APPLICATION NOTE

Analysis of Volatile Organic Compounds (VOCs) in Water using HS-GC-MS (Versa): EPA Method 8260

AN189v1; April 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 and 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 sample 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 GC-MS system with guidance from EPA Method 5021.[3] See our technical note on EPA Method 5021 for more information.

Headspace (HS) 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 Versa headspace sampler combined with the SCION 8300 GC and SQ 8700 MS.

The Versa is a static headspace sampler so all data collected for this application note was acquired using a loop method.

Experimental

Instrument parameters for the GC-MS can be found in Table 1. Table 2 show the headspace sampler parameters for the Versa loop method. The system showed excellent specificity with compounds resolving well from one another and exhibiting good peak shape.

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

Table 2 Versa headspace sampler settings

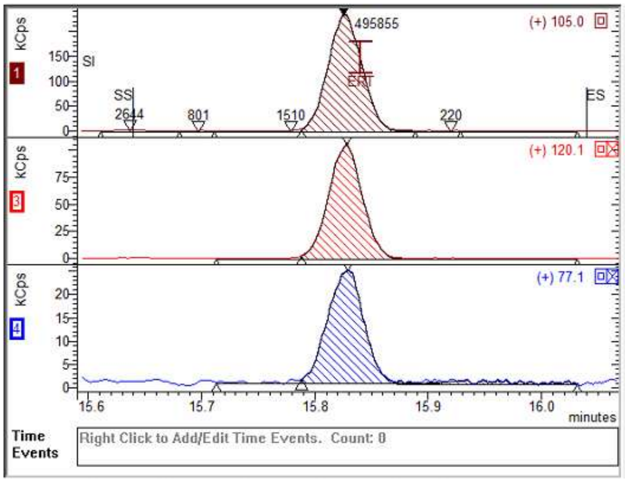
Variable	Value
GC Cycle Time	35.00 min
Valve Oven Temp.	120 °C
Transfer Line Temp.	120 °C
Platen/ Sample Temp.	70 °C
Platen Temp Equil. Time	1.00 min
Sample Equil. Time	1.00 min
Mixer	ON
Mixing Level	Medium
Mixing Time	10.00 min
Mixer Stabilize Time	0.50 min
Pressurize	5 PSIG
Pressurize Time	0.15 min
Pressure Equil. Time	0.20 min
Loop Fill Pressure	3 PSIG
Loop Fill Time	1.00 min
Inject Time	0.50 min
Software	Versa App

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 3. 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 methanol. 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 and IS (100 ppb) was added. QC spiked water samples were prepared with both 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.

To improve system sensitivity, Selective Ion Monitoring (SIM) was employed. Each VOC had a single quantifier ion and two qualifier ions selected (Table 3). 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 1,3,5-trimethylbenzene.



Right Click to Add/Edit Time Events. Count: 0

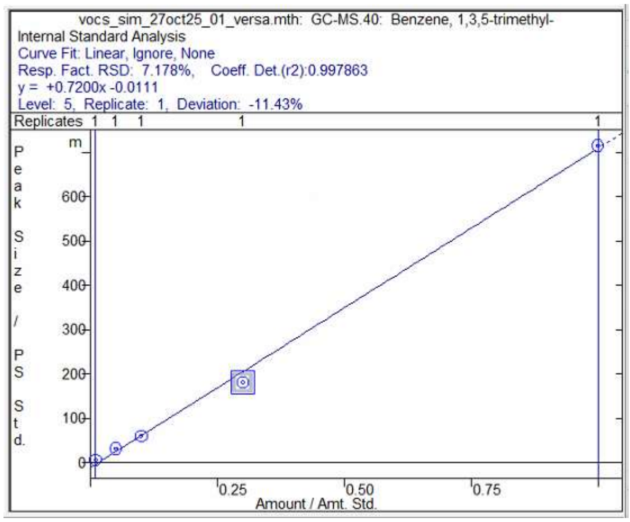
Figure 1 Example of 1,3,5-trimethylbenzene from linearity sample (30 ppb) ran using static headspace sampling showing quantifier (top) and qualifier plots (middle and bottom)

Results

Table 3 shows the correlation coefficient (r^2) and the RSD (%) for the system precision (n=6) for the selected VOCs. The r^2 values found were all >0.997 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 3 Correlation coefficient and system precision results for VOCs

VOC	Correlation coefficient (r^2)	RSD (%)	SIM Ions (Quantifier and qualifiers)
2-Chlorotoluene	0.9993	1.48	91, 89,126
4-Isopropyltoluene	0.9978	1.28	119, 91, 134
Benzene	0.9997	0.32	78, 77, 51
1,3,5-trimethylbenzene	0.9979	1.18	105, 120, 77
tert-Butylbenzene	0.9985	0.98	119, 91, 134
Ethylbenzene	0.9997	1.28	91, 106, 65
Isopropylbenzene	0.9991	0.72	105, 120, 79
m-Xylene & p-Xylene	0.9987	1.06	91, 106, 105
n-Propylbenzene	0.9989	1.09	91, 120, 65
o-Xylene	0.9992	1.34	91, 106, 105
sec-Butylbenzene	0.9985	1.11	105, 134, 91



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vocs_sim_27oct25_01_versa.mth:  GC-MS.40:  Benzene, 1,3,5-trimethyl-
Internal Standard Analysis
Curve Fit: Linear, Ignore, None
Resp. Fact. RSD:  7.178%,    Coeff. Det.(r2):0.997863
y =  +0.7200x -0.0111
Level:  5,  Replicate:  1,  Deviation:  -11.43%
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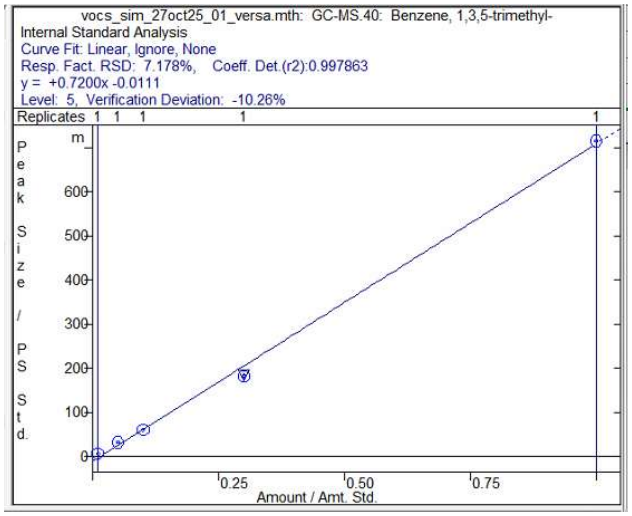
Figure 2 Example from linearity ran using static headspace sampling for 1,3,5-tirmethylbenzene (30 ppb) showing correlation coefficient (r^2) from data review in MSWS.

An example of the calibration curve calculated for 1,3,5-trimethyllbenzene can be seen in Figure 2 which shows the correlation coefficient as 0.9979.

The system precision of each target analyte was ≤2.68%. EPA method 8260 states that the RSD should be ≤20% for each target analyte therefore this was an excellent result.[3]



vocs_sim_27oct25_01_versa.mth: GC-MS.40: Benzene, 1,3,5-trimethyl-
Internal Standard Analysis
Curve Fit: Linear, Ignore, None
Resp. Fact. RSD: 7.178%, Coeff. Det.(r2):0.997863
y = +0.7200x -0.0111
Level: 5, Verification Deviation: -10.26%

Figure 3 Example QC spiked sample for 1,3,5-trimethylbenzene from data review in MSWS, showing recovery of 89.74%.

Figure 3 shows an example of the data review in MSWS for 1,3,5-trimethylbenzene in a QC spiked sample. The recovery is calculated using the verification deviation and then an average was calculated across the samples analyzed.

Table 4 shows the average recovery and precision results for the selected compounds' QC spiked samples (n=6). Recovery was determined to be between 85.67-125.19% with precision ≤2.62% across all VOCs.

The precision results for the QC spiked samples had an RSD (%) much lower than ≤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 comfortably sit within this range.

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 for more information.

Table 4 Average recovery and precision results from spiked samples

VOC	Recovery (%)	RSD (%)
2-Chlorotoluene	94.58	1.50
4-Isopropyltoluene	86.46	1.25
Benzene	96.83	0.32
1,3,5-trimethylbenzene	88.05	1.12
tert-Butylbenzene	89.05	1.17
Ethylbenzene	95.03	1.25
Isopropylbenzene	91.36	0.72
m-Xylene & p-Xylene	91.67	1.04
n-Propylbenzene	90.77	1.13
o-Xylene	96.18	1.32
sec-Butylbenzene	86.47	1.07

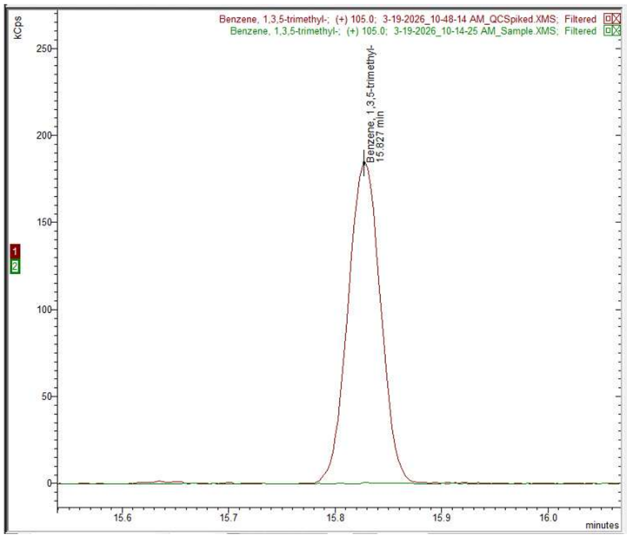


Figure 4 Example overlaid QC spiked sample (red) and water sample (green) for 1,3,5-trimethylbenzene.

Figure 4 shows an example of an overlaid QC spiked sample and a water sample for 1,3,5-trimethylbenzene. From the samples analyzed, VOCs were found to be less than the LOQ.

Conclusion

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

A LOQ of 1 ppb was achieved and QC spiked samples and an internal standard were utilized to confirm a good working method and account for variation in sampling respectively.

Excellent system precision, linearity and recovery results were achieved with all compounds meeting the acceptance criteria stated in EPA method 8260. In the analyzed water samples, VOCs were found to be less than the LOQ.

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
Versa Static Headspace Vial Sampler 230V	SC150800200
Versa Static Headspace Vial Sampler 110V	SC150800100

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

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