US EPA Method 8260 Using the Teledyne Tekmar Atomx XYZ with Thermo Scientific™ TRACE™ 1610 Gas Chromatograph (GC) and Thermo Scientific™ ISQ™ 7610 MS Mass Spectrometry (MS) System with ExtractaBrite™ Source

APPLICATION NOTE:	AN2206
TECHNOLOGY:	P+T VOC
INDUSTRY:	ENV

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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) VOC sample preparation system combined with a Thermo Scientific TRACE 1610 Gas Chromatograph (GC) and ISQ 7610 Mass Spectrometry (MS) system with an ExtractaBrite Source was used to create a working linear calibration curve, method detection limits (MDLs) 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) 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 0.2 parts per billion (ppb) to 200 ppb for all compounds, while the soil calibration curve was prepared from 0.5 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 0.2 ppb water standards and seven 0.5 ppb soil standards were prepared for MDL and precision calculations. Also, seven 20 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). GC-MS conditions are shown 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	140 °C	Methanol Needle Rinse	Off
Transfer Line Temp	140 °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	2.00 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	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 °C
Purge Temp	20 °C	MCS Bake Temp	200 °C
MCS Purge Temp	20 °C		
Dry Purge Time	1.00 min		
Dry Purge Flow	100 mL/min	Trap	#9
Dry Purge Temp	20 °C	Chiller Tray	Off
		Purge Gas	Nitrogen

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Table II Teledyne Tekmar Atomx XYZ Soil Method Conditions			
Standby	Variable	Purge	Variable
Valve Oven Temp	140 °C	Purge Temp	20 °C
Transfer Line Temp	140 °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	40 °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	2.00 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		2.00 min
Water Volume	10.00 mL	Bake	400 mL/min
Sweep Water Time	0.25 min	Bake Time	260 °C
Sweep Water Flow	100 mL/min	Bake Flow	180 °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 Conditions	
Column	TG-VMS, 20 m x 0.18 mm, 1µm Film, Helium – 0.8 mL/min
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, 50:1 Split, Purge Flow 0.5 mL/min
Thermo Scientific ISQ 7610 MS Conditions	
Temp	Transfer Line 230 °C; Ion Source 280 °C
Scan	Range 35 *amu* to 260 *amu*, Solvent Delay 0.50 min, Dwell/Scan Time 0.15 sec
Current	Emission Current 25 µA, Gain 3.00E+005

Results

The relative standard deviation (%RSD) of the RRFs 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 10 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 (0.2 ppb – 200 ppb)				Method Detection Limit (n=7, 0.2 ppb)		Mid-Point Check (n=7, 20 ppb)	
	Retention Time	Quant Ion	RRF (≤20% RSD r^2≥0.99)	Average RRF	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
Dichlorodifluoromethane	1.17	85	8.6	0.868	0.03	6.5	3.6	93
Chloromethane	1.32	50	5.4	1.37	0.07	6.9	4.1	85
Vinyl Chloride	1.38	62	7.7	0.565	0.04	7.3	3.4	96
Bromomethane	1.63	94	6.2	0.622	0.04	7.7	0.8	88
Chloroethane	1.72	64	18.8	0.438	0.05	6.9	2.3	113
Trichlorofluoromethane	1.85	101	7.2	1.25	0.03	5.7	2.9	101
Diethyl Ether	2.14	74	11.1	0.133	0.04	6.4	1.9	104
1,1-Dichloroethene1	2.28	61	0.999	0.082	0.11	7.8	2.6	121
1,1,2-Trichlorotrifluoroethane	2.33	101	5.3	0.191	0.06	11.2	2.6	100
Iodomethane2,8	2.38	142	11.5	0.156	0.06	8.9	9.0	40
Carbon Disulfide	2.71	76	6.7	0.081	0.07	8.3	2.0	104
Acetonitrile	2.72	41	11.2	0.311	0.07	9.1	1.8	115
Allyl Chloride	2.73	76	15.9	0.078	0.06	7.5	1.6	108
Methylene Chloride	2.81	49	15.0	0.393	0.07	7.0	1.9	109
Acetone3	2.88	58	18.5	0.046	0.67	6.4	4.7	76
trans-1,2-Dichloroethene	2.97	96	7.1	0.131	0.05	8.0	3.1	95

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Compound	Calibration (0.2 ppb – 200 ppb)				Method Detection Limit (n=7, 0.2 ppb)		Mid-Point Check (n=7, 20 ppb)	
	Retention Time	Quant Ion	RRF (≤20% RSD r^2≥0.99)	Average RRF	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
Methyl Acetate	3.04	43	12.9	0.433	0.06	8.5	2.9	126
Methyl-tert-butyl Ether (MTBE)	3.14	73	6.3	0.757	0.05	7.5	2.6	107
tert-Butyl Alcohol (TBA)4	3.34	59	15.1	0.039	0.32	8.6	6.4	129
Diisopropyl Ether	3.59	45	6.4	1.25	0.02	3.5	3.0	111
1,1-Dichloroethane	3.61	63	6.7	0.321	0.03	5.0	3.3	105
Vinyl Acetate	3.63	43	5.4	0.541	0.06	7.8	3.2	112
Acrylonitrile	3.69	53	7.3	0.141	0.08	11.4	2.4	108
Chloroprene	3.69	53	6.2	0.141	0.06	8.7	2.3	107
Ethyl-tert-butyl- Ether (ETBE)	3.96	59	6.8	0.657	0.04	6.3	2.7	113
Ethyl Acetate	3.97	88	12.8	0.021	0.10	16.4	3.4	95
cis-1,2-Dichloroethene	4.18	96	6.3	0.247	0.06	10.0	3.4	92
2,2-Dichloropropane	4.28	77	6.7	0.303	0.05	7.2	5.1	108
Bromochloromethane	4.37	128	6.5	0.118	0.05	7.9	2.1	96
Chloroform	4.48	83	6.5	0.630	0.04	6.2	2.8	100
Carbon Tetrachloride	4.58	117	3.7	0.226	0.05	9.0	3.7	99
Tetrahydrofuran	4.62	42	9.4	0.249	0.06	10.5	8.8	102
Methyl Acrylate	4.64	55	10.9	0.242	0.03	4.2	3.2	106
Dibromofluoromethane (SURR)	4.66	111	7.2	0.428		1.6	1.1	106
1,1,1-Trichloroethane	4.67	97	5.4	0.355	0.03	5.8	3.6	100
1,1-Dichloropropene	4.78	75	5.8	0.143	0.09	13.9	4.5	98
2-Butanone (MEK)5	4.81	72	14.8	0.027	0.12	7.8	4.0	84
Benzene	5.02	78	8.0	0.669	0.02	3.6	3.9	94
Propionitrile	5.05	54	11.5	0.063	0.05	7.9	2.7	106
Methacrylonitrile	5.08	41	8.9	0.404	0.12	16.6	3.0	111
1,2-Dichloroethane-d4 (SURR)	5.17	65	4.7	0.119		1.6	0.7	118
Pentafluorobenzene (IS)	5.17	168						
tert-Amyl Methyl Ether (TAME)	5.22	73						
1,2-Dichloroethane	5.26	62	6.2	0.769	0.03	4.2	3.0	106
Isobutanol	5.40	43	8.1	0.293	0.03	3.5	2.4	112
Isopropyl Acetate	5.59	43	14.3	0.031	0.06	8.8	8.8	112
Trichloroethene2	5.63	95	7.1	1.09	0.05	7.9	3.2	113
1,4-Diflourobenzene (IS)	5.67	114	13.2	0.617	0.04	2.4	4.0	96
Dibromomethane	6.03	93						
1,2-Dichloropropane	6.14	63	7.4	0.238	0.06	9.4	2.9	97
			6.2	0.327	0.05	6.9	2.6	107

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

Compound	Calibration (0.2 ppb – 200 ppb)				Method Detection Limit (n=7, 0.2 ppb)		Mid-Point Check (n=7, 20 ppb)	
	Retention Time	Quant Ion	RRF (≤20% RSD r²≥0.99)	Average RRF	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
Bromodichloromethane	6.23	83	6.6	0.610	0.04	5.6	3.2	107
Methyl Methacrylate	6.45	69	7.6	0.274	0.05	7.5	3.2	106
Propyl Acetate	6.61	43	10.0	0.876	0.06	9.5	2.9	118
2-Chloroethyl Vinyl Ether	6.86	63	5.7	0.228	0.02	2.9	3.1	121
cis-1,3-Dichloropropene	6.88	75	6.7	0.602	0.03	5.1	3.3	106
Toluene-d8 (SURR)	7.06	98	2.3	0.386		1.6	1.8	97
Toluene	7.12	92	6.6	0.642	0.07	9.6	3.8	94
2-Nitropropane	7.34	43	17.6	0.113	0.21	12.1	3.7	104
Tetrachloroethene	7.49	164	11.2	0.482	0.05	6.8	4.6	78
4-Methyl-2-Pentanone5	7.52	100	9.3	0.023	0.16	10.7	3.6	99
trans-1,3-Dichloropropene	7.55	75	6.6	0.451	0.04	6.2	3.4	105
1,1,2-Trichloroethane	7.68	83	7.4	0.327	0.02	3.8	2.9	98
Ethyl Methacrylate	7.74	69	7.9	0.434	0.05	8.6	2.4	113
Dibromochloromethane	7.83	129	9.5	0.361	0.02	3.5	3.2	105
1,3-Dichloropropane	7.91	76	7.7	0.493	0.02	3.4	2.9	101
1,2-Dibromoethane	8.00	107	6.8	0.332	0.05	7.3	2.3	96
Butyl Acetate1	8.21	43	0.997	0.756	0.02	5.2	2.7	111
2-Hexanone5	8.26	43	9.0	0.179	0.23	12.1	2.9	89
Chlorobenzene-d5 (IS)	8.45	117						
Chlorobenzene	8.46	112	6.8	0.983	0.04	6.0	3.4	96
Ethylbenzene	8.50	91	6.8	1.61	0.03	4.7	3.5	99
1,1,1,2-Tetrachloroethane	8.52	131	10.2	0.335	0.03	8.7	4.0	105
m-,p-Xylene6	8.63	106	5.8	0.652	0.10	7.8	3.9	99
o-Xylene	8.95	106	5.9	0.689	0.05	8.6	4.0	98
Bromoform	8.98	173	16.7	0.268	0.02	3.5	1.9	106
Styrene	9.00	104	4.9	1.20	0.04	5.6	3.1	99
Isopropylbenzene	9.19	105	5.3	1.74	0.04	6.4	4.2	104
Amyl Acetate1	9.31	43	0.998	0.728	0.03	8.8	3.8	94
4-Bromofluorobenzene (SURR)	9.38	95	3.0	0.843		2.5	1.7	101
cis-1,4-Dichloro-2-Butene	9.43	75	14.1	0.508	0.04	5.3	7.0	109
Bromobenzene	9.44	156	8.4	0.724	0.06	8.6	6.4	105
n-Propylbenzene	9.49	91	8.3	3.28	0.06	9.2	5.5	105
1,1,2,2-Tetrachloroethane1	9.54	83	6.4	0.654	0.05	8.8	1.4	85
2-Chlorotoluene	9.58	91	7.8	2.06	0.05	8.2	5.1	104

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

Compound	Calibration (0.2 ppb – 200 ppb)				Method Detection Limit (n=7, 0.2 ppb)		Mid-Point Check (n=7, 20 ppb)	
	Retention Time	Quant Ion	RRF (≤20% RSD r^2≥0.99)	Average RRF	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
1,2,3-Trichloropropane	9.62	75	8.4	0.603	0.06	9.7	3.9	107
1,3,5-Trimethylbenzene	9.63	105	9.0	2.40	0.06	8.7	5.8	104
trans-1,4-dichloro-2-butene	9.66	53	11.1	0.211	0.06	8.9	3.7	127
4-Chlorotoluene	9.70	91	8.8	2.21	0.08	10.5	4.5	103
tert-Butylbenzene	9.85	119	8.8	2.09	0.06	8.7	5.4	111
Pentachloroethane	9.85	77	10.6	0.200	0.07	10.2	5.4	116
1,2,4-Trimethylbenzene	9.90	105	9.2	2.46	0.07	10.4	4.7	104
sec-Butylbenzene	9.98	105	9.4	3.06	0.07	10.2	6.0	103
p-Isopropyltoluene	10.08	119	8.2	2.53	0.08	11.3	5.5	107
1,3-Dichlorobenzene	10.10	146	8.1	1.44	0.06	8.3	4.8	102
1,4-Dichlorobenzene-d4 (IS)	10.16	152						
1,4-Dichlorobenzene	10.17	146	9.8	1.55	0.07	9.0	4.7	98
n-Butylbenzene	10.36	91	10.2	2.69	0.08	9.5	5.3	110
Hexachloroethane7	10.43	117	12.3	0.831	0.02	12.7	6.8	101
1,2-Dichlorobenzene	10.46	146	8.9	1.46	0.07	9.5	4.2	104
1,2-Dibromo-3-Chloropropane1	10.99	157	0.993	0.160	0.03	8.4	2.6	94
Nitrobenzene	11.35	123	19.1	0.029	0.11	7.0	5.1	114
Hexachlorobutadiene	11.43	225	19.9	0.271	0.08	12.3	4.9	114
1,2,4-Trichlorobenzene	11.45	180	10.7	1.18	0.08	9.6	4.9	100
Naphthalene	11.66	128	10.2	3.34	0.04	5.4	3.3	105
1,2,3-Trichlorobenzene	11.78	180	9.8	1.20	0.05	6.7	4.3	99

1. Compound used a linear calibration Calibration curve from 0.5-200 ppb Calibration curve
2. from 2.5-500 ppb Calibration curve from 1-1000 ppb Calibration curve from 0.5-500 ppb
3. Calibration curve from 0.4-400 ppb Calibration curve from 0.25-100 ppb Analyte is a
4. poor purger and broke down after several injections of the mid-point check
5.
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7.
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Table V US EPA Method 8260 Soil 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=7, 20 ppb)	
	Retention Time	Quant Ion	RRF (≤20% RSD r^2≥0.99)	Average RRF	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
Dichlorodifluoromethane	1.17	85	13.5	1.40	0.05	3.2	4.2	94
Chloromethane	1.31	50	13.4	2.66	0.16	7.2	3.4	87
Vinyl Chloride	1.36	62	11.2	1.38	0.07	4.3	3.8	94
Bromomethane1	1.61	94	0.999	1.32	0.17	5.2	1.9	108
Chloroethane	1.71	64	8.6	0.868	0.10	5.6	3.8	76
Trichlorofluoromethane	1.84	101	13.2	1.96	0.05	3.1	3.7	100
Diethyl Ether	2.14	74	7.0	0.684	0.07	3.8	2.4	105
1,1-Dichloroethene	2.27	61	15.0	1.81	0.11	5.2	3.2	97
1,1,2-Trichlorotrifluoroethane	2.33	101	13.8	1.30	0.06	3.8	3.5	102
Iodomethane1	2.38	142	0.998	1.10	0.02	5.5	6.9	95
Acetonitrile	2.70	41	12.2	3.68	0.11	5.6	3.2	101
Allyl Chloride	2.71	76	12.0	0.764	0.11	5.8	3.1	99
Carbon Disulfide	2.71	76	13.3	0.768	0.11	5.7	3.2	98
Methylene Chloride1	2.81	49	0.999	2.80	0.14	4.3	2.6	121
Acetone1,2	2.90	58	0.997	0.138	2.32	6.4	3.1	128
trans-1,2-Dichloroethene	2.99	96	10.3	1.51	0.09	4.7	3.2	96
Methyl Acetate1	3.05	43	0.999	1.81	0.12	5.2	2.6	110
Methyl-tert-butyl Ether (MTBE)	3.15	73	6.0	1.41	0.09	5.7	3.3	111
tert-Butyl Alcohol (TBA)3	3.35	59	14.0	0.087	0.43	3.8	3.6	91
Diisopropyl Ether	3.59	45	10.2	3.00	0.06	3.9	3.1	103
Vinyl Acetate	3.59	43	10.5	1.32	0.08	5.0	3.3	105
Chloroprene	3.60	53	12.0	0.962	0.08	5.4	4.8	104
1,1-Dichloroethane	3.62	63	11.0	1.26	0.08	4.5	4.0	100
Acrylonitrile	3.67	53	11.5	0.253	0.12	6.2	3.0	98
Ethyl Acetate	3.96	88	16.7	0.038	0.19	15.3	3.1	104
Ethyl-tert-butyl- Ether (ETBE)	3.97	59	13.1	1.55	0.09	6.8	3.6	101
cis-1,2-Dichloroethene	4.18	96	8.5	0.796	0.08	4.6	3.9	94
2,2-Dichloropropane	4.28	77	9.8	0.869	0.07	4.5	4.2	98
Bromochloromethane	4.37	128	11.0	0.327	0.08	4.3	3.7	95
Chloroform	4.48	83	10.4	1.23	0.08	4.1	3.7	97
Carbon Tetrachloride	4.58	117	13.9	0.786	0.09	6.8	4.0	99
Tetrahydrofuran	4.65	42	4.7	0.518	0.06	4.0	6.5	95
Methyl Acrylate	4.65	55	9.3	0.452	0.05	3.3	2.7	97
1,1,1-Trichloroethane	4.65	97	9.9	0.975	0.04	2.9	4.8	99

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Table V US EPA Method 8260 Soil 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=7, 20 ppb)	
	Retention Time	Quant Ion	RRF (≤20% RSD r^2≥0.99)	Average RRF	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
Dibromofluoromethane (SURR)	4.66	111	5.1	0.427		4.9	1.7	107
1,1-Dichloropropene	4.78	75	9.1	0.819	0.08	5.3	4.2	98
2-Butanone (MEK)2	4.80	72	4.8	0.045	0.42	8.3	3.9	94
Benzene	5.03	78	5.1	2.38	0.05	3.1	3.7	98
Propionitrile	5.06	54	13.1	0.099	0.17	8.6	4.0	93
Methacrylonitrile	5.09	41	4.5	0.594	0.12	6.3	3.4	100
1,2-Dichloroethane-d4 (SURR)	5.16	65	12.2	0.140		3.3	1.4	104
Pentafluorobenzene (IS)	5.17	168						
tert-Amyl Methyl Ether (TAME)	5.22	73	10.5	1.27	0.11	7.3	3.9	102
1,2-Dichloroethane	5.24	62	9.4	0.704	0.06	3.0	2.8	103
Isobutanol	5.40	43	7.6	0.058	0.18	14.0	5.7	93
Isopropyl Acetate	5.58	43	6.9	1.56	0.07	4.8	2.6	102
Trichloroethene	5.62	95	9.0	1.45	0.09	6.0	2.7	103
1,4-Diflourobenzene (IS)	5.68	114						
Dibromomethane	6.03	93	9.4	0.388	0.04	2.3	2.8	96
1,2-Dichloropropane	6.15	63	5.1	0.626	0.08	5.3	3.2	101
Bromodichloromethane	6.23	83	6.1	0.812	0.05	3.3	2.8	101
Methyl Methacrylate	6.45	69	10.3	0.343	0.12	7.8	3.2	92
Propyl Acetate	6.62	43	8.2	1.25	0.10	6.0	2.8	97
2-Chloroethyl Vinyl Ether	6.87	63	11.3	0.272	0.05	3.8	2.7	100
cis-1,3-Dichloropropene	6.88	75	9.7	0.905	0.05	3.9	3.0	103
Toluene-d8 (SURR)	7.06	98	1.9	0.386		1.8	1.2	98
Toluene	7.12	92	4.5	1.28	0.07	4.9	4.4	93
2-Nitropropane	7.33	43	4.4	0.151	0.16	9.9	4.9	102
Tetrachloroethene	7.49	164	5.4	0.675	0.08	5.0	4.7	93
4-Methyl-2-Pentanone2	7.53	100	7.9	0.024	0.30	7.6	3.5	88
trans-1,3-Dichloropropene	7.55	75	9.4	0.552	0.06	4.5	3.5	102
1,1,2-Trichloroethane	7.69	83	9.1	0.319	0.08	5.3	3.6	101
Ethyl Methacrylate	7.75	69	6.9	0.450	0.07	4.8	3.1	95
Dibromochloromethane	7.83	129	13.2	0.340	0.07	5.4	3.2	103
1,3-Dichloropropane	7.91	76	8.4	0.573	0.07	5.1	3.1	101
1,2-Dibromoethane	8.00	107	9.2	0.360	0.06	4.2	3.0	99
Butyl Acetate	8.22	43	11.1	0.898	0.04	2.8	2.8	100
2-Hexanone2	8.26	43	13.1	0.202	0.16	3.1	3.2	88

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Compound	Calibration (0.5 ppb – 200 ppb)				Method Detection Limit (n=7, 0.5 ppb)		Mid-Point Check (n=7, 20 ppb)	
	Retention Time	Quant Ion	RRF (≤20% RSD r^2≥0.99)	Average RRF	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
Chlorobenzene-d5 (IS)	8.45	117	5.0					
Chlorobenzene	8.46	112	7.2	1.33	0.05	2.9	3.5	95
Ethylbenzene	8.51	91	16.0	2.33	0.06	3.5	4.3	96
1,1,1,2-Tetrachloroethane	8.52	131	9.3	0.347	0.10	7.5	4.0	101
m-,p-Xylene4	8.63	106	6.2	0.947	0.12	3.9	4.1	98
o-Xylene	8.95	106	17.2	0.919	0.04	2.9	3.6	99
Bromoform	8.98	173	4.3	0.224	0.06	5.1	3.7	99
Styrene	8.99	104	8.8	1.46	0.05	3.1	3.3	98
Isopropylbenzene	9.19	105	0.997	2.25	0.08	5.7	4.2	102
Amyl Acetate1	9.31	43	3.4	0.766	0.04	4.1	3.9	79
4-Bromofluorobenzene (SURR)	9.37	95	6.3	0.892		1.1	1.8	101
cis-1,4-Dichloro-2-Butene	9.44	75	3.8	0.567	0.12	6.2	3.0	102
Bromobenzene	9.44	156	15.1	0.830	0.04	2.5	4.5	96
n-Propylbenzene	9.49	91	5.9	4.64	0.06	4.0	6.4	100
1,1,2,2-Tetrachloroethane	9.55	83	6.0	0.434	0.08	4.6	4.7	108
2-Chlorotoluene	9.58	91	4.1	2.69	0.05	3.2	5.7	102
1,2,3-Trichloropropane	9.62	75	5.3	0.606	0.04	2.3	4.9	96
1,3,5-Trimethylbenzene	9.64	105	6.2	3.03	0.12	7.8	6.8	97
trans-1,4-dichloro-2-butene	9.66	53	5.1	0.288	0.06	4.3	4.8	102
4-Chlorotoluene	9.71	91	7.5	2.84	0.05	3.3	5.7	100
tert-Butylbenzene	9.85	119	8.1	2.56	0.11	7.8	6.0	105
Pentachloroethane	9.85	77	7.9	0.281	0.13	8.6	6.8	106
1,2,4-Trimethylbenzene	9.90	105	8.7	3.08	0.09	5.7	5.8	96
sec-Butylbenzene	9.98	105	6.5	3.97	0.09	6.4	5.9	104
p-Isopropyltoluene	10.08	119	4.2	3.18	0.09	5.9	6.6	102
1,3-Dichlorobenzene	10.11	146		1.66	0.06	3.3	5.1	97
1,4-Dichlorobenzene-d4 (IS)	10.16	152						
1,4-Dichlorobenzene	10.17	146	8.2	1.72	0.07	3.7	5.3	96
n-Butylbenzene	10.36	91	10.5	3.45	0.06	4.0	6.8	102
Hexachloroethane5	10.44	117	17.5	0.842	0.05	7.0	5.9	92
1,2-Dichlorobenzene	10.45	146	5.5	1.50	0.05	3.2	4.6	100
1,2-Dibromo-3-Chloropropane	10.99	157	9.7	0.146	0.07	5.5	6.0	97
Nitrobenzene6	11.36	123	6.0	0.026	0.35	13.4	5.8	93
Hexachlorobutadiene	11.43	225	9.0	0.470	0.07	4.5	6.9	104

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Compound	Calibration (0.5 ppb – 200 ppb)				Method Detection Limit (n=7, 0.5 ppb)		Mid-Point Check (n=7, 20 ppb)	
	Retention Time	Quant Ion	RRF (≤20% RSD r^2≥0.99)	Average RRF	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
1,2,4-Trichlorobenzene	11.45	180	18.7	1.19	0.10	5.5	6.0	95
Naphthalene[6]	11.66	128	11.1	2.74	0.12	5.8	4.4	105
1,2,3-Trichlorobenzene	11.78	180	18.0	1.13	0.11	6.1	4.6	95

1. Compound used linear calibration
2. Calibration from 1.25-500 ppb
3. Calibration from 2.5-1000 ppb
4. Calibration from 1-400 ppb
5. Calibration from 0.25-100 ppb
6. Calibration from 1-200 ppb

Figure 1 Total Ion Chromatogram (TIC) of a US EPA 8260 Water Method 10 ppb VOC Standard Indicating Consistent Peak Shapes for all Compounds with Minimal Water Interference.

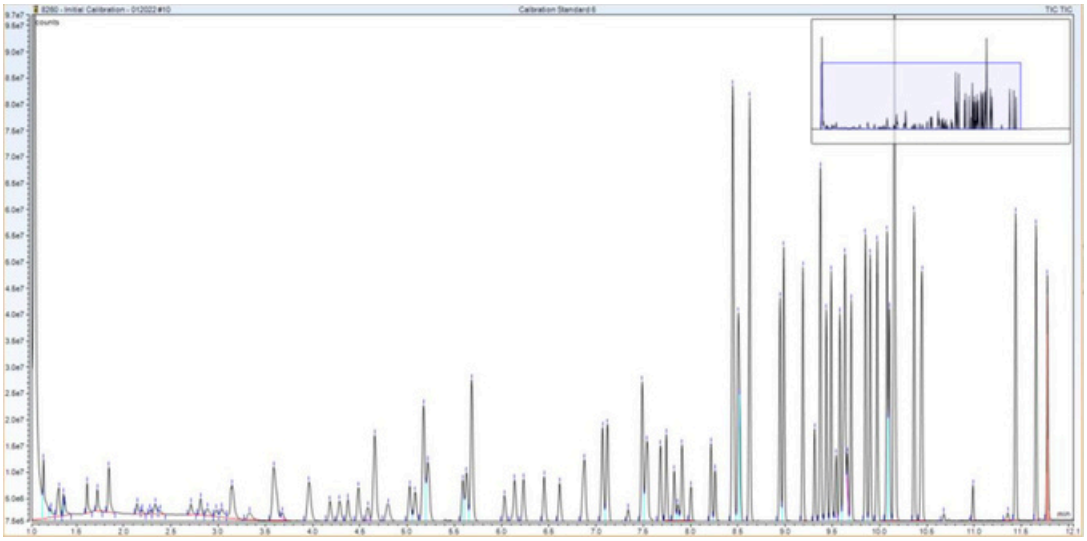

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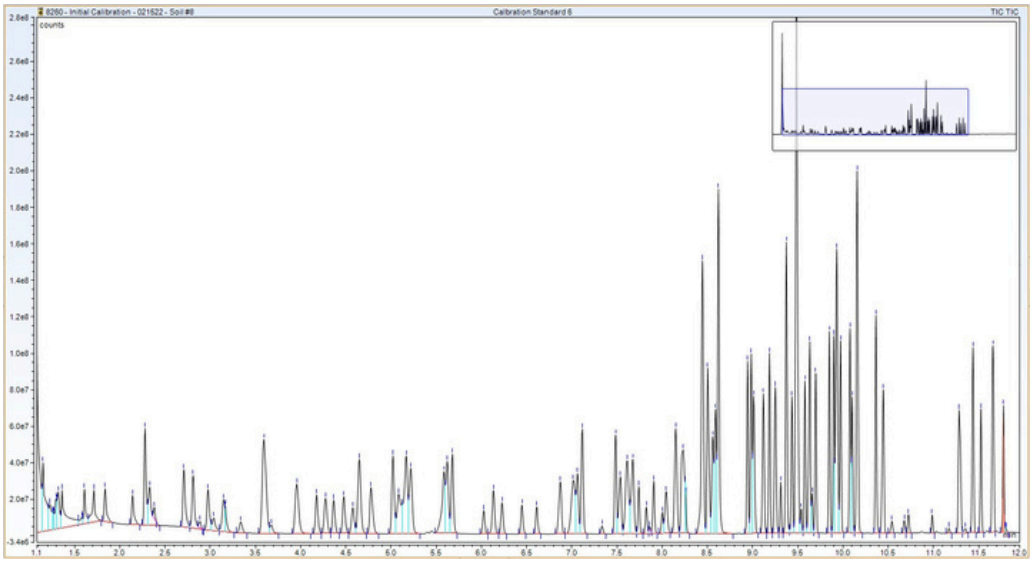
Figure 2 Total Ion Chromatogram (TIC) of a US EPA 8260 Soil Method 10 ppb VOC Standard Indicating Consistent Peak Shapes for all Compounds with Minimal Water Interference.



Conclusion

This study demonstrates the capability of the Teledyne Tekmar Atomx XYZ P&T system to process VOCs in water and soil samples following US EPA Method 8260, in conjunction with Methods 5030 and 5035, with detection by a Thermo Scientific TRACE 1610 GC and ISQ 7610 MS with an ExtractaBrite Source. The %RSD of the calibration curve passed all method requirements. Furthermore, MDL and precision for seven 0.2 ppb standards for the water method, and seven 0.5 ppb standards for the soil method, showed minimal interference from excessive water and resulted in values <0.25 ppb for all water method compounds and most soil method compounds. The mid-point calibration check with precision and accuracy for seven 20 ppb water standards displayed an average of 3.7% RSD and an average recovery of 103% for compounds of interest. The mid-point calibration check with precision and accuracy for seven 20 ppb soil standards displayed an average of 4% RSD and an average recovery of 99% 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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