Using Alternative Carrier Gas for US EPA 524.3

Application Note

Abstract

Due to regional shortages and increasing costs of helium, the preferred carrier gas in gas chromatography, alternative carrier gas options are in ever increasing demand. This study will evaluate the Teledyne Tekmar Atomx Multimatrix Autosampler in conjunction with a Thermo Scientific TRACE 1310 / ISQ GC/MS, while employing hydrogen carrier gas and nitrogen purge gas to perform USEPA Method 524.3. A working calibration curve, precision and accuracy study (% Diff) and 4-bromofluorobenzene (BFB) tune evaluation will be presented to determine the validity of alternative gases to helium for EPA Method 524.3.

Introduction

The USEPA developed Method 524.3, Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry, to determine volatile organic compounds (VOCs) in drinking water to keep the public safe.[1] Helium is the preferred GC carrier gas, as well as the preferred purge gas. Unfortunately, helium is a nonrenewable gas which has issues with limited supply and increasing costs. Conversely, alternative carrier gases such as hydrogen and nitrogen are renewable and can be derived from generators. Many laboratories are making the conscious effort to cut costs and shift to helium-free instruments and methodologies, but must also be able to produce reliable results.

This study will evaluate using renewable gas sources - hydrogen for the carrier gas and nitrogen for the purge gas - as substitutes for helium in USEPA Method 524.3. Calibration standards from 0.5 ppb to 50.0 ppb will be evaluated per the method criteria. A calibration curve will be used to determine if the regression value (r^2) is ≥ 0.995. A precision and accuracy test will be evaluated from the midpoint of the calibration at 10.0 ppb.

Teledyne Tekmar's Atomx is a VOC sample prep system that integrates a purge and trap concentrator with a multimatrix autosampler. It employs an 80-position vial autosampler capable of running water, soil and automated methanol extractions. Also, it has the capability of prepping samples ahead, while the previous sample is being analyzed by the GC, allowing for faster analysis time. The Atomx has the ability to automatically add Internal Standards (IS) and Surrogate Sample (SS) from three separate standard addition vessels, with volume increments of 1.0 to 20.0 µL. These features, along with the capability of using alternatives gases, can increase throughput and cost savings.

Experimental-Instrument Conditions

For this study, the Atomx was coupled to a Thermo Scientific TRACE 1310 GC/MS with ISQ MS Detector. The analytical trap employed was a #9 (proprietary) trap. Hydrogen was used as the carrier gas and nitrogen as the purge gas. The GC was equipped with an Rtx®-VMS 20 m x 0.18 mm x 1 µm column. The GC/MS parameters are outlined in Tables I and II. Table III outlines the Atomx conditions.

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Table I: GC Parameters	
GC:	Thermo Scientific TRACE 1310
Column	Rtx®-VMS 20m x 0.18 mm ID x 1.0 µm
Oven Program:	35 °C for 3 min; 14 °C/min to 100 °C; 25 to 210; hold for 2 min
Inlet:	200 °C
Column Flow	0.8 mL/min
Gas:	Hydrogen
Split:	60:1
Pressure:	5 psi

Table II: MS Parameters	
MS:	ISQ
Source:	280 °C
Solvent Delay:	0.8 min
Scan Range:	35 - 260
Scans:	0.15 sec
Emission Current:	40 µA
Detector Gain:	3.00 e5
Chrom Filter Width:	2.0 sec
MS Transfer Line:	230 °C

Table III: Atomx 524.3 Water Parameters			
Variable	Value	Variable	Value
Valve Oven Temp	140 °C	Dry Purge Flow	100 mL/min
Transfer Line Temp	140 °C	Dry Purge Temp	20 °C
Sample Mount Temp	90 °C	Methanol Needle Rinse	Off
Water Heater Temp	90 °C	Methanol Needle Rinse Volume	3.0 mL
Sample Vial Temp	20 °C	Water Needle Rinse Volume	7.0 mL
Sample Equilibrate Time	0.00 min	Sweep Needle Time	0.25 min
Soil Valve Temp	50 °C	Desorb Preheat Time	245 °C
Standby Flow	10 mL/min	GC Start Signal	Start of Desorb
Purge Ready Temp	40 °C	Desorb Time	2 min
Condensate Ready Temp	45 °C	Drain Flow	300 mL/min
Presweep Time	0.25 min	Desorb Temp	250 °C
Prime Sample Fill Volume	3.0 mL	Methanol Glass rinse	Off
Sample Volume	5.0 mL	Number of Methanol Glass Rinses	1
Sweep Sample Time	0.25 min	Methanol Glass Rinse Volume	3.0 mL
Sweep Sample Flow	100 mL/min	Number of Water Bake Rinses	1
Sparge Vessel Heater	OFF	Water Bake Rinse Volume	7.0 mL
Sparge Vessel Temp	20 °C	Bake Rinse Sweep Time	0.25 min
Prepurge Time	0.00 min	Bake Rinse Sweep Flow	100 mL/min
Prepurge Flow	0 mL/min	Bake Rinse Drain Time	0.40 min
Purge Time	8.00 min	Bake Time	2.00 min
Purge Flow	55 mL/min	Bake Flow	400 mL/min
Purge Temp	20 °C	Bake Temp	280 °C
Condensate Purge Temp	20 °C	Condensate Bake Temp	200 °C
Dry Purge Time	0.5 min		

(Parameters highlighted in yellow were not used.)

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Results and Discussion

A 50.0 ppm stock standard was brought to volume with methanol. This stock was diluted in reagent water which was made with 0.625g/L ascorbic acid and 5g/L maleic acid, as required by Method 524.3, to make the calibration standards from 0.5 ppb to 50 ppb. These standards were placed into headspace free 40 mL VOA vials.

Internal Standard (IS) and Surrogate standards (SS) were made in methanol at a concentration of 50.0 ppm. IS/SS were dispensed by the internal standard vessels in 5.0 µL aliquots for a final concentration of 50.0 ppb.

Thermo TraceFinder software was used to process the calibration data for each compound using extracted ion mode. The primary and secondary characteristic ions are prescribed in Method 524.3. The method states that a linear or quadratic regression line can be used and weighting can be applied. To calculate the R^2 value in this study, quadratic regression and weighting was used (see Table IV).

Precision and accuracy data was determined by running eight replicates (n=8) at 10.0 ppb standard which is the midpoint calibration standard (see Table IV and Figure 1). The precision is represented by Relative Standard Deviation (%RSD). The accuracy is evaluated by the Average % Difference. This value represents the theoretical concentration compared to the actual concentrations calculated off the calibration curve.

Also, each calibration standard was determined for accuracy. All calibrations points are required to be ±30.0% except for the lowest calibration standard; in this case it is 0.5 ppb which can be ±50.0% (see Table V).

To verify that the mass spectrometer is properly tuned, 4-bromofluorobenzene (BFB) ion ratios must meet the criteria stated in USEPA 524.3. This was evaluated by running a blank spiked with BFB surrogate standard and analyzing the ion ratios. These criteria were achieved under this method, as evidenced in Figure 2.

Table IV: Calibration Curve, Precision & Accuracy Data for 10 ppb Standard

Compound	R^2 Value	Precision Data of 10 ppb n=8		
		Avg. Conc.	Avg. % Diff	% RSD
1,1,1,2-Tetrachloroethane	0.9986	9.187	-8.13	5.91
1,1,1-Trichloroethane	0.9985	9.695	-3.05	7.01
1,1,2,2-Tetracholorethane	0.9995	10.649	6.49	12
1,1,2-Trichloroethane	0.9979	9.585	-4.15	12.92
1,1-Dichloroethane	0.9971	9.196	-8.04	3.3
1,1-Dichloroethene	0.999	9.633	-3.67	8.99
1,1-Dichloropropene	0.9989	9.759	-2.41	8.41
1,2,3 Trichlorobenzene	0.9991	9.724	-2.76	6.28
1,2,3-Trichloropropane	0.9994	10.164	1.64	6.97
1,2,3-Trimethyl-Benzene	0.9979	8.438	-15.62	13.66
1,2,4-Trichlorobenzene	0.9979	10.591	5.91	7.44
1,2,4-Trimethylbenzene	0.9994	9.84	-1.6	5.62
1,2-Dibromo-3-Chloropropane (DBCP)	0.9991	10.249	2.49	14.14

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Compound	R^2 Value	Precision Data of 10 ppb n=8		
		Avg. Conc.	Avg. % Diff	% RSD
1,2-Dibromoethane (EDB)	0.9975	9.041	-9.59	9.76
1,2-Dichlorobenzene	0.9993	9.969	-0.31	9.51
1,2-Dichloroethane	0.9994	9.685	-3.15	7.94
1,2-Dichloropropane	0.9989	9.59	-4.1	5.9
1,3,5-Trimethylbenzene	0.9995	9.99	-0.1	7.26
1,3-Butadiene	0.9972	9.226	-7.74	12.44
1,3-Dichlorobenzene	0.9985	9.699	-3.01	7.5
1,3-Dichloropropane	0.9984	9.52	-4.8	12.25
1,4-Dichlorobenzene	0.999	9.697	-3.03	4.82
2-Chlorotoluene	0.9995	10.31	3.1	9.03
4-Chlorotoluene	0.9996	9.942	-0.58	8.73
Allyl Chloride	0.9974	9.637	-3.63	12.82
Benzene	0.9992	9.687	-3.13	7.28
Brodichloromethane	0.999	9.934	-0.66	6.91
Bromobenzene	0.9993	10.342	3.42	10.99
Bromochloromethane	0.9986	9.068	-9.32	6.51
Bromoform	0.9963	9.226	-7.74	16.13
Butane, 1-chloro-	0.9986	9.455	-5.45	4.53
Carbon Disulfide	0.9991	9.699	-3.01	9.88
Carbon Tetrachloride	0.9974	9.566	-4.34	6.98
Chlorobenzene	0.9995	9.435	-5.65	4.45
Chloroform	0.9993	9.589	-4.11	7.84
Chloromethane	0.9978	9.755	-2.45	18.39
cis-1,3-Dichloropropene	0.9974	9.882	-1.18	7.09
Dibromochloromethane	0.9978	8.881	-11.19	8.43
Dichlorodifluoromethane	0.9973	8.705	-12.95	12.06
Diethyl Ether	0.998	9.683	-3.17	8.27
Difluorochloromethane	0.9966	8.966	-10.34	15.55
Diisopropyl Ether	0.9996	9.168	-8.32	6.93
Ethylbenzene	0.9984	9.273	-7.27	8.61
Ethylmethacrylate	0.998	9.354	-6.46	12.8

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Table IV: Calibration Curve, Precision & Accuracy Data for 10 ppb Standard				
Compound	R^2 Value	Precision Data of 10 ppb n=8		
		Avg. Conc.	Avg. % Diff	% RSD
Ethyl-tert-Butyl-Ether (ETBE)	0.9988	9.137	-8.63	6.98
Hexachlorobutadiene	0.9996	9.915	-0.85	9.67
Isopropylbenzene	0.999	9.261	-7.39	13.4
m,p-Xylene	0.9984	18.694	-6.53	9.46
Methane, iodo-	0.9961	5.83	-41.7	13.48
Methyl Acetate	0.9976	9.078	-9.22	11.16
Methylene Chloride	0.9993	9.514	-4.86	9.45
Methyl-tert-butyl-Ether (MTBE)	0.9991	9.904	-0.96	8.16
n-Propylbenzene	0.9996	10.124	1.24	9.34
Naphthalene	0.9995	10.022	0.22	8.34
n-Butylbenzene	0.9993	10.458	4.58	13.52
o-Xylene	0.9976	9.274	-7.26	12.76
Pentachloroethane	0.9995	9.86	-1.4	6.04
p-Isopropyltoluene	0.9994	9.714	-2.86	6.27
sec-Butylbenzene	0.9996	9.919	-0.81	6.55
Styrene	0.9996	9.397	-6.03	13.42
tert-Butyl Alcohol	0.9988	9.138	-8.62	6.98
tert-Butylbenzene	0.9998	9.761	-2.39	7.02
Tetrachloroethylene	0.9982	8.605	-13.95	9.89
Tetrahydrofuran	0.9984	9.241	-7.59	6.9
Toluene	0.9979	9.352	-6.48	13.01
trans 1,3-Dichloropropene	0.9973	9.535	-4.65	12.3
trans-1,2-Dichloroethene	0.9988	9.462	-5.38	8.47
trans-1,4-Dichloro-2-butene	0.9998	10.496	4.96	12.13
Trichloroethene	0.9994	9.537	-4.63	9.95
Trichloroethylene	0.9988	9.629	-3.71	3.23
Trichlorofluoromethane	0.998	9.851	-1.49	10.01
Vinyl Chloride	0.9972	9.461	-5.39	10.94

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Table V: Accuracy Data for Each Calibration Standard									
	0.5	1	2	5	10	20	30	40	50
1,1,1,2-Tetrachloroethane	1.81	-7.04	6.5	1.46	1.2	-6.85	3.8	-2.8	1.94
1,1,1-Trichloroethane	-1.94	4.97	-2.37	0.93	5.46	-2.49	-6.76	0.88	3.16
1,1,2,2-Tetracholorethane	0.7	-1.3	0.38	-1.58	-0.62	-2.37	9.41	-2.99	-1.82
1,1,2-Trichloroethane	0.76	-1.8	0.76	-0.72	0.07	-1.29	4.36	1.26	-0.93
1,1-Dichloroethane	-9.08	0.79	2.49	-1.26	12.94	-2.17	-5.4	-0.12	1.73
1,1-Dichloroethene	-10.79	-0.77	0.91	5.07	11.91	-0.2	-9.54	1.53	1.77
1,1-Dichloropropene	-9.84	0.95	1.36	2.77	10.03	-1.76	-4.8	-0.59	1.82
1,2,3 Trichlorobenzene	1.59	-4.93	4.46	-2.56	-0.25	-2.71	8.66	-1.67	-2.58
1,2,3-Trichloropropane	4.57	0.03	0.63	-3.36	-1.69	-8.14	11.69	-2.99	-0.85
1,2,3-Trimethyl-Benzene	-0.75	-0.61	4.52	0.03	1.11	-4.79	-5.47	6.22	-0.27
1,2,4-Trichlorobenzene	2.62	-7.71	3.87	5.34	-5.7	-2.68	7.19	-1.79	-1.19
1,2,4-Trimethylbenzene	-0.39	0.93	0.29	-2.8	3.71	-3.95	3.62	-1.72	0.29
1,2-Dibromo-3-chloropropane (DBCP)	1.07	0.94	-8.01	3.75	1.82	-4.65	10.45	-5.08	-0.6
1,2-Dibromoethane (EDB)	-0.41	1.7	-1.34	-1.51	0.75	-2.19	5.53	-1.53	-1.07
1,2-Dichlorobenzene	0.94	-3.64	3.08	1.75	1.61	-10.91	9.42	-2.09	-0.24
1,2-Dichloroethane	-0.68	1.72	-0.24	-3.74	7.88	-5.01	-1.02	-1.62	2.72
1,2-Dichloropropane	-1.54	2.06	2.7	-2.14	3.92	-5.13	-2.81	-0.12	3.05
1,3,5-Trimethylbenzene	0.47	-1.98	2.06	0.52	-0.02	-4.87	6.13	-2.55	0.19
1,3-Butadiene	-5.19	10.18	1.36	-0.97	0.78	-2.76	-13.53	2.45	8.12
1,3-Dichlorobenzene	2.2	-4.37	1.07	-5.62	5.03	-2.97	9.06	-1.93	-2.77
1,3-Dichloropropane	-1.9	3.56	0.25	2.28	-1.53	-5.68	3.12	-2.13	2.03
1,4-Dichlorobenzene	1.71	-5.38	4.74	-2.6	1.28	-4.53	8.12	-1.88	-1.56
2-Chlorotoluene	0.43	-2.57	4.03	-0.18	-2.8	-3.48	7.84	-2.37	-0.93
4-Chlorotoluene	-0.05	-0.83	2.77	-2.41	1.08	-4.63	6.23	-1.13	-1.04
Allyl Chloride	-11.31	26.51	-4.45	-7.38	2.29	-6.93	-7.63	4.71	4.52
Benzene	-1.99	3.56	1.41	-2.27	4.76	-4.8	-4.11	-0.4	3.83
Brodichloromethane	-2.66	4.97	2.11	-4.16	3.65	-4.73	-2.07	1.6	1.28
Bromobenzene	0.69	-1.56	1.31	-2.75	-0.1	-2.94	10.16	-2.5	-2.47
Bromochloromethane	-5.85	12.94	-0.8	-5.61	5.95	-2	-8.9	-2.23	6.52
Bromoform	-3.17	7.32	-0.03	-4.56	-0.78	-2.02	5.05	-0.71	-1.12
Butane, 1-chloro-	-3.29	6.89	0.54	-3.76	4.41	-4.19	-4.59	1.38	2.59

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Table V: Accuracy Data for Each Calibration Standard									
	0.5	1	2	5	10	20	30	40	50
Carbon Disulfide	-1.88	4.24	-1.3	-0.52	6.3	-3.82	-9.1	1.7	4.37
Carbon Tetrachloride	6.57	-12.99	-2.62	0.12	12.7	-0.62	-4.95	2.33	-0.71
Chlorobenzene	0.86	-3.44	3.45	0.79	-1.35	-1.45	2.31	-2.19	1.02
Chloroform	-2.99	3.95	5.4	-3.81	5.28	-6.82	-6.59	-0.32	5.88
Chloromethane	-34.89	23.21	-2.01	10.82	11.17	0.37	-14.56	3.44	2.52
cis-1,3-Dichloropropene	-2.12	4.59	0.12	-3.59	5.33	-3.91	-2.46	-0.02	2.08
Dibromochloromethane	0.34	-2.48	5.17	-4.84	2.36	-3.62	3.94	0.34	-1.29
Dichlorodifluoromethane	-28.54	2.52	-7.7	3.81	10.07	-1.55	-15.34	-1.19	10.54
Diethyl ether	-1.29	2.79	0.12	-2.17	2.85	-0.9	-3.67	0.97	1.31
Difluorochloromethane	-12.2	20.7	-1.31	2.36	5.07	-1.04	-15.75	8.05	3.17
Diisopropyl ether	-1.12	4.78	-4.84	-4.08	8.71	-3.06	-1.32	0.63	0.27
Ethylbenzene	-0.66	0.46	0.95	3.19	-0.3	-4.92	0	-1.03	2.33
Ethylmethacrylate	-1.15	3.17	-1.92	1.01	-0.5	-3.22	4.14	-1.7	0.15
Ethyl-tert-butyl-Ether (ETBE)	-0.7	2.45	-1.77	-2.87	5.25	-2.56	-0.13	-0.15	0.47
Hexachlorobutadiene	6.77	-15.18	1.97	1.12	2.99	0.73	4.1	-1.21	-1.77
Isopropylbenzene	0	-1.46	2.39	3	-1.93	-3.07	0	-0.28	1.36
m,p-Xylene	-0.87	0.1	3.16	2.08	-1.89	-4	0.04	-0.66	2.04
Methane, iodo-	3.01	0.5	-11.79	-9.37	8.13	9.21	-1.31	5.57	-6.28
Methyl acetate	-24.74	-12.23	0.62	-4.25	7.03	-0.85	-7	4.72	-0.3
Methylene Chloride	-3.77	8.11	0.86	-6.47	5.27	-2.64	-6.58	3.93	1.3
Methyl-tert-Butyl-Ether (MTBE)	-2.67	6.06	-0.24	-4.05	3.51	-2.12	-2.85	1.17	1.18
n-Propylbenzene	-0.57	0.47	1.83	-0.56	-0.48	-5.26	6.37	-0.85	-0.96
Naphthalene	1.14	2.98	1.54	-0.69	1.77	-6.94	11.25	-5.1	-0.17
n-Butylbenzene	0.93	-3.47	3.23	0	1.25	-3.91	3.36	-2.83	1.43
o-Xylene	-2.57	4.78	0.69	1.48	-0.04	-5.01	-1.7	-1.07	3.46
Pentachloroethane	1.02	-1.49	-1.77	0.96	2.38	-4.31	6.5	-3.79	0.41
p-Isopropyltoluene	0.64	-1.22	-0.24	0.1	0.75	-3.44	6.04	-1.82	-0.85
sec-Butylbenzene	1.11	-3.65	2.16	2.72	-0.91	-3.95	4.37	-3.2	1.33
Styrene	0.41	-3.5	5.25	2.03	-1.89	-4.26	2.93	-4.04	3.07
tert-Butyl Alcohol	-0.7	2.47	-1.82	-2.76	5.21	-2.52	-0.29	-0.11	0.51
tert-Butylbenzene	-1.9	3.12	1.61	0.53	0.55	-8.67	5.94	-3.5	2.31
Tetrachloroethylene	-1	1.93	0.39	-1.88	5.33	-5.38	-3.35	3.67	0.29

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Table V: Accuracy Data for Each Calibration Standard									
	0.5	1	2	5	10	20	30	40	50
Tetrahydrofuran	-1.39	5.67	-5.13	-5.48	8.72	-1.87	-1.28	1.3	-0.58
Toluene	-0.53	1.2	-1.35	3.45	0.35	-4	0.24	-1.57	2.22
trans 1,3-dichloropropene	-1.55	2.28	2.1	-0.22	-0.49	-4.17	1.37	0.24	0.48
trans-1,2-Dichloroethene	-1.51	4.11	-2.5	-1.03	6.27	-1.84	-8.14	1.13	3.51
trans-1,4-dichloro-2-butene	-0.76	2.13	-0.04	-2.08	-2.84	-3.3	9.87	1.24	-4.57
Trichloroethene	9.61	-14.5	-4.82	5.99	9.12	-3.53	-2.71	-0.82	1.65
Trichloroethylene	1.96	-6.56	6.36	-4.46	3.76	0.56	-1.17	-2.23	1.77
Trichlorofluoromethane	-2.04	4.13	0.09	-1.45	4.85	-1.23	-12.21	3.64	4.32
Vinyl Chloride	-2.1	3.93	0.59	-0.42	4.22	-3.68	-9.27	0.94	6

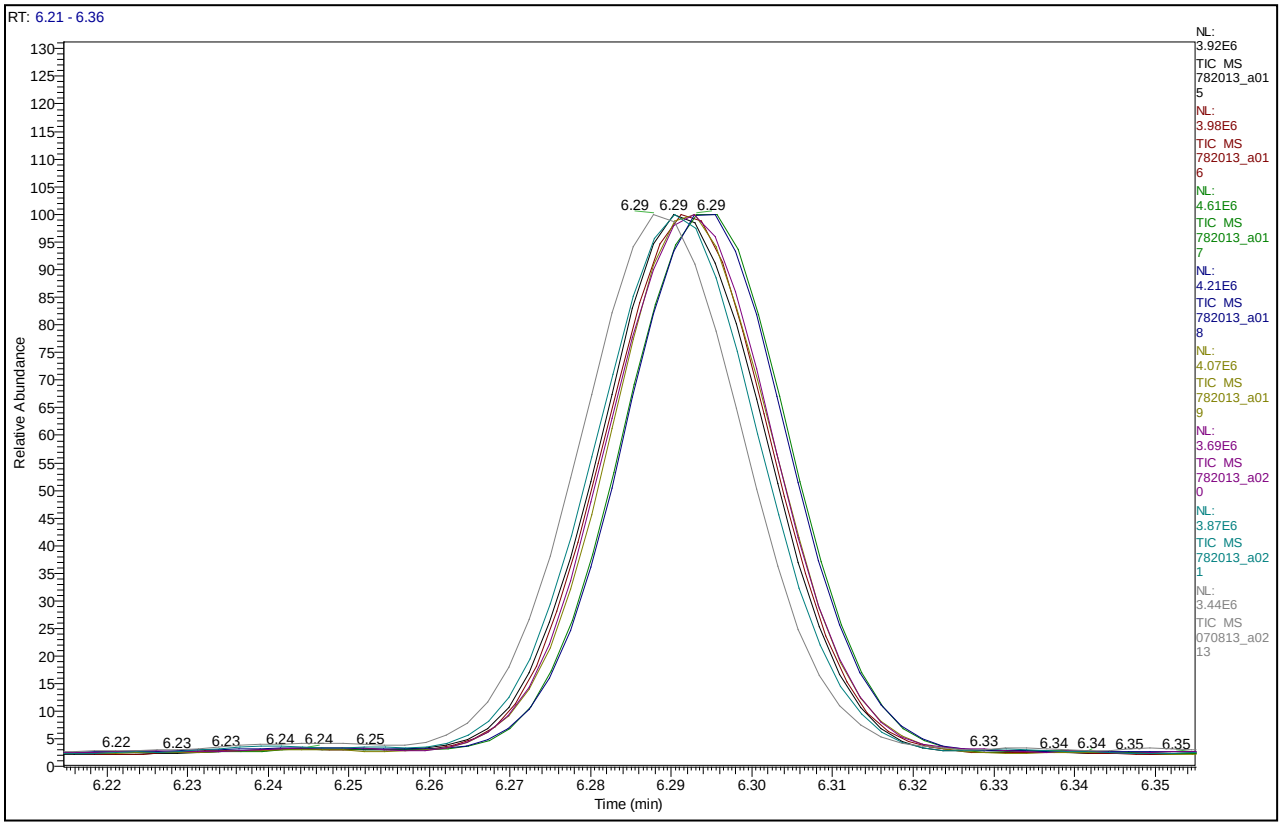

Figure 1: TIC of 10 ppb Toluene in Eight Repetitions for Precision and Accuracy.

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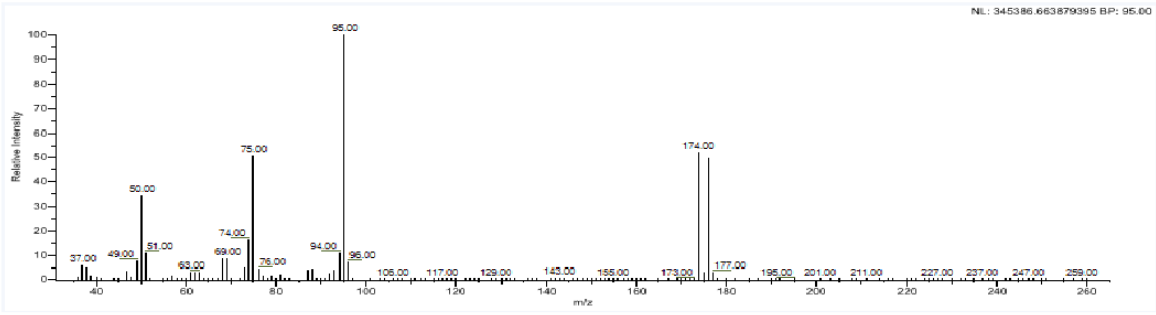

m/z	Ion Abundance Criteria	% Relative Abundance	Pass/fail
95	equals 100% of Base Peak	100.0	Pass
96	greater than or equal to 5% AND less than or equal to 9% of m/z 95	7.1	Pass
173	less than 2% of m/z 174	0.8	Pass
174	greater than 50% of m/z 95	52.1	Pass
175	greater than or equal to 5% AND less than or equal to 9% of m/z 174	6.0	Pass
176	greater than 95% AND less than 105% of m/z 174	95.2	Pass
177	greater than or equal to 5% AND less than or equal to 10% of m/z 176	5.8	Pass

Figure 2: Passing BFB Tune using Hydrogen Carrier Gas.

Conclusions

The Atomx Multimatrix Autosampler, used in conjunction with a Thermo Scientific TRACE 1310 / ISQ GC/MS, was able to pass all performance requirements of USEPA Method 524.3 using hydrogen carrier gas and nitrogen purge gas. This is both a budget and environmentally friendlier way to analyze VOCs. As this study demonstrates, these readily available gases are viable alternatives for this analysis, although the method itself must be further evaluated and updated to allow for their use.

Acknowledgement

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References

1. *Method 524.3. Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry*; U.S. EPA, Office of Ground Water and Drinking Water, U.S. Government Printing Office: Washington, D.C., 2009.

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