance from EPA method 8260 using the SCION Instruments HT3 headspace sampler with the SCION 8300 GC and SQ 8700 MS.

The HT3 headspace sampler was used in both static mode using a loop method and dynamic mode using a trap method. Both sampling methods achieved the LOQ of 1 ppb. QC spiked samples and an internal standard were utilized to confirm a good working method and account for variation in sampling respectively.

Good system precision, linearity and recovery results were achieved with most compounds meeting the acceptance criteria stated in EPA method 8260 when using either static or dynamic headspace sampling. Analyzed water samples were shown to have no VOCs ≥LOQ.

It was determined that by using dynamic headspace sampling a larger signal and signal to noise ratio was achieved than when using static headspace sampling.

SCION instruments recommends checking with local regulatory authorities to ensure all testing and reporting requirements are met, or contact the SCION applications team for assistance.

References

1. United States Environmental Protection Agency, https://www.epa.gov/east-palestine-oh-train-derailment/what-are-svocs-and-vocs, (accessed Feb 2026).
2. United States Environmental Protection Agency, https://www.epa.gov/indoor-air-quality-iaq/volatile-organic-compounds-impact-indoor-air-quality#Health_Effects, (accessed Feb 2026).
3. United States Environmental Protection Agency, https://www.epa.gov/esam/epa-method-8260d-sw-846-volatile-organic-compounds-gas-chromatography-mass-spectrometry-gcms, (date accessed Feb 2026).

Ordering Information

Ordering Information for the 8300 GC	
Part	**Part Number**
SCION SQ SELECT, w/8300, SSL-T21; 120V	SCIONSQ83SEL311
SCION SQ SELECT, w/8300, SSL-T21; 230V	SCIONSQ83SEL312
HT3 Headspace Autosampler 230V	SC149300100
HT3 Headspace Autosampler 110V	SC149300000
HT3 Dynamic Headspace Autosampler 230V	SC14930010S
HT3 Dynamic Headspace Autosampler 110V	SC14930000S
Suggested Consumables	
Part	**Part Number**
LINER 1177 78.5 L x 6.3 OD x 1.2MM ID S/SL STRAIGHT PK/5	41312108
SCION-624MS 30 m x 0.25 mm x 1.4 µm	SC32591
15% Graphite/85% Vespel Ferrule 1/16" with 0.4 mm hole pk/10	41312148
BTO Septa 9 mm, pk/50	CR298713
Vial, Crimp, Headspace, 20mL Clear Glass 22.5x75mm. 20mm Bevelled Edge. Rounded-Flat Bottom. 100pcs/pk.	41311008
20 mm Aluminium Crimp Cap with 20 mm Natural PTFE/White Silicone Septa 3mm Thick	41311010
Trap VOCARB 3000	SC145864003

For ordering info on the SCION 8500 GC, which offers greater functionality with the option of up to 4 detectors (including MS), please contact your local SCION sales representative.

For more information, please contact:

E: sales-eu@scioninstruments.com

W: www.scioninstruments.com