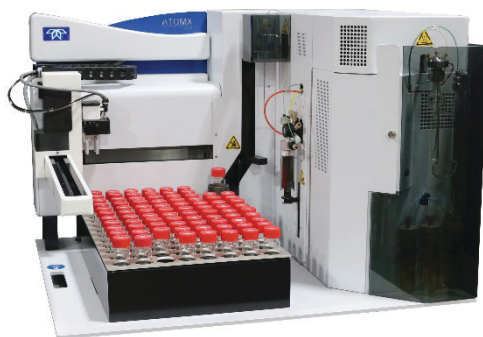


# US EPA Method 8260 with the Tekmar Atomx XYZ P&T System and Agilent 7890B GC/5977A MS



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## Abstract

US EPA Method 8260 in conjunction with Methods 5030 and 5035 was used to determine the concentration of volatile organic compounds (VOCs) in water and soil matrices. The Teledyne Tekmar Atomx XYZ purge and trap (P&T) system along with an Agilent 7890B Gas Chromatograph (GC)/5977A Mass Spectrometer (MS) was used to create a working linear calibration curve and method detection limits (MDLs) for target compounds.

## Introduction

The Atomx XYZ is Teledyne Tekmar's most advanced P&T system and is based on the time-tested Atomx instrument platform. The concentrator's efficient trap cooling design reduces sample cycle time by as much as 14% over the previous model. Combined with its 84-position soil and water autosampler, the result is more samples tested per 12-hour period. An innovative moisture control system (MCS) improves water vapor removal by as much as 60%, thereby reducing peak interference and increasing GC column lifespan. In addition to other refinements, the Atomx XYZ incorporates a precision-machined valve manifold block to reduce potential leak sources and ensure the system is both reliable and robust.

## Experimental

### Sample Preparation

Calibration working standards in concentrations of 50 ppm and 100 ppm were prepared in methanol from the following Restek® standards: 8260B MegaMix®, 8260B Acetate, California Oxygenates, VOA (Ketones), 502.2 Calibration Mix, 2-Chloroethyl Vinyl Ether, and Hexachloroethane. In total, the standards contained 91 compounds.

Soil and water calibration curves were prepared from 0.5 ppb to 200 ppb for all compounds. The 50 ppm calibration working standard was diluted to create 0.5, 2, 5, 10, and 20 ppb concentrations, while the 100 ppm calibration working standard was diluted to create 50, 100, and 200 ppb concentrations. The relative response factor (RF) was calculated for each compound using one of four internal standards: Pentafluorobenzene, 1,4-Difluorobenzene, Chlorobenzene-d5, and 1,4-Dichlorobenzene-d4. Surrogate standards consisted of: Dibromofluoromethane,

1,2-Dichloroethane-d4, Toluene-d8, and Bromofluorobenzene. Internal and surrogate standards were prepared in methanol from Restek standards at a concentration of 25 ppm, after which 5 µL was then mixed with each 5 mL sample for a resulting concentration of 25 ppb.

Seven 0.5 ppb (water method) and seven 1 ppb (soil method) standards were prepared to calculate the method detection limit (MDL), accuracy, and precision calculations. All calibration and MDL standards were analyzed with the Atomx XYZ conditions in Table 1 (water method) and Table 2 (soil method). GC-MS conditions are shown in Table 3.

## Experimental Instrument Conditions

**Table 1.** Teledyne Tekmar Atomx XYZ Water Method Conditions.

Standby	Variable	Desorb	Variable
Valve Oven Temp	140 °C	Methanol Needle Rinse	Off
Transfer Line Temp	140 °C	Methanol Needle Rinse Volume	0.00 mL
Sample Mount Temp	90 °C	Water Needle Rinse Volume	7.00 mL
Water Heater Temp	90 °C	Sweep Needle Time	0.25 min
Sample Vial Temp	20 °C	Desorb Preheat Temp	245 °C
Soil Valve Temp	100 °C	GC Start Signal	Begin Desorb
Standby Flow	10 mL/min	Desorb Time	2.00 min
MCS Ready Temp	45 °C	Drain Flow	300 mL/min
Purge Ready Temp	40 °C	Desorb Temp	250 °C
Purge	Variable	Bake	Variable
Sample Equilibrate Time	0.00 min	Methanol Glass Rinse	Off
Pre-sweep Time	0.25 min	Number of Methanol Glass Rinses	0
Prime Sample Fill Volume	3.00 mL	Methanol Glass Rinse Volume	0.00 mL
Sample Volume	5.00 mL	Water Bake Rinses	1
Sweep Sample Time	0.25 min	Water Bake Rinse Volume	7.00 mL
Sweep Sample Flow	100 mL/min	Bake Rinse Sweep Time	0.25 min
Spurge Vessel Heater	Off	Bake Rinse Sweep Flow	100 mL/min
Spurge Vessel Temp	20 °C	Bake Rinse Drain Time	0.40 min
Pre-purge Time	0.00 min	Bake Time	2.00 min
Pre-purge Flow	0 mL/min	Bake Flow	200 mL/min
Purge Time	11.00 min	Bake Temp	260 °C
Purge Flow	40 mL/min	MCS Bake Temp	200 °C
Purge Temp	20 °C		
MCS Purge Temp	20 °C		
Dry Purge Time	1.00 min	Trap	Vocarb® 3000 (K)
Dry Purge Flow	100 mL/min	Purge Gas	Helium
Dry Purge Temp	20 °C		

**Table 2.** Teledyne Tekmar Atomx XYZ Soil Method Conditions.

Standby	Variable	Desorb	Variable
Valve Oven Temp	140 °C	Methanol Needle Rinse	Off
Transfer Line Temp	140 °C	Methanol Needle Rinse Volume	0.00 mL
Sample Mount Temp	90 °C	Water Needle Rinse Volume	7.00 mL
Water Heater Temp	90 °C	Sweep Needle Time	0.25 min
Sample Vial Temp	40 °C	Desorb Preheat Temp	245 °C
Soil Valve Temp	100 °C	GC Start Signal	Begin Desorb
Standby Flow	10 mL/min	Desorb Time	2.00 min
MCS Ready Temp	45 °C	Drain Flow	300 mL/min
Purge Ready Temp	40 °C	Desorb Temp	250 °C
Purge	Variable	Bake	Variable
Pre-purge Time	0.00 min	Bake Time	2.00 min
Pre-Purge Flow	0 mL/min	Bake Flow	400 mL/min
Pre-heat Mix Speed	Slow	Bake Temp	280 °C
Sample Pre-heat Time	0.00 min	MCS Bake Temp	200 °C
Pre-sweep Time	0.25 min		
Water Volume	10.00 mL		
Sweep Water Time	0.25 min	Trap	Vocarb® 3000 (K)
Sweep Water Flow	100 mL/min	Purge Gas	Helium
Sparge Vessel Heater	Off		
Purge Mix Speed	Medium		
Purge Time	11.00 min		
Purge Flow	40 mL/min		
Purge Temp	20 °C		
MCS Purge Temp	20 °C		
Dry Purge Time	1.00 min		
Dry Purge Flow	100 mL/min		
Dry Purge Temp	20 °C		

**Table 3.** Agilent 7890B GC and 5977A MSD System Conditions.

Agilent 7890B GC Conditions	
Column	Restek VMS, 20m x 0.18 mm, 1µm Film, Helium – 1 mL/min
Oven Profile	35 °C, 4 min, 15 °C/min to 85 °C, 30 °C/min to 225 °C, 2 min hold, Run Time 14.00 min
Inlet	180 °C, 120:1 Split, 19.752 psi
Agilent 5977A MSD Conditions	
Temp	Transfer Line 225 °C; Source 230 °C; Quad 150 °C
Scan	Range 35 m/z to 260 m/z, Solvent Delay 0.50 min, Normal Scanning
Gain	Gain Factor 10.00, Autotune

## Results

The relative standard deviation (%RSD) of the response factors (RF) for the calibration curve, MDL, accuracy, and precision data are shown in Table 4 (water) and Table 5 (soil). To demonstrate the Atomx XYZ's improved ability to remove excess water, a blank analyzed by the previous Atomx model was overlaid with a blank analyzed by the Atomx XYZ and is shown in Figure 1. Figure 2 (water) Figure 3 (soil) display a 50 ppb standard, indicating excellent peak resolution with minimal water inference for all VOCs.

**Table 4.** US EPA Method 8260 Water Calibration, Accuracy, and Precision Data.

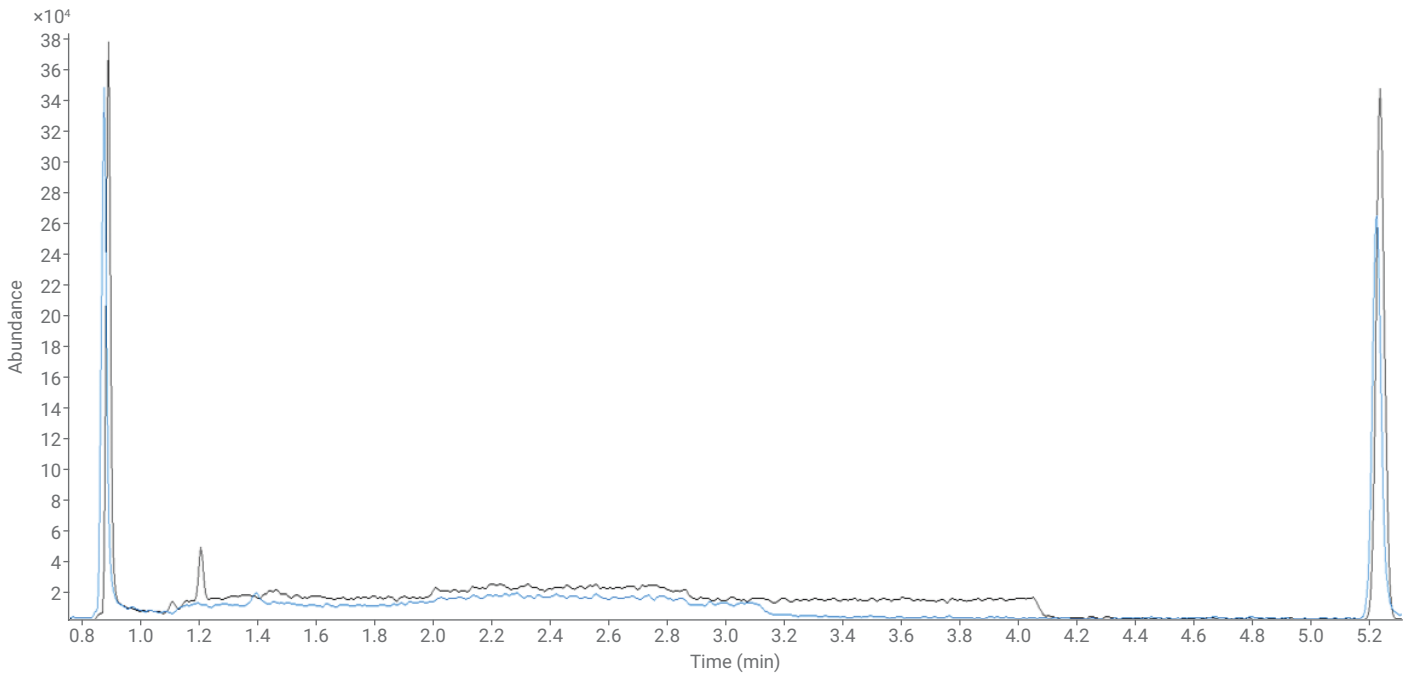
Compound	Calibration			Accuracy and precision (n = 7, 0.5 ppb) <sup>1</sup>		
	Linearity RF (%RSD)	MDL (ppb)	Average RF	Average Conc. (ppb)	Accuracy (%)	Precision (%RSD)
Pentafluorobenzene (IS)						
Dichlorodifluoromethane	4.72	0.15	0.360	0.43	85	11.4
Chloromethane	7.95	0.23	0.326	0.57	115	12.7
Vinyl chloride	5.49	0.10	0.493	0.51	103	6.24
Bromomethane	9.08	0.23	0.535	0.57	114	12.6
Chloroethane	4.44	0.28	0.309	0.58	116	15.3
Trichlorofluoromethane	4.93	0.13	0.859	0.44	89	9.27
Diethyl ether	8.57	0.15	0.316	0.53	105	8.68
1,1,2-Trichlorotrifluoroethane	7.10	0.18	0.488	0.44	87	13.6
Methyl Acetate	12.75	0.35	0.211	0.63	127	17.5
1,1-Dichloroethene	5.13	0.19	0.557	0.53	106	11.3
Carbon Disulfide	5.77	0.08	1.54	0.51	101	5.88
Iodomethane	16.84	0.19	0.589	0.61	122	9.84
Acetone <sup>2</sup>	0.999	0.27	0.116	0.56	111	16.1
Allyl Chloride	7.13	0.17	0.343	0.50	100	11.0
Acetonitrile	5.70	0.17	0.353	0.51	102	9.80
Methylene Chloride	4.76	0.10	0.555	0.58	117	5.17
tert-Butanol (TBA)	11.84	0.11	0.117	0.60	120	18.3
Methyl-tert-butyl ether (MTBE)	6.74	0.13	1.30	0.54	108	7.41
Vinyl acetate	9.29	0.12	0.390	0.41	81	9.51
Diisopropyl ether	6.98	0.10	0.668	0.52	104	5.77
trans-1,2-Dichloroethene	5.40	0.10	0.518	0.56	113	5.36
Acrylonitrile	15.5	0.45	0.148	0.58	116	24.1
1,1-Dichloroethane	4.27	0.16	0.532	0.50	101	10.0
Chloroprene	4.94	0.26	0.379	0.47	93	17.0
Ethyl-tert-butyl-ether (ETBE)	6.26	0.12	0.819	0.53	106	7.55
2,2-Dichloropropane	9.33	0.09	0.542	0.43	87	6.98
cis-1,2-Dichloroethene	4.87	0.18	0.462	0.49	98	12.2
Isobutanol	5.25	0.39	0.197	0.55	110	21.8
2-Butanone (MEK)	6.77	0.36	0.089	0.56	112	19.6
Ethyl Acetate	5.25	0.41	0.197	0.56	111	23.2
Methyl acrylate	8.03	0.33	0.245	0.58	117	19.0
Bromochloromethane	5.07	0.12	0.294	0.48	96	8.33
Chloroform	5.76	0.14	0.659	0.54	107	7.41
Methacrylonitrile	6.18	0.34	0.111	0.60	119	18.3
1,1,1-Trichloroethane	7.44	0.21	0.586	0.54	109	13.0
Dibromofluoromethane (SURRE)	3.28		0.438	24	94	1.96
Carbon Tetrachloride	9.16	0.17	0.544	0.40	79	12.5
1,1-Dichloropropene	5.68	0.09	0.475	0.51	102	5.88
1,2-Dichloroethane-d4 (SURRE)	1.76		0.380	25	99	4.69
Benzene	4.54	0.11	1.39	0.55	110	7.27
1,2-Dichloroethane	5.06	0.11	0.392	0.55	110	5.45
Isopropyl acetate	4.04	0.12	0.531	0.58	117	6.90
tert-Amyl methyl ether (TAME)	5.07	0.10	0.982	0.52	104	5.77
1,4-Difluorobenzene (IS)						
Trichloroethylene	5.97	0.14	0.334	0.53	106	7.55

Compound	Calibration			Accuracy and precision (n = 7, 0.5 ppb) <sup>1</sup>		
	Linearity RF (%RSD)	MDL (ppb)	Average RF	Average Conc. (ppb)	Accuracy (%)	Precision (%RSD)
1,2-Dichloropropane	3.77	0.26	0.213	0.52	105	15.4
Dibromomethane	5.37	0.14	0.227	0.55	110	7.27
Methyl methacrylate	7.00	0.27	0.163	0.57	113	14.0
Propyl acetate	6.30	0.26	0.185	0.58	115	13.8
Bromodichloromethane	8.50	0.19	0.336	0.48	96	12.5
2-Chloroethyl vinyl ether	7.29	0.27	0.141	0.56	112	14.3
cis-1,3-Dichloropropene	9.66	0.12	0.379	0.47	93	8.51
Toluene-d8 (SURR)	1.40		1.10	24	96	1.38
4-Methyl-2-pentanone	9.42	0.27	0.127	0.56	113	16.1
Toluene	5.59	0.08	1.18	0.52	105	5.77
trans-1,3-Dichloropropene	12.0	0.21	0.336	0.49	98	13.7
Ethyl methacrylate	6.74	0.12	0.271	0.49	97	8.16
Tetrachloroethene	13.7	0.21	0.409	0.61	122	11.0
1,1,2-Trichloroethane	9.68	0.13	0.254	0.49	98	8.16
1,3-Dichloropropane	4.19	0.13	0.387	0.51	103	7.84
Chlorobenzene-d5 (IS)						
2-Hexanone	16.3	0.35	0.098	0.61	121	18.0
Dibromochloromethane	9.22	0.09	0.353	0.50	100	6.00
Butyl acetate	4.43	0.10	0.235	0.55	110	5.45
1,2-Dibromoethane	4.76	0.15	0.282	0.52	104	9.62
Chlorobenzene	4.48	0.14	0.898	0.55	110	8.18
1,1,1,2-Tetrachloroethane	7.44	0.15	0.331	0.54	109	8.52
Ethylbenzene	7.36	0.11	1.45	0.54	107	6.48
m,p-Xylene	6.96	0.17	1.11	1.09	109	4.59
o-Xylene	6.94	0.07	1.13	0.53	105	3.77
Styrene	8.09	0.09	0.970	0.52	104	5.58
Bromoform	10.5	0.19	0.247	0.48	96	12.3
Amyl Acetate	7.96	0.11	0.260	0.52	104	6.69
Isopropylbenzene	5.89	0.13	1.45	0.53	106	7.55
cis-1,4-Dichloro-2-butene	8.38	0.11	0.094	0.50	99	7.20
Bromofluorobenzene (SURR)	3.22		0.381	25	100	3.64
Bromobenzene	11.3	0.19	0.551	0.60	120	10.0
n-Propylbenzene	7.10	0.11	1.65	0.52	105	6.92
1,4-Dichlorobenzene-d4 (IS)						
1,1,2,2-Tetrachloroethane	7.35	0.10	0.644	0.51	101	5.88
1,2,3-Trichloropropane	6.57	0.18	0.720	0.54	108	10.6
trans-1,4-Dichloro-2-butene	6.57	0.18	0.720	0.54	108	10.6
2-Chlorotoluene	6.80	0.16	1.77	0.58	115	8.62
1,3,5-Trimethylbenzene	9.00	0.11	2.29	0.50	100	7.20
4-Chlorotoluene	8.37	0.04	2.11	0.53	107	1.89
tert-Butylbenzene	7.85	0.09	2.69	0.49	97	6.12
1,2,4-Trimethylbenzene	7.03	0.06	2.40	0.51	101	3.73
sec-Butylbenzene	7.84	0.04	2.93	0.52	104	1.92
1,3-Dichlorobenzene	6.38	0.08	1.52	0.55	110	3.64
p-Isopropyltoluene	5.93	0.09	2.57	0.53	105	5.66
1,4-Dichlorobenzene	5.08	0.07	1.57	0.52	105	3.85
n-Butylbenzene	7.26	0.09	2.28	0.52	104	5.77

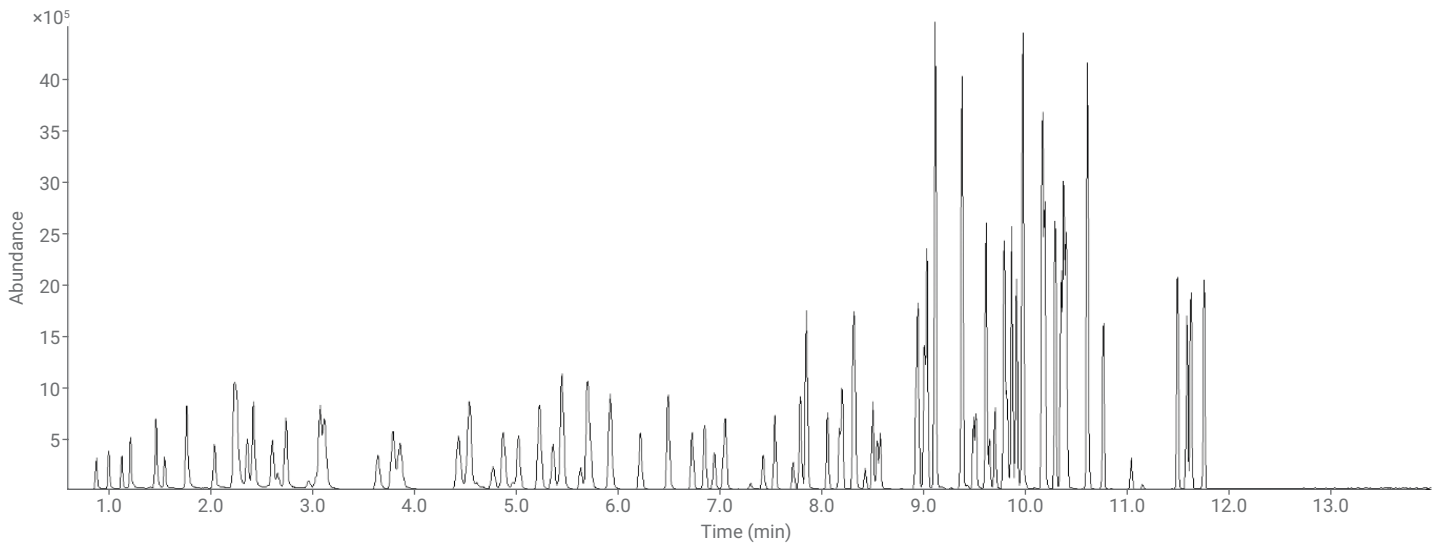
Compound	Calibration			Accuracy and precision (n = 7, 0.5 ppb) <sup>1</sup>		
	Linearity RF (%RSD)	MDL (ppb)	Average RF	Average Conc. (ppb)	Accuracy (%)	Precision (%RSD)
1,2-Dichlorobenzene	7.79	0.12	1.50	0.54	108	7.04
Hexachloroethane	13.4	0.17	0.473	0.45	89	11.1
1,2-Dibromo-3-chloropropane	6.15	0.32	0.157	0.48	97	20.8
1,2,4-Trichlorobenzene	9.97	0.08	1.15	0.55	111	5.45
Hexachlorobutadiene	12.2	0.13	0.570	0.50	99	8.00
Naphthalene	9.04	0.08	2.55	0.53	107	4.91
1,2,3-Trichlorobenzene	9.16	0.15	1.11	0.54	108	8.70

<sup>1</sup> Data from seven 0.5 ppb samples.

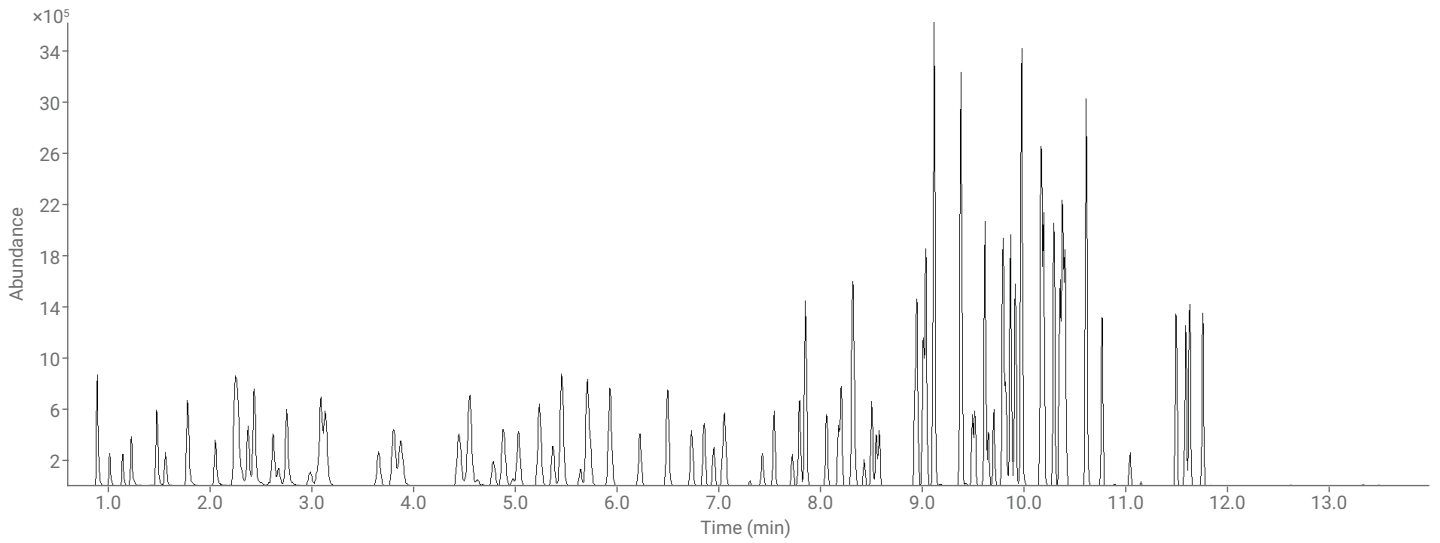
<sup>2</sup> Compound was linear regressed.



**Figure 1.** Chromatogram of Blanks Analyzed on the Atomx XYZ (blue) Versus the Atomx (black). These Results Indicate Improved Water Removal by the Atomx XYZ MCS.



**Figure 2.** Total Ion Chromatogram of the Water Method 50 ppb VOC Standard Indicating Consistent Peak Shapes for all Compounds with No Water Interference.



**Figure 3.** Total Ion Chromatogram of the Soil Method 50 ppb VOC Standard Indicating Consistent Peak Shapes for all Compounds with No Water Interference.

## Conclusions

This study demonstrates the capability of the Teledyne Tekmar Atomx XYZ P&T system to process VOCs in water and soil samples following the US EPA Method 8260 in conjunction with Methods 5030 and 5035 with detection by an Agilent 7890B GC/5977A MS. The %RSD of the calibration curve passed all method requirements. Furthermore, MDL, precision, and accuracy for seven 0.5 ppb and seven 1 ppb standards showed no interference from excessive water.

By making additional, appropriate changes to the GC oven temperature program, the GC/MS cycle time may also be reduced, increasing laboratory throughput in a 12-hour period.

## References

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