US EPA Method 8260 with the Tekmar Atomx XYZ and the Thermo Scientific™ TRACE™ 1610 GC and ISQ™ 7610 MS System with an HeSaver-H_2Safer™ SSL Injector

APPLICATION NOTE:	AN2303
TECHNOLOGY:	P+T VOC
INDUSTRY:	ENV

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Abstract

As helium supplies become scarcer and more expensive, customers have been seeking alternative carrier gases or ways to conserve helium without sacrificing system performance. This application note will evaluate the Teledyne Tekmar Atomx XYZ Purge and Trap (P&T) system in conjunction with a Thermo Scientific™ TRACE™ 1610 gas chromatograph (GC) equipped with an iConnect™ split/splitless injector, upgraded to work in HeSaver-H2Safer mode, coupled to the Thermo Scientific™ ISQ™ 7610 single quadrupole mass spectrometer (MS) with an ExtractaBrite ion source performing US EPA Method 8260, in conjunction with Methods 5030 and 5035, to determine the concentration of Volatile Organic Compounds (VOCs) in drinking water matrices. It will be shown that using nitrogen as the purge gas, along with the HeSaver-H_2Safer™ SSL injector, significantly reduces helium gas consumption during analysis. The method was validated by a working average response factor (RF) calibration curve, method detection limits (MDL), and a mid-point calibration check with accuracy and precision 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 life span. 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.

Sample Preparation

A working 50 parts per million (ppm) calibration standard was prepared in methanol from Restek® standards: 8260B MegaMix®, 8260B Acetate, California Oxygenates, VOA (Ketones), 502.2 Calibration Mix, Hexachloroethane, and 2-Chloroethyl Vinyl Ether. In total, the standard contained 97 compounds.

The water calibration curve was prepared from 0.5 parts per billion (ppb) to 200 ppb for all compounds,
while the soil calibration curve was prepared from 1 ppb to 200 ppb. The relative response factor (RRF) was calculated for each compound using one of the 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 4-Bromofluorobenzene. Internal and surrogate standards were prepared together in methanol from Restek standards at a concentration of
25 ppm, after which 5 microliters (µL) was then mixed with each 5 milliliters (mL) sample for a resulting concentration of 25 ppb.

Seven 0.5 ppb water standards and seven 2 ppb soil standards were prepared for MDL and precision calculations. Also, ten, 10 ppb water and soil standards were prepared as a mid-point calibration check and
were assessed using the precision and accuracy of each analyte's recovery. All calibration, MDL, and mid-point calibration check samples were analyzed with the Atomx XYZ conditions in Table I (water method) and Table II (soil method) and the GC/MS conditions in Table III.

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Experimental Instrument Conditions

Table I Teledyne Tekmar Atomx XYZ Water Method Conditions			
Standby	Variable	Desorb	Variable
Valve Oven Temp	150 °C	Methanol Needle Rinse	Off
Transfer Line Temp	150 °C	Water Needle Rinse Volume	7.00 mL
Sample Mount Temp	90 °C	Sweep Needle Time	0.25 min
Water Heater Temp	90 °C	Desorb Preheat Temp	245 °C
Sample Vial Temp	20 °C	GC Start Signal	Begin Desorb
Soil Valve Temp	50 °C	Desorb Time	0.50 min
Standby Flow	10 mL/min	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	Water Bake Rinses	5
Prime Sample Fill Volume	3.00 mL	Water Bake Rinse Volume	10.00 mL
Sample Volume	5.00 mL	Bake Rinse Sweep Time	0.25 min
Sweep Sample Time	0.25 min	Bake Rinse Sweep Flow	100 mL/min
Sweep Sample Flow	100 mL/min	Bake Rinse Drain Time	0.40 min
Sparge Vessel Heater	Off	Bake Time	4.00 min
Purge Time	11.00 min	Bake Flow	200 mL/min
Purge Flow	40 mL/min	Bake Temp	280 °C
Purge Temp	20 °C	MCS Bake Temp	200 °C
MCS Purge Temp	20 °C		
Dry Purge Time	1.00 min	Trap	#9
Dry Purge Flow	100 mL/min	Chiller Tray	Off
Dry Purge Temp	20 °C	Purge Gas	Nitrogen

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Table II Teledyne Tekmar Atomx XYZ Soil Method Conditions			
Standby	Variable	Purge	Variable
Valve Oven Temp	150 °C	Purge Temp	20 °C
Transfer Line Temp	150 °C	Condensate Purge Temp	20 °C
Sample Mount Temp	90 °C	Dry Purge Time	2.00 min
Water Heater Temp	90 °C	Dry Purge Flow	100 mL/min
Sample Vial Temp	20 °C	Dry Purge Temp	20 °C
Soil Valve Temp	100 °C	Methanol Needle Rinse	Off
Standby Flow	10 mL/min	Water Needle Rinse Volume	7.00 mL
Purge Ready Temp	40 °C	Sweep Needle Time	0.25 min
Purge	Variable	Desorb Preheat Temp	245 °C
Pre-purge Time	0.00 min	GC Start Signal	Begin Desorb
Pre-Purge Flow	0 mL/min	Desorb Time	0.50 min
Pre-heat Mix Speed	Slow	Drain Flow	300 mL/min
Sample Pre-heat Time	0.00 min	Desorb Temp	250 °C
Pre-sweep Time	0.25 min		4.00 min
Water Volume	10.00 mL	Bake	200 mL/min
Sweep Water Time	0.25 min	Bake Time	270 °C
Sweep Water Flow	100 mL/min	Bake Flow	200 °C
Sparge Vessel Heater	Off	Bake Temp	#9
Purge Mix Speed	Medium	MCS Bake Temp	Nitrogen
Purge Time	11.00 min	Trap	
Purge Flow	40 mL/min	Purge Gas	

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Table III Thermo Scientific TRACE 1610 GC and ISQ 7610 MS System Conditions	
Thermo Scientific TRACE 1610 GC	
Column	TG VMS, 20m x 0.18 mm, 1µm Film
Oven Profile	35 °C, 3 min, 12°C/min to 85 °C, 25°C/min to 225 °C, 2 min Hold, Run Time 14.767 min
Inlet	200 °C, 60:1 Split, purge flow 5.0 mL/min, 0.4 min Helium Delay
Pressurizing Gas	Nitrogen
Carrier Gas	Helium – 0.3 mL/min
ISQ 7610 MS System Conditions	
Temp	Transfer Line 230°C; Ion Source 280°C
Scan	Range 35 amu to 260 amu, Solvent Delay 1.55 min, Dwell/Scan Time 0.10 sec.
Current	Emission Current 30 µA, Gain 3.00E+005

Results

The relative standard deviation (%RSD) of the RFs for the calibration curve, MDL, precision, and mid-point calibration check accuracy and precision data are shown in Table IV (water) and Table V. (soil). Figure 1 (water) and Figure 2 (soil) display a 20 ppb standard, indicating excellent peak resolution with minimal water inference for all VOCs.

Table IV US EPA Method 8260 Water Calibration, Method Detection Limit, and Mid-Point Calibration Check Data

Compound	Calibration (0.5 ppb – 200 ppb)				Method Detection Limit (n=7, 0.5 ppb)		Mid-Point Check (n=10, 10 ppb)	
	Retention Time	Quant Ion	RF (≤20% RSD r^2≥0.99)	Average Relative Response Factor	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
Dichlorodifluoromethane	1.70	85	11.8	1.76	0.10	5.80	6.80	97
Chloromethane	1.91	50	10.1	2.54	0.11	6.61	6.28	100
Vinyl Chloride	2.00	62	8.05	2.29	0.06	3.54	6.37	102
Bromomethane	2.36	94	7.12	1.19	0.06	3.49	4.64	102
Chloroethane	2.52	64	5.63	1.45	0.11	7.08	9.01	94
Trichlorofluoromethane	2.70	101	6.12	2.04	0.09	5.28	6.49	101
Diethyl Ether	3.12	74	3.90	0.368	0.05	3.07	4.10	97
1,1-Dichloroethene	3.34	61	8.82	0.401	0.19	10.2	5.70	102
1,1,2-Trichlorotrifluoroethane	3.42	101	9.52	0.375	0.14	8.46	5.81	103
Iodomethane[1]	3.49	142	0.995	0.063	1.22	2.97	2.77	130
Carbon Disulfide	3.89	76	9.98	0.251	0.12	7.30	4.99	95
Allyl Chloride	3.93	76	8.06	0.248	0.15	8.35	5.72	96
Methylene Chloride[2]	4.06	49	0.997	1.01	0.19	5.66	2.57	122
Acetone[2]	4.12	58	0.995	0.082	0.59	11.2	5.55	120
trans-1,2-Dichloroethene	4.26	96	10.6	0.271	0.13	6.99	6.06	95

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Table IV US EPA Method 8260 Water Calibration, Method Detection Limit, and Mid-Point Calibration Check Data

Compound	Calibration (0.5 ppb – 200 ppb)				Method Detection Limit (n=7, 0.5 ppb)		Mid-Point Check (n=10, 10 ppb)	
	Retention Time	Quant Ion	RF (≤20% RSD r^2≥0.99)	Average Relative Response Factor	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
Methyl Acetate	4.30	43	12.3	0.954	0.14	8.84	4.22	96
Methyl-tert-butyl Ether (MTBE)	4.40	73	4.24	1.62	0.08	5.25	2.73	102
tert-Butyl Alcohol (TBA)	4.54	59	8.14	0.074	0.69	8.02	8.08	95
Diisopropyl Ether	4.86	45	4.32	1.89	0.07	4.76	2.56	100
Acetonitrile	4.86	41	6.18	0.691	0.10	6.29	2.39	103
Acrylonitrile	4.92	53	2.65	0.448	0.13	8.55	5.45	100
Chloroprene	4.92	53	3.71	0.446	0.12	7.60	5.36	100
1,1-Dichloroethane	4.95	63	6.48	0.821	0.08	4.54	3.81	102
Ethyl-tert-butyl- Ether (ETBE)	5.26	59	6.37	1.83	0.14	10.0	2.71	99
Vinyl Acetate	5.26	43	5.66	1.28	0.12	8.98	10.5	85
cis-1,2-Dichloroethene	5.53	96	7.26	0.347	0.14	8.13	3.61	101
2,2-Dichloropropane	5.65	77	8.05	0.645	0.09	5.49	7.95	88
Bromochloromethane	5.74	128	11.1	0.154	0.12	7.46	1.62	99
Chloroform	5.83	83	7.28	0.934	0.07	4.22	3.39	103
Carbon Tetrachloride	5.97	117	6.71	0.363	0.15	9.58	4.67	97
Methyl Acrylate	5.98	55	7.16	0.506	0.12	8.49	4.30	97
Ethyl Acetate	5.99	88	5.20	0.685	0.11	7.06	4.36	99
Tetrahydrofuran	6.00	42	8.98	0.303	0.16	9.92	4.40	101
Dibromofluoromethane (SURR)	6.02	111	3.86	0.773		2.38	1.50	103
1,1,1-Trichloroethane	6.03	97	5.33	0.555		4.56	4.63	104
2-Butanone (MEK)	6.16	72	7.32	0.142	0.07	9.52	5.30	105
1,1-Dichloropropene	6.17	75	4.49	0.297	0.45	7.15	5.47	97
Benzene	6.42	78	2.98	1.21	0.11	4.22	4.05	100
Propionitrile	6.44	54	10.6	0.124	0.07	9.89	4.09	98
Methacrylonitrile	6.46	41	4.31	0.524	0.18	6.41	3.37	102
Pentafluorobenzene (IS)	6.53	168			0.10			
1,2-Dichloroethane-d4 (SURR)	6.55	65						
tert-Amyl Methyl Ether (TAME)	6.57	73	4.37	0.420		1.82	2.33	103
1,2-Dichloroethane	6.62	62	5.61	1.53	0.04	2.56	2.30	97
Isobutanol	6.92	43	7.03	0.767	0.10	5.34	2.33	106
Isopropyl Acetate	6.92	43	6.58	1.27	0.10	6.38	3.67	100
Trichloroethene	7.02	95	5.26	1.27	0.13	8.94	3.56	100
1,4-Diflourobenzene (IS)	7.06	114	6.80	0.624	0.16	9.80	3.14	107
Dibromomethane	7.44	93						
1,2-Dichloropropane	7.53	63	6.74	0.300	0.16	9.87	2.83	105
Bromodichloromethane	7.60	83	5.23	0.601	0.05	3.14	2.64	102
Methyl Methacrylate	7.76	69	4.38	0.830	0.07	4.57	1.88	103
Propyl Acetate	7.89	43	8.93	0.398	0.14	9.51	4.37	96
			6.14	0.964	0.10	7.26	4.27	98

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Table IV US EPA Method 8260 Water Calibration, Method Detection Limit, and Mid-Point Calibration Check Data

Compound	Calibration (0.5 ppb – 200 ppb)				Method Detection Limit (n=7, 0.5 ppb)		Mid-Point Check (n=10, 10 ppb)	
	Retention Time	Quant Ion	RF (≤20% RSD r^2≥0.99)	Average Relative Response Factor	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
2-Chloroethyl Vinyl Ether	8.12	63	9.21	0.498	0.17	11.6	3.08	96
cis-1,3-Dichloropropene	8.16	75	3.89	0.928	0.10	7.27	3.38	98
Toluene-d8 (SURR)	8.32	98	1.97	0.792		1.29	1.92	100
Toluene	8.36	92	6.55	0.855	0.17	10.8	4.88	95
2-Nitropropane	8.66	43	5.06	0.645	0.14	9.44	4.92	97
4-Methyl-2-Pentanone	8.66	100	6.98	0.029	0.29	7.28	5.15	91
Tetrachloroethene	8.67	164	10.6	0.360	0.09	4.85	4.49	116
trans-1,3-Dichloropropene	8.69	75	6.05	0.824	0.08	5.66	3.71	96
1,1,2-Trichloroethane	8.81	83	6.61	0.434	0.11	8.09	4.06	102
Ethyl Methacrylate	8.82	69	9.74	0.676	0.06	4.70	4.41	97
Dibromochloromethane	8.95	129	5.37	0.458	0.07	4.93	3.54	100
1,3-Dichloropropane	9.02	76	4.60	0.884	0.05	3.31	4.14	101
1,2-Dibromoethane	9.13	107	4.05	0.410	0.05	3.47	3.62	101
Butyl Acetate	9.23	43	6.63	0.944	0.14	9.69	5.50	98
2-Hexanone	9.30	43	6.05	0.165	0.36	8.92	6.41	97
Chlorobenzene-d5 (IS)	9.51	117						
Chlorobenzene	9.52	112						
Ethylbenzene	9.54	91	2.10	1.11	0.05	2.88	3.98	98
1,1,1,2-Tetrachloroethane	9.57	131	2.80	1.97	0.07	4.33	4.68	95
m-,p-Xylene	9.65	106	5.63	0.404	0.07	5.13	3.65	98
o-Xylene	9.95	106	3.26	0.690	0.16	5.42	5.08	97
Styrene	9.99	104	2.02	0.722	0.11	7.36	4.24	96
Bromoform	10.01	173	6.86	1.23	0.10	7.15	4.35	97
Isopropylbenzene	10.17	105	13.1	0.261	0.07	5.76	4.51	99
Amyl Acetate[1]	10.25	43	2.71	1.84	0.09	6.23	5.42	96
4-Bromofluorobenzene (SURR)	10.37	95	11.9	1.03	0.11	8.89	3.60	98
cis-1,4-Dichloro-2-Butene	10.41	75	3.20	1.27		2.19	2.72	100
Bromobenzene	10.44	156	12.1	0.511	0.18	15.8	6.19	94
n-Propylbenzene	10.46	91	5.42	0.832	0.09	5.80	4.20	99
1,1,2,2-Tetrachloroethane	10.50	83	5.17	4.80	0.09	5.81	4.26	99
2-Chlorotoluene	10.57	91	10.6	1.27	0.05	3.87	5.27	97
1,3,5-Trimethylbenzene	10.59	105	4.15	3.66	0.08	4.75	4.43	100
1,2,3-Trichloropropane	10.59	75	10.1	3.30	0.10	7.00	3.89	98
trans-1,4-dichloro-2-butene	10.62	53	10.1	1.44	0.17	8.34	4.57	106
4-Chlorotoluene	10.68	91	7.12	0.668	0.10	6.62	5.35	97
Pentachloroethane	10.81	77	4.52	3.34	0.05	3.11	3.91	100
tert-Butylbenzene	10.81	119	7.35	0.440	0.17	12.0	4.44	100
1,2,4-Trimethylbenzene	10.85	105	8.92	3.06	0.13	9.37	6.02	92
			9.96	3.37	0.05	4.07	3.92	99

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Table IV US EPA Method 8260 Water Calibration, Method Detection Limit, and Mid-Point Calibration Check Data

Compound	Calibration (0.5 ppb – 200 ppb)				Method Detection Limit (n=7, 0.5 ppb)		Mid-Point Check (n=10, 10 ppb)	
	Retention Time	Quant Ion	RF (≤20% RSD r²≥0.99)	Average Relative Response Factor	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
sec-Butylbenzene	10.93	105	7.38	3.84	0.08	5.80	5.37	100
p-Isopropyltoluene	11.02	119	11.3	2.93	0.11	8.87	4.90	100
1,3-Dichlorobenzene	11.08	146	5.73	1.72	0.07	4.41	3.94	99
1,4-Dichlorobenzene-d4 (IS)	11.13	152						
1,4-Dichlorobenzene	11.14	146	9.08	1.70	0.10	6.19	4.11	101
n-Butylbenzene	11.31	91	11.2	3.04	0.09	6.40	5.00	97
Hexachloroethane	11.41	117	9.22	0.910	0.05	6.04	4.97	93
1,2-Dichlorobenzene	11.42	146	5.76	1.67	0.08	4.83	4.45	100
1,2-Dibromo-3-Chloropropane	11.95	157	8.81	0.195	0.14	9.22	6.39	99
Nitrobenzene	12.33	123	13.0	0.033	0.27	12.4	9.52	89
Hexachlorobutadiene	12.38	225	9.49	0.236	0.19	11.2	7.56	99
1,2,4-Trichlorobenzene	12.40	180	6.84	0.773	0.14	7.92	4.39	101
Naphthalene	12.62	128	9.18	2.20	0.08	5.34	5.37	96
1,2,3-Trichlorobenzene	12.74	180	8.67	0.664	0.15	8.90	5.01	104

[1]Compound used a quadratic calibration

[2] Compound used a linear calibration

Table V US EPA Method 8260 Soil Calibration, Method Detection Limit, and Mid-Point Calibration Check Data

Compound	Calibration (1 ppb – 200 ppb)				Method Detection Limit (n=7, 2 ppb)		Mid-Point Check (n=10, 10 ppb)	
	Retention Time	Quant Ion	RF (≤20% RSD r²≥0.99)	Average Relative Response Factor	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
Dichlorodifluoromethane	1.71	85	13.7	1.36	0.24	3.39	4.74	103
Chloromethane[1]	1.91	50	0.995	2.47	0.19	2.82	5.53	100
Vinyl Chloride	2.00	62	11.5	1.77	0.24	3.48	5.21	97
Bromomethane	2.36	94	11.1	0.946	0.20	3.03	5.47	94
Chloroethane	2.51	64	8.39	1.14	0.25	4.08	6.01	92
Trichlorofluoromethane	2.70	101	12.4	1.77	0.21	3.09	5.58	99
Diethyl Ether	3.11	74	7.08	0.075	0.21	3.22	7.14	91
1,1-Dichloroethene	3.34	61	13.5	0.054	0.43	5.85	4.97	105
1,1,2-Trichlorotrifluoroethane	3.42	101	13.8	0.126	0.28	3.85	6.87	101
Iodomethane[2]	3.52	142	0.996	0.006	2.42	7.19	8.12	105
Allyl Chloride	3.93	76	9.46	0.054	0.45	6.80	9.64	94

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Table V US EPA Method 8260 Soil Calibration, Method Detection Limit, and Mid-Point Calibration Check Data

Compound	Calibration (1 ppb – 200 ppb)				Method Detection Limit (n=7, 2 ppb)		Mid-Point Check (n=10, 10 ppb)	
	Retention Time	Quant Ion	RF (≤20% RSD $r^2 \geq 0.99$)	Average Relative Response Factor	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
Carbon Disulfide	3.93	76	7.47	0.054	0.50	7.64	9.55	94
Methylene Chloride[1]	4.06	49	0.999	0.618	1.09	17.3	6.77	98
Acetone[1]	4.11	58	0.999	0.093	4.73	6.40	5.35	95
trans-1,2-Dichloroethene	4.25	96	10.5	0.040	0.18	2.57	7.28	107
Methyl Acetate	4.28	43	12.5	0.332	0.64	8.02	5.42	100
Methyl-tert-butyl Ether (MTBE)	4.40	73	3.54	0.869	0.16	2.61	3.60	93
tert-Butyl Alcohol (TBA)	4.52	59	12.7	0.054	1.57	4.74	2.74	88
Acetonitrile	4.85	41	5.57	0.435	0.38	5.89	6.05	105
Diisopropyl Ether	4.86	45	4.72	1.60	0.18	2.92	4.73	95
Acrylonitrile	4.93	53	11.1	0.135	0.17	2.79	4.07	92
Chloroprene	4.93	53	10.8	0.135	0.19	3.17	3.99	93
1,1-Dichloroethane	4.95	63	11.5	0.367	0.34	5.28	5.96	93
Vinyl Acetate	5.25	43	4.08	0.604	0.40	7.74	7.78	83
Ethyl-tert-butyl- Ether (ETBE)	5.25	59	3.13	1.03	0.09	1.54	3.55	93
cis-1,2-Dichloroethene	5.53	96	10.1	0.162	0.35	5.56	4.12	92
2,2-Dichloropropane	5.64	77	11.2	0.386	0.29	4.49	3.31	93
Bromochloromethane	5.74	128	8.63	0.054	0.57	8.43	7.47	93
Chloroform	5.83	83	8.73	0.589	0.34	5.26	4.88	94
Carbon Tetrachloride	5.97	117	9.46	0.142	0.51	7.72	4.77	96
Methyl Acrylate	5.97	55	14.0	0.163	0.21	3.29	3.67	85
Ethyl Acetate	5.98	88	11.6	0.344	0.55	7.63	3.51	91
Tetrahydrofuran	5.99	42	6.65	0.137	0.50	7.90	6.86	95
Dibromofluoromethane (SURR)	6.02	111	2.66	0.578		2.31	2.54	101
1,1,1-Trichloroethane	6.03	97	11.7	0.286		2.21	4.27	96
2-Butanone (MEK)[2]	6.14	72	16.5	0.107	0.14	5.47	6.53	85
1,1-Dichloropropene	6.17	75	7.18	0.082	0.95	5.70	5.39	97
Benzene	6.42	78	12.6	0.479	0.38	1.60	4.42	92
Propionitrile	6.43	54	8.56	0.055	0.10	7.16	8.42	94
Methacrylonitrile	6.45	41	12.3	0.310	0.44	4.31	2.99	91
Pentafluorobenzene (IS)	6.52	168			0.27			
1,2-Dichloroethane-d4 (SURR)	6.55	65						
tert-Amyl Methyl Ether (TAME)	6.57	73						
1,2-Dichloroethane	6.62	62	4.64	0.286		5.49	2.05	98
Isopropyl Acetate	6.91	43	7.49	0.920	0.21	3.44	3.35	90
Isobutanol	6.92	43	10.0	0.302	0.62	9.79	3.72	93
Trichloroethene	7.03	95	2.79	0.748	0.42	6.65	3.44	95
1,4-Diflourobenzene (IS)	7.06	114	6.02	0.760	0.40	6.60	3.60	93
Dibromomethane	7.43	93	10.6	0.248	0.24	3.40	6.96	105
1,2-Dichloropropane	7.53	63						
			11.7	0.137	0.32	4.88	8.69	89
			8.19	0.358	0.15	2.36	4.64	94

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Table V US EPA Method 8260 Soil Calibration, Method Detection Limit, and Mid-Point Calibration Check Data

Compound	Calibration (1 ppb – 200 ppb)				Method Detection Limit (n=7, 2 ppb)		Mid-Point Check (n=10, 10 ppb)	
	Retention Time	Quant Ion	RF (≤20% RSD $r^2 \geq 0.99$)	Average Relative Response Factor	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
Bromodichloromethane	7.60	83	6.87	0.578	0.27	4.47	4.44	92
Methyl Methacrylate	7.75	69	4.80	0.175	0.37	6.19	4.46	90
Propyl Acetate	7.88	43	8.81	0.584	0.11	1.78	2.67	92
2-Chloroethyl Vinyl Ether	8.11	63	7.52	0.207	0.40	8.88	4.88	78
cis-1,3-Dichloropropene	8.16	75	3.75	0.576	0.19	3.29	3.88	89
Toluene-d8 (SURR)	8.31	98	2.70	0.829		1.54	1.54	102
Toluene	8.36	92	9.49	0.663	0.45	5.98	4.34	100
2-Nitropropane	8.66	43	10.8	0.469	0.27	4.31	2.64	94
4-Methyl-2-Pentanone	8.66	100	5.11	0.017	1.78	11.8	3.72	91
Tetrachloroethene	8.67	164	19.2	0.279	0.39	5.64	15.2	105
trans-1,3-Dichloropropene	8.69	75	5.07	0.521	0.24	4.26	3.01	88
1,1,2 Trichloroethane	8.81	83	3.94	0.288	0.23	3.94	2.97	93
Ethyl Methacrylate	8.82	69	6.42	0.425	0.30	4.87	3.76	91
Dibromochloromethane	8.95	129	5.28	0.308	0.19	3.42	4.28	91
1,3-Dichloropropane	9.02	76	3.88	0.529	0.23	3.92	3.21	93
1,2-Dibromoethane	9.12	107	7.87	0.231	0.26	4.62	2.98	90
Butyl Acetate	9.23	43	8.94	0.724	0.22	3.60	3.28	91
2-Hexanone	9.29	43	14.2	0.131	1.09	6.74	3.63	91
Chlorobenzene-d5 (IS)	9.50	117						
Chlorobenzene	9.52	112						
Ethylbenzene	9.54	91	10.5	0.943	0.16	2.44	3.68	94
1,1,1,2-Tetrachloroethane	9.56	131	11.7	1.83	0.15	2.30	3.77	94
m-,p-Xylene	9.64	106	6.95	0.329	0.19	3.29	3.50	95
o-Xylene	9.95	106	11.1	0.678	0.28	2.22	3.94	93
Styrene	9.98	104	10.4	0.730	0.16	2.54	3.73	92
Bromoform	10.00	173	7.32	1.26	0.16	2.75	4.14	91
Isopropylbenzene	10.17	105	6.18	0.203	0.30	5.69	3.67	85
Amyl Acetate	10.24	43	10.2	2.03	0.20	3.36	3.75	94
4-Bromofluorobenzene (SURR)	10.36	95	6.28	0.860	0.42	7.58	3.48	85
cis-1,4-Dichloro-2-Butene	10.40	75	2.19	1.10		1.38	1.88	105
Bromobenzene	10.43	156	7.88	0.246	0.41	7.75	5.00	88
n-Propylbenzene	10.45	91	10.1	0.739	0.29	4.47	5.26	95
1,1,2,2-Tetrachloroethane	10.49	83	7.50	4.54	0.20	3.09	5.46	98
2-Chlorotoluene	10.56	91	4.69	0.757	0.27	4.40	7.08	94
1,3,5-Trimethylbenzene	10.58	105	7.48	3.24	0.26	4.08	5.03	97
1,2,3-Trichloropropane	10.59	75	6.38	3.12	0.24	3.88	5.55	96
trans-1,4-dichloro-2-butene	10.61	53	11.7	0.826	0.57	8.78	6.93	101
4-Chlorotoluene	10.67	91	5.36	0.344	0.28	4.45	5.77	94
Pentachloroethane	10.80	77	7.57	3.00	0.23	3.59	6.31	94
			8.41	0.412	0.15	2.43	5.52	99

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Table V US EPA Method 8260 Soil Calibration, Method Detection Limit, and Mid-Point Calibration Check Data

Compound	Calibration (1 ppb – 200 ppb)				Method Detection Limit (n=7, 2 ppb)		Mid-Point Check (n=10, 10 ppb)	
	Retention Time	Quant Ion	RF (≤20% RSD $r^2 \geq 0.99$)	Average Relative Response Factor	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
tert-Butylbenzene	10.80	119	7.94	3.03	0.33	5.25	6.89	95
1,2,4-Trimethylbenzene	10.85	105	5.82	3.14	0.24	3.92	6.05	95
sec-Butylbenzene	10.92	105	8.16	4.02	0.20	3.15	5.29	98
p-Isopropyltoluene	11.01	119	8.49	3.03	0.22	3.65	5.34	95
1,3-Dichlorobenzene	11.07	146	9.54	1.70	0.37	5.68	5.10	95
1,4-Dichlorobenzene-d4 (IS)	11.12	152						
1,4-Dichlorobenzene	11.13	146	5.76	1.74	0.25	3.77	5.00	94
n-Butylbenzene	11.29	91	8.73	3.39	0.26	4.29	4.67	93
Hexachloroethane	11.40	117	10.1	1.19	0.11	3.42	5.58	96
1,2-Dichlorobenzene	11.41	146	5.73	1.55	0.31	4.73	5.18	95
1,2-Dibromo-3-Chloropropane	11.94	157	7.63	0.130	0.50	8.85	4.50	88
Nitrobenzene[1]	12.31	123	0.999	0.024	0.87	10.4	7.55	93
Hexachlorobutadiene	12.37	225	14.2	0.055	0.29	4.07	5.30	99
1,2,4-Trichlorobenzene	12.39	180	8.96	0.789	0.39	6.09	5.33	88
Naphthalene	12.61	128	7.25	1.42	0.33	5.29	4.95	93
1,2,3-Trichlorobenzene	12.73	180	6.76	0.601	0.47	7.24	5.23	91

[1]Compound used linear calibration

[2] Compound used quadratic calibration

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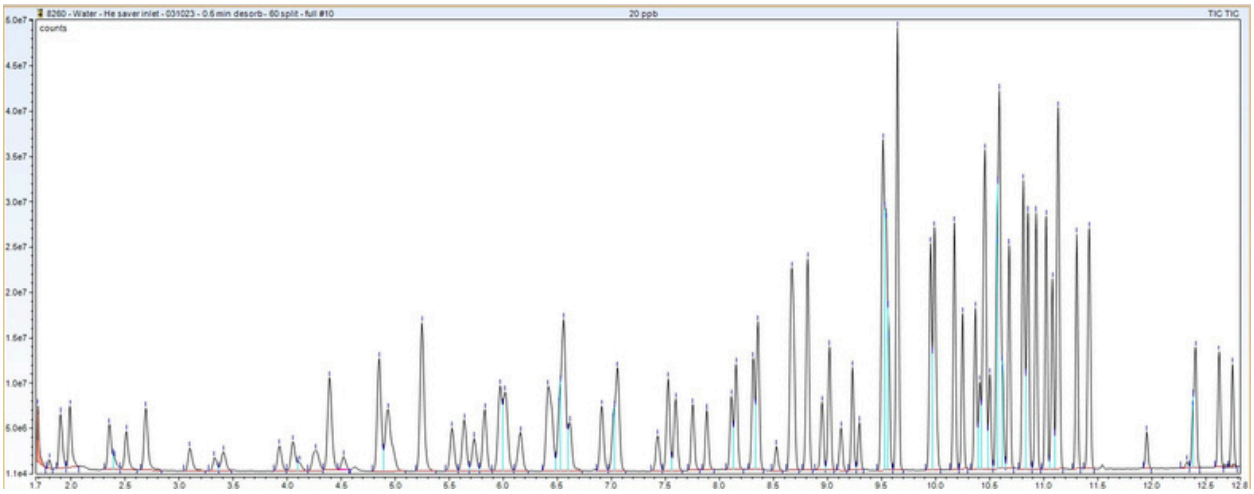

Figure 1 Total ion chromatogram (TIC) of a US EPA 8260 water method 20 ppb VOC standard indicating consistent peak shapes for all compounds with minimal water interference.

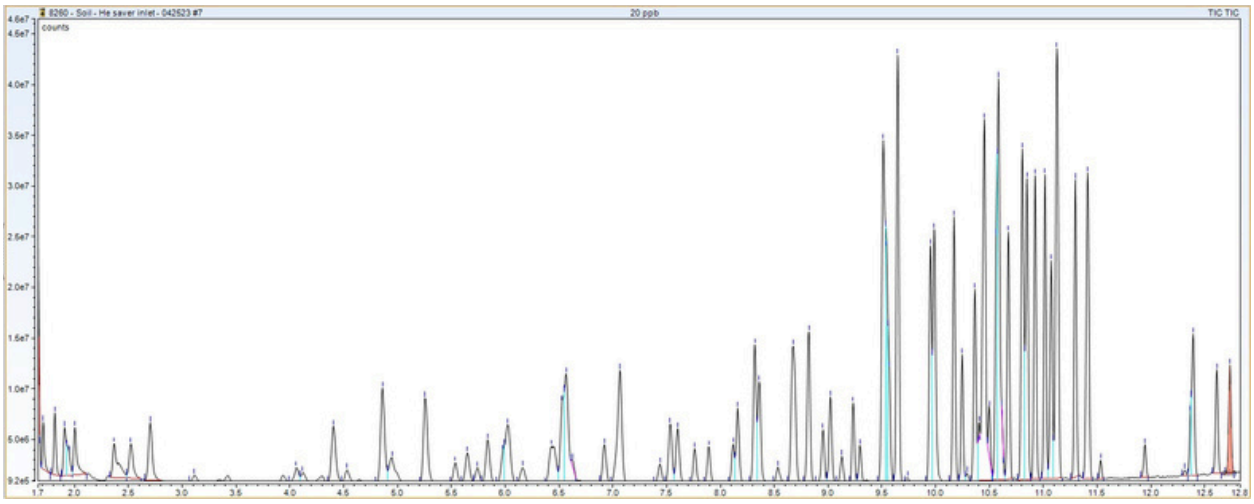

Figure 2 TIC of a US EPA 8260 soil method 20 ppb VOC standard indicating consistent peak shapes for all compounds with minimal water interference.

Conclusion

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 a Thermo Scientific™ TRACE™ 1610 gas chromatograph (GC) equipped with an iConnect™ split/splitless injector, upgraded to work in HeSaver-H2Safer mode, coupled to the Thermo Scientific™ ISQ™ 7610 single quadrupole mass spectrometer (MS) with an ExtractaBrite ion source. The %RSD of the calibration curve passed all method requirements. Furthermore, MDL and precision, for seven 0.5 ppb standards for the water method and seven 2 ppb standards for the soil method, showed minimal interference from excessive water. The mid-point calibration check with precision and accuracy for ten, 10 ppb water standards displayed an average of 4.6% RSD and an average recovery of 100% for compounds of interest. The mid-point calibration check with precision and accuracy for ten, 10 ppb soil standards displayed an average of 5% RSD and an average recover of 94% for compounds of interest.

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.

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1. *Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*; US EPA, Office of Solid Waste, SW-846 Method 8260B, Revision 2, December 1996.

2. *Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*; US EPA, Office of Solid Waste, SW-846 Method 8260C, Revision 3, August 2006.

3. *Purge and Trap for Aqueous Samples*; US EPA, Office of Solid Waste, SW-846 Method 5030B, Revision 2, December 1996.

4. *Purge and Trap for Aqueous Samples*; US EPA, Office of Solid Waste, SW-846 Method 5030C, Revision 3, May 2003.

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