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TECHNOLOGY: P+T VOC
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US EPA Method 8260 Using the Teledyne Tekmar Atomx XYZ with Agilent 7890B GC/5977B MS and Hydrogen as an Alternative Carrier Gas

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Abstract

As helium supplies become increasingly scarce and expensive, laboratories have begun seeking alternative carrier gases that are both readily available and economical. This application note will evaluate the use of hydrogen as the carrier gas for US EPA Method 8260, in conjunction with Methods 5030 and 5035, 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)/5977B mass spectrometer (MS) was used to create a working linear calibration curve, method detection limits (MDLs) for target compounds and a mid-point calibration check 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.

Sample Preparation

A working 50 parts per million (ppm) or milligram per liter (mg/L) calibration standard was 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 standard contained 97 compounds.

The water calibration curve was prepared from 0.5 parts per billion (ppb) or microgram per liter (µg/L) to 200 ppb and the soil calibration curve was prepared from 2 ppb to 200 ppb for all compounds. The relative response factor (RRF) 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 4-Bromofluorobenzene. Internal and surrogate standards were prepared in methanol from Restek standards at a concentration of 25 ppm, after which 5 microliters (µL) was then mixed with each 5 milliliter (mL) sample for a resulting concentration of 25 ppb.

Seven 0.5 ppb (water method) and seven 2 ppb (soil method) standards were prepared to calculate the method detection limit (MDL) and precision calculations. Seven 20 ppb water and soil standards were prepared for the mid-point calibration check, precision and accuracy. All calibration, MDL and mid-point calibration check standards were analyzed using the Atomx XYZ conditions shown in Table I (water method) and Table II (soil method). GC-MS conditions are shown in Table III.

Experimental Instrument Conditions

Table I Teledyne Tekmar Atomx XYZ Water Method Conditions			
Standby	**Variable**	**Desorb**	**Variable**
Valve Oven Temp	140 ºC	Water Needle Rinse Volume	7.00 mL
Transfer Line Temp	140 ºC	Sweep Needle Time	0.25 min
Sample Mount Temp	90 ºC	Desorb Preheat Temp	245 ºC
Water Heater Temp	90 ºC	Desorb Time	2.00 min
Sample Cup Temp	20 ºC	Drain Flow	300 mL/min
Soil Valve Temp	100 ºC	Desorb Temp	250 ºC
Standby Flow	10 mL/min	Methanol Needle Rinse	Off
Purge Ready Temp	40 ºC	GC Start Signal	Begin Desorb
Purge	**Variable**	**Bake**	**Variable**
Sample Equilibrate Time	0.00 min	Methanol Glass Rinse	Off
Presweep Time	0.25 min	Water Bake Rinses	1
Prime Sample Fill Volume	3.00 mL	Water Bake Rinse Volume	7.00 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	2.00 min
Purge Time	11.00 min	Bake Flow	200 mL/min
Purge Flow	40 mL/min	Bake Temp	260 ºC
Purge Temp	20 ºC	MCS Bake Temp	180 ºC
MCS Purge Temp	20 ºC		
Dry Purge Time	0.5 min		
Dry Purge Flow	100 mL/min	**Trap**	#9
Dry Purge Temp	20 ºC	**Purge Gas**	Nitrogen

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Table II Teledyne Tekmar Atomx XYZ Soil Method Conditions			
Standby	**Variable**	**Purge**	**Variable**
Valve Oven Temp	140 ºC	Purge Temp	20 ºC
Transfer Line Temp	140 ºC	MCS Purge Temp	20 ºC
Sample Mount Temp	90 ºC	Dry Purge Time	1.00 min
Water Heater Temp	90 ºC	Dry Purge Flow	100 mL/min
Sample Cup Temp	40 ºC	Dry Purge Temp	20 ºC
Soil Valve Temp	100 ºC	**Desorb**	**Variable**
Standby Flow	10 mL/min	Methanol Needle Rinse	Off
Purge Ready Temp	40 ºC	Water Needle Rinse Volume	7.00 mL
Purge	**Variable**	Sweep Needle Time	0.25 min
Prepurge Time	0.00 min	Desorb Preheat Temp	245 ºC
Prepurge Flow	0 mL/min	GC Start Signal	Begin Desorb
Preheat Mix Speed	Slow	Desorb Time	2.00 min
Sample Preheat Time	0.00 min	Drain Flow	300 mL/min
Presweep Time	0.25 min	Desorb Temp	250 ºC
Water Volume	10.00 mL	**Bake**	**Variable**
Sweep Water Time	0.25 min	Bake Time	2.00 min
Sweep Water Flow	100 mL/min	Bake Flow	200 mL/min
Sparge Vessel Heater	Off	Bake Temp	260 ºC
Purge Mix Speed	Medium	MCS Bake Temp	180 ºC
Purge Time	11.00 min	**Trap**	#9
Purge Flow	40 mL/min	**Purge Gas**	Nitrogen

Table III Agilent 7890B GC/5977B MS System Conditions	
Agilent 7890B GC Conditions	
Column	Rtx® VMS, 20 m x 0.18 mm, 1 µm Film, Hydrogen – 0.8 mL/min
Oven Profile	35 ºC, 2 min, 10 ºC/min to 85 ºC, 30 ºC/min to 225 ºC, 1 min Hold, Run Time 12.67 min
Inlet	200 ºC, 80:1 Split, Septum Purge Flow 0.5 mL/min, Carrier Gas: Hydrogen
Agilent 5977B MS Conditions	
Temp	Transfer Line 250 ºC; Source 230 ºC; Quad 150 ºC
Scan	Range 35 *m/z* to 260 *m/z*, Solvent Delay 0.50 min, Normal Scanning
Current	Gain Factor 3.00, Extraction Source Tune

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Results

The relative standard deviation (%RSD) of the RRFs for the calibration curve, MDL, precision and mid-point verification 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 Calibration Check (n=7, 20 ppb)	
	Retention Time	Quant Ion	IS	RRF (≤20% RSD r^2≥0.99)	Average RRF	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
Dichlorodifluoromethane	0.786	85	1	10.1	1.46	0.14	9.24	5.06	82
Chloromethane	0.886	50	1	7.97	1.00	0.20	11.4	4.43	87
Vinyl Chloride	0.912	62	1	12.6	1.30	0.12	7.56	6.87	82
Bromomethane	1.08	94	1	14.8	1.20	0.18	8.28	6.34	92
Chloroethane	1.15	64	1	11.0	1.06	0.16	9.79	5.17	88
Trichlorofluoromethane	1.22	101	1	6.59	2.13	0.12	8.48	5.73	92
Diethyl Ether	1.41	59	1	3.97	0.848	0.13	7.85	4.84	95
1,1-Dichloroethene	1.51	61	1	8.74	1.25	0.18	11.1	6.20	89
Carbon Disulfide	1.52	76	1	6.39	1.80	0.13	7.48	5.22	82
1,1,2-Trichlorotrifluoroethane	1.54	101	1	8.11	0.977	0.10	6.57	4.98	90
Iodomethane	1.59	142	1	19.8	1.39	0.06	4.16	5.09	92
Allyl Chloride	1.81	41	1	5.51	0.999	0.19	9.85	3.49	96
Methylene Chloride	1.90	49	1	7.38	1.12	0.19	9.54	6.98	92
Acetone[1]	1.96	43	1	0.999	0.119	0.35	8.10	5.48	102
trans-1,2-Dichloroethene	2.01	61	1	7.78	1.16	0.13	7.92	6.33	90
Methyl Acetate	2.06	43	1	10.8	0.573	0.14	7.98	5.86	91
Methyl-tert-butyl Ether (MTBE)	2.12	73	1	9.53	3.33	0.12	7.23	5.10	92
tert-Butyl Alcohol (TBA)	2.25	59	1	7.96	0.058	0.70	8.91	9.41	108
Acetonitrile[1]	2.30	41	1	0.999	0.146	0.12	7.23	8.79	100
Diisopropyl Ether	2.44	45	1	8.35	2.70	0.15	8.81	4.32	95
Chloroprene	2.46	53	1	9.14	1.26	0.08	4.54	5.21	88
1,1-Dichloroethane	2.49	63	1	5.08	1.81	0.14	8.08	5.28	95
Acrylonitrile	2.54	53	1	10.5	0.401	0.08	4.91	5.04	93
Ethyl-tert-butyl- Ether (ETBE)	2.72	59	1	5.87	2.95	0.12	8.05	5.33	98
Vinyl Acetate	2.73	43	1	9.19	1.62	0.13	7.71	5.46	93
cis-1,2-Dichloroethene	2.92	61	1	10.2	1.33	0.17	10.7	8.60	92
2,2-Dichloropropane	2.99	77	1	8.59	0.886	0.15	9.33	3.53	89
Bromochloromethane	3.06	49	1	6.55	0.499	0.17	10.2	6.10	94
Chloroform	3.15	83	1	6.09	1.32	0.12	7.47	5.49	93
Carbon Tetrachloride	3.22	117	1	11.2	0.927	0.16	9.62	4.55	91
1,1,1-Trichloroethane	3.28	97	1	9.75	1.03	0.15	8.81	6.14	94
Methyl Acrylate	3.28	55	1	7.87	0.514	0.14	7.82	4.90	94
Tetrahydrofuran	3.28	42	1	4.70	0.241	0.20	11.4	2.86	92
Dibromofluoromethane (SURR)	3.29	111	1	7.79	0.759		2.38	3.49	104
Ethyl Acetate	3.30	43	1	9.14	0.552	0.15	8.22	2.76	90
1,1-Dichloropropene	3.39	75	1	9.02	0.991	0.09	5.72	6.69	88

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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 Calibration Check (n=7, 20 ppb)	
	Retention Time	Quant Ion	IS	RRF (≤20% RSD r^2≥0.99)	Average RRF	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
2-Butanone (MEK)	3.43	43	1	12.1	0.097	0.34	7.62	1.07	98
Benzene	3.60	78	1	5.74	3.45	0.13	7.64	6.30	92
Propionitrile	3.68	52	1	11.0	0.137	0.18	11.1	3.79	94
Methacrylonitrile	3.69	41	1	13.6	0.438	0.15	9.29	7.02	87
1,2-Dichloroethane-d4 (SURR)	3.73	65	2	3.89	0.644		2.55	3.35	97
Pentafluorobenzene (IS 1)	3.74	168							
tert-Amyl Methyl Ether (TAME)	3.76	73	1	5.16	1.99	0.15	9.49	5.08	96
1,2-Dichloroethane	3.79	62	1	7.59	1.04	0.08	3.93	6.05	92
Isobutanol	4.11	43	1	10.0	0.888	0.15	8.62	4.37	93
Isopropyl Acetate	4.12	43	1	9.21	0.880	0.16	8.66	4.33	92
Trichloroethene	4.13	95	1	8.97	0.981	0.19	10.2	6.43	90
1,4-Diflourobenzene (IS 2)	4.19								
Dibromomethane	4.51	174	2	8.49	0.449	0.13	8.79	4.73	99
1,2-Dichloropropane	4.61	63	2	7.77	0.546	0.16	8.12	5.38	90
Bromodichloromethane	4.70	83	2	9.81	0.592	0.17	9.80	5.54	91
Methyl Methacrylate	4.93	41	2	6.14	0.332	0.13	8.38	2.93	92
Propyl Acetate	5.10	43	2	9.49	0.482	0.14	7.10	5.10	92
2-Chloroethyl Vinyl Ether	5.33	63	2	8.86	0.359	0.09	5.49	4.92	89
cis-1,3-Dichloropropene	5.34	75	2	9.01	0.764	0.17	9.86	5.60	92
Toluene-d8 (SURR)	5.52	98	3	2.51	1.82		1.18	2.15	99
Toluene	5.57	91	2	7.71	3.08	0.10	5.21	6.83	92
2-Nitropropane[1]	5.84	43	2	0.999	0.133	0.05	3.66	6.78	94
Tetrachloroethene	5.96	166	2	8.34	1.39	0.16	10.3	4.62	97
4-Methyl-2-Pentanone	6.05	43	2	10.8	0.150	0.37	8.40	3.02	91
trans-1,3-Dichloropropene	6.06	75	2	8.61	0.678	0.14	9.08	5.22	93
1,1,2-Trichloroethane	6.22	97	2	7.51	0.579	0.14	9.08	5.45	94
Ethyl Methacrylate	6.32	69	2	7.41	0.632	0.19	9.94	5.14	90
Dibromochloromethane	6.39	129	2	6.14	0.581	0.11	5.34	5.28	95
1,3-Dichloropropane	6.50	76	2	6.68	0.955	0.13	7.93	5.41	95
1,2-Dibromoethane	6.60	107	2	9.71	0.537	0.18	10.7	5.89	93
Butyl Acetate	6.96	43	2	10.5	0.510	0.15	9.46	5.22	92
2-Hexanone	7.02	43	2	8.54	0.095	0.33	7.77	4.43	97
Chlorobenzene-d5 (IS 3)	7.21	117							
Chlorobenzene	7.23	112	3	5.87	1.32	0.10	6.27	5.57	96
Ethylbenzene	7.30	91	3	8.15	2.13	0.03	1.93	6.54	92
1,1,1,2-Tetrachloroethane	7.32	131	3	5.46	0.374	0.10	5.79	7.23	96
m-,p-Xylene	7.46	91	3	5.61	1.64	0.27	8.33	5.32	92
o-Xylene	7.83	91	3	5.55	1.75	0.14	8.20	6.42	94
Bromoform	7.87	173	3	11.3	0.283	0.21	14.1	5.06	93
Styrene	7.88	104	3	7.43	1.38	0.07	4.02	5.05	101
Isopropylbenzene	8.11	105	3	4.46	2.19	0.15	9.41	6.13	95
Amyl Acetate	8.28	43	3	8.03	0.351	0.08	4.56	5.12	93

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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 Calibration Check (n=7, 20 ppb)	
	Retention Time	Quant Ion	IS	RRF (≤20% RSD r^2≥0.99)	Average RRF	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
4-Bromofluorobenzene (SURR)	8.30	95	4	3.45	0.789		1.75	1.62	98
Bromobenzene	8.35	77	3	6.09	0.690	0.17	10.2	7.16	94
cis-1,4-Dichloro-2-Butene	8.38	75	3	12.7	0.157	0.14	8.91	6.25	89
n-Propylbenzene	8.42	91	3	5.43	2.47	0.16	9.44	6.71	95
1,1,2,2-Tetrachloroethane	8.49	83	3	11.7	0.367	0.19	11.4	5.46	93
2-Chlorotoluene	8.50	91	3	10.6	1.47	0.16	9.44	6.17	91
1,2,3-Trichloropropane	8.56	75	3	10.3	0.369	0.21	13.2	5.08	91
trans-1,4-dichloro-2-butene	8.56	75	3	6.46	0.354	0.21	12.9	5.06	94
1,3,5-Trimethylbenzene	8.58	105	3	7.45	1.88	0.05	3.14	6.26	94
4-Chlorotoluene	8.63	91	3	9.19	1.52	0.16	9.37	5.92	94
Pentachloroethane	8.77	77	3	17.2	2.09	0.14	7.41	4.97	88
tert-Butylbenzene	8.79	119	3	4.72	1.71	0.07	4.39	6.60	95
1,2,4-Trimethylbenzene	8.84	105	3	6.72	1.91	0.09	5.28	6.38	95
sec-Butylbenzene	8.91	105	3	6.65	2.38	0.07	4.65	6.64	95
p-Isopropyltoluene	9.01	119	3	6.05	2.13	0.12	7.65	6.59	95
1,3-Dichlorobenzene	9.02	146	3	4.16	1.17	0.09	5.88	5.17	97
1,4-Dichlorobenzene-d4 (IS 4)	9.08	152							
1,4-Dichlorobenzene	9.09	146	4	5.56	1.58	0.13	7.44	4.33	96
n-Butylbenzene	9.28	91	4	7.28	2.33	0.13	7.56	6.07	94
Hexachloroethane	9.33	201	4	9.42	0.640	0.13	18.5	5.80	97
1,2-Dichlorobenzene	9.34	146	4	4.55	1.49	0.09	5.21	4.27	99
1,2-Dibromo-3-Chloropropane	9.84	75	4	12.0	0.082	0.15	7.80	7.91	98
Nitrobenzene[1]	10.17	77	4	0.999	0.049	0.13	8.48	6.88	102
Hexachlorobutadiene	10.23	225	4	8.25	0.404	0.24	13.3	6.70	93
1,2,4-Trichlorobenzene	10.23	180	4	8.94	1.01	0.15	9.13	5.24	100
Naphthalene	10.42	128	4	4.45	3.03	0.13	7.49	3.82	96
1,2,3-Trichlorobenzene	10.52	180	4	8.72	1.01	0.14	9.35	4.25	97

1) Compound used a linear calibration.

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

Compound	Calibration (2 ppb – 200 ppb)					Method Detection Limit (n=7, 2 ppb)		Mid-Point Calibration Check (n=7, 20 ppb)	
	Retention Time	Quant Ion	IS	RRF (≤20% RSD r^2≥0.99)	Average RRF	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
Dichlorodifluoromethane	0.781	85	1	8.98	1.87	0.58	8.60	3.65	97
Chloromethane	0.876	50	1	9.99	1.02	0.65	9.53	4.87	94
Vinyl Chloride	0.912	62	1	7.30	1.32	0.67	9.99	3.83	95
Bromomethane	1.08	94	1	14.1	1.26	0.64	7.71	3.61	94
Chloroethane	1.14	64	1	11.8	1.16	0.45	6.73	4.66	97
Trichlorofluoromethane	1.22	101	1	4.56	2.81	0.51	7.92	2.58	94

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Compound	Calibration (2 ppb – 200 ppb)					Method Detection Limit (n=7, 2 ppb)		Mid-Point Calibration Check (n=7, 20 ppb)	
	Retention Time	Quant Ion	IS	RRF (≤20% RSD r^2≥0.99)	Average RRF	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
Diethyl Ether	1.40	59	1	9.13	0.867	0.40	6.31	3.53	93
1,1-Dichloroethene	1.50	61	1	7.59	1.40	0.45	7.08	4.44	92
Carbon Disulfide	1.51	76	1	8.90	1.84	0.44	7.00	3.56	88
1,1,2-Trichlorotrifluoroethane	1.53	101	1	6.77	1.26	0.47	7.06	3.09	96
Iodomethane	1.58	142	1	12.3	1.57	0.27	7.93	6.82	92
Allyl Chloride	1.81	41	1	13.9	1.20	0.58	8.47	4.16	88
Methylene Chloride	1.88	49	1	11.1	1.21	0.54	7.24	3.65	93
Acetone[1]	1.95	43	1	0.999	0.152	1.09	6.48	8.77	95
trans-1,2-Dichloroethene	1.99	61	1	7.59	1.33	0.41	6.17	2.50	94
Methyl Acetate	2.05	43	1	12.0	0.606	0.78	10.1	3.51	91
Methyl-tert-butyl Ether (MTBE)	2.10	73	1	4.97	3.35	0.31	4.79	2.81	98
tert-Butyl Alcohol (TBA)	2.24	59	1	10.5	0.153	3.48	10.7	3.39	91
Acetonitrile	2.29	41	1	10.0	0.113	0.43	6.52	5.38	100
Diisopropyl Ether	2.42	45	1	6.57	2.87	0.36	6.00	4.19	94
Chloroprene	2.45	53	1	6.73	1.45	0.59	8.52	4.28	93
1,1-Dichloroethane	2.47	63	1	5.63	2.09	0.60	9.01	2.21	95
Acrylonitrile	2.53	53	1	7.66	0.396	0.95	9.34	8.89	98
Ethyl-tert-butyl- Ether (ETBE)	2.72	59	1	5.41	3.14	0.49	8.18	2.90	97
Vinyl Acetate	2.72	43	1	8.64	1.74	0.48	7.09	2.38	95
cis-1,2-Dichloroethene	2.91	61	1	6.11	1.50	0.36	5.38	2.40	98
2,2-Dichloropropane	2.99	77	1	6.68	1.90	0.69	9.74	4.36	95
Bromochloromethane	3.07	49	1	7.71	0.774	0.56	8.38	4.35	92
Chloroform	3.16	83	1	3.93	2.44	0.53	9.73	9.43	82
Carbon Tetrachloride	3.23	117	1	7.48	1.62	0.60	9.94	6.99	82
1,1,1-Trichloroethane	3.29	97	1	7.03	1.67	0.53	8.88	6.73	91
Tetrahydrofuran	3.29	42	1	9.43	0.341	0.61	9.11	6.59	90
Methyl Acrylate	3.30	55	1	4.58	0.658	0.62	8.89	6.05	88
Dibromofluoromethane (SURR)	3.30	111	1	5.10	0.895		4.44	4.98	96
Ethyl Acetate	3.31	43	1	11.6	0.740	0.52	8.02	6.00	89
1,1-Dichloropropene	3.39	75	1	7.77	1.24	0.37	5.79	5.91	97
2-Butanone (MEK)	3.45	43	1	10.9	0.144	1.66	8.65	9.41	88
Benzene	3.61	78	1	6.70	4.17	0.46	7.10	4.10	95
Propionitrile	3.68	52	1	15.3	0.161	0.37	5.45	6.23	88
Methacrylonitrile	3.69	41	1	5.08	0.360	0.44	6.16	3.59	98
1,2-Dichloroethane-d4 (SURR)	3.73	65	2				5.21	4.36	97
Pentafluorobenzene (IS 1)	3.75	168							
tert-Amyl Methyl Ether (TAME)	3.77	73	1	6.30	2.30	0.38	6.51	3.18	97
1,2-Dichloroethane	3.80	62	1	6.04	1.11	0.36	5.63	6.77	95
Isobutanol	4.13	43	1	6.40	1.04	0.40	6.17	4.06	95
Isopropyl Acetate	4.13	43	1	6.40	1.04	0.40	6.17	4.06	95
Trichloroethene	4.13	95	1	7.37	1.23	0.63	9.57	4.58	94
1,4-Diflourobenzene (IS 2)	4.19								

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

Compound	Calibration (2 ppb – 200 ppb)					Method Detection Limit (n=7, 2 ppb)		Mid-Point Calibration Check (n=7, 20 ppb)	
	Retention Time	Quant Ion	IS	RRF (≤20% RSD r^2≥0.99)	Average RRF	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
Dibromomethane	4.51	174	2	6.59	0.519	0.25	3.79	1.92	95
1,2-Dichloropropane	4.61	63	2	4.82	0.613	0.58	7.97	4.14	95
Bromodichloromethane	4.70	83	2	9.54	0.740	0.43	6.57	3.73	94
Methyl Methacrylate	4.94	41	2	6.53	0.346	0.69	10.4	3.18	89
Propyl Acetate	5.10	43	2	9.63	0.549	0.62	9.09	3.11	96
2-Chloroethyl Vinyl Ether	5.34	63	2	0.505	0.340	0.71	10.3	3.65	93
cis-1,3-Dichloropropene	5.35	75	2	7.71	0.922	0.63	10.1	3.50	97
Toluene-d8 (SURR)	5.52	98	3	3.08	1.80		1.44	1.53	101
Toluene	5.57	91	2	7.08	3.77	0.45	6.74	3.22	94
2-Nitropropane	5.85	43	2	13.8	0.099	0.60	8.91	3.73	95
Tetrachloroethene	5.96	166	2	9.72	1.68	0.56	8.66	4.31	98
4-Methyl-2-Pentanone	6.06	43	2	5.56	0.160	1.60	8.69	4.47	98
trans-1,3-Dichloropropene	6.06	75	2	7.51	0.820	0.31	5.01	3.33	95
1,1,2-Trichloroethane	6.22	97	2	7.46	0.618	0.51	8.15	3.65	95
Ethyl Methacrylate	6.32	69	2	7.75	0.642	0.54	8.62	4.47	95
Dibromochloromethane	6.38	129	2	3.39	0.673	0.53	8.11	2.02	95
1,3-Dichloropropane	6.50	76	2	6.92	0.998	0.25	3.94	4.00	95
1,2-Dibromoethane	6.60	107	2	4.56	0.560	0.52	8.55	3.95	93
Butyl Acetate	6.97	43	2	5.81	0.571	0.41	5.99	4.49	94
2-Hexanone	7.02	43	2	5.51	0.119	1.34	7.13	6.90	89
Chlorobenzene-d5 (IS 3)	7.21	117							
Chlorobenzene	7.23	112	3	5.81	1.66	0.28	4.44	1.85	94
Ethylbenzene	7.30	91	3	6.67	2.67	0.61	9.17	3.06	93
1,1,1,2-Tetrachloroethane	7.32	131	3	6.03	0.468	0.55	8.75	2.18	96
m-,p-Xylene	7.46	91	3	6.25	2.05	0.64	4.97	2.81	94
o-Xylene	7.83	91	3	5.70	2.15	0.44	6.59	1.94	94
Bromoform	7.87	173	3	4.94	0.314	0.47	7.59	4.65	98
Styrene	7.89	104	3	7.29	1.76	0.46	7.04	2.07	93
Isopropylbenzene	8.11	105	3	5.75	2.85	0.35	5.32	2.72	95
Amyl Acetate	8.28	43	3	9.04	0.382	0.45	6.89	4.24	92
4-Bromofluorobenzene (SURR)	8.30	95	4	2.62	0.767		3.44	3.76	98
Bromobenzene	8.35	77	3	6.52	0.803	0.45	6.67	3.25	95
cis-1,4-Dichloro-2-Butene	8.38	75	3	5.73	0.153	0.60	9.14	4.08	94
n-Propylbenzene	8.42	91	3	7.25	3.23	0.21	3.25	1.79	96
1,1,2,2-Tetrachloroethane	8.50	83	3	12.1	0.370	0.38	6.39	2.77	94
2-Chlorotoluene	8.50	91	3	7.77	1.77	0.56	8.68	2.56	95
1,2,3-Trichloropropane	8.56	75	3	8.45	0.365	0.37	5.80	2.89	96
1,3,5-Trimethylbenzene	8.58	105	3	7.79	2.42	0.31	4.77	2.60	96
trans-1,4-dichloro-2-butene	8.61	75	3	4.52	0.232	0.46	6.21	3.99	97
4-Chlorotoluene	8.63	91	3	8.85	1.90	0.37	5.67	2.53	94
Pentachloroethane	8.79	77	3	11.3	0.258	0.88	12.5	4.39	89
tert-Butylbenzene	8.79	119	3	6.68	2.27	0.24	3.66	2.57	96

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

Compound	Calibration (2 ppb – 200 ppb)					Method Detection Limit (n=7, 2 ppb)		Mid-Point Calibration Check (n=7, 20 ppb)	
	Retention Time	Quant Ion	IS	RRF (≤20% RSD r^2≥0.99)	Average RRF	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
1,2,4-Trimethylbenzene	8.84	105	3	7.15	2.41	0.36	5.37	2.38	97
sec-Butylbenzene	8.91	105	3	7.71	3.19	0.35	5.37	2.88	97
p-Isopropyltoluene	9.02	119	3	6.49	2.92	0.37	5.97	2.60	96
1,3-Dichlorobenzene	9.02	146	3	8.40	1.52	0.35	5.40	2.68	97
1,4-Dichlorobenzene-d4 (IS 4)	9.08	152							
1,4-Dichlorobenzene	9.09	146	4	7.68	2.06	0.43	6.51	3.75	96
n-Butylbenzene	9.29	91	4	9.08	3.35	0.46	6.96	4.44	93
Hexachloroethane	9.33	201	4	9.80	0.948	0.28	9.24	2.92	93
1,2-Dichlorobenzene	9.35	146	4	7.13	1.87	0.27	4.11	3.71	96
1,2-Dibromo-3-Chloropropane	9.84	75	4	15.4	0.101	0.69	10.3	5.59	88
Nitrobenzene	10.17	77	4	13.3	0.063	0.71	9.50	2.74	97
Hexachlorobutadiene	10.24	225	4	8.98	0.726	0.37	5.88	5.44	95
1,2,4-Trichlorobenzene	10.24	180	4	5.15	1.31	0.20	3.15	4.46	98
Naphthalene	10.42	128	4	6.10	3.14	0.37	5.68	3.50	95
1,2,3-Trichlorobenzene	10.53	180	4	3.87	1.21	0.40	6.29	4.30	95

1) Compound used a linear calibration.

Figure 1 Total Ion Chromatogram (TIC) of a Water Method 20 ppb Standard Indicating Consistent Peak Shapes for all Compounds with Minimal Water Interference.

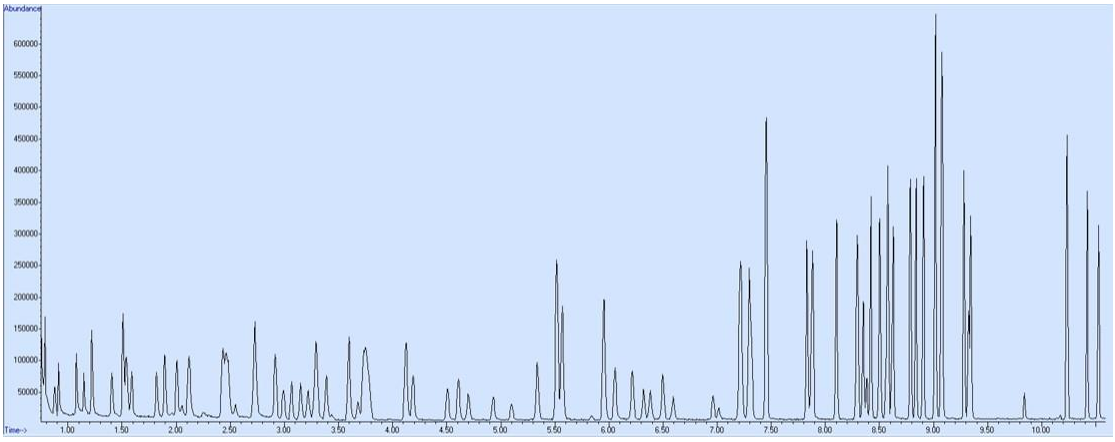

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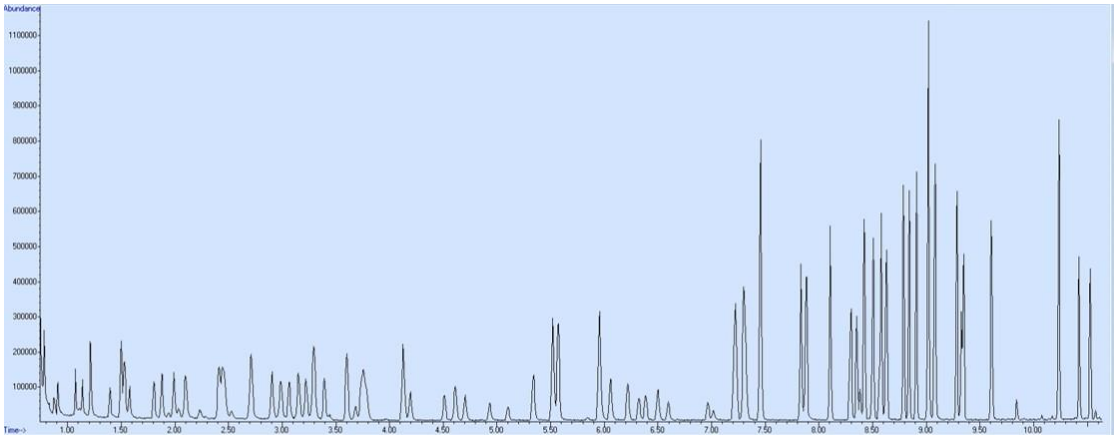
Figure 2 Total Ion Chromatogram (TIC) of a Soil Method 20 ppb Standard Indicating Consistent Peak Shapes for all Compounds with Minimal Water Interference.



Conclusion

This study demonstrates the capability of the Teledyne Tekmar Atomx XYZ P&T system to process VOCs in water and soil samples following US EPA Method 8260 with detection by an Agilent 7890B GC and 5977B MS. Additionally, economical and readily available gases such as hydrogen for carrier gas and nitrogen for purge gas, were successfully used for this analysis; and are practical alternatives for US EPA 8260.

The %RSD of the calibration curve passed all method requirements. Furthermore, MDL and precision for seven 0.5 ppb standards (water method) and seven 2 ppb standards (soil method) showed minimal interference from excessive water and resulted in an average value of 0.15 ppb for the water method and 0.55 ppb for the soil method. The mid-point verification check with precision and accuracy for seven 20 ppb water standards displayed an average of 5.4% RSD for all compounds and an average recovery of 93%. The mid-point verification check with precision and accuracy for seven 20 ppb soil standards displayed all compounds having an average of 4% RSD with an average recovery of 94%. 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

1. Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS); US EPA, Office of Solid Waste, SW-846 Method 8260D, Revision 4, February 2017. [Online] https://www.epa.gov/sites/production/files/2017-04/documents/method 8260d update_vi_final_03-13-2017.pdf (accessed March 25, 2022).