US EPA Method 8260 Using the Teledyne Tekmar Atomx XYZ and Thermo Scientific™ TRACE™ 1310 GC and ISQ™ 7000 MS System with an ExtractaBrite Source

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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.[1,2,3,4,5,6] The Teledyne Tekmar Atomx XYZ purge and trap (P&T) system along with a Thermo Scientific™ TRACE™ 1310 Gas Chromatograph (GC) and ISQ™ 7000 Mass Spectrometer (MS) with an ExtractaBrite source was used to create a working linear calibration curve, method detection limits (MDLs) and an initial demonstration of capability (IDC) 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 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 96 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 1 ppb to 200 ppb. 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 4-Bromofluorobenzene. Internal and surrogate standards were prepared together in methanol from Restek standards at a concentration of 25 parts per million (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 standards and seven 1 ppb soil standards were prepared for MDL and precision calculations. Seven 20 ppb water and soil standards were prepared for the assessment of the IDC and precision and accuracy. All calibration, MDL and IDC 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 ºC	Methanol Needle Rinse	Off
Transfer Line Temp	140 ºC	Methanol Needle Rinse Volume	0.00 mL
Sample Mount Temp	90 ºC	Water Needle Rinse Volume	7.00 mL
Water Heater Temp	90 ºC	Sweep Needle Time	0.25 min
Sample Vial Temp	20 ºC	Dry Purge Temp	20 ºC
Soil Valve Temp	100 ºC	Desorb Preheat Temp	245 ºC
Standby Flow	10 mL/min	GC Start Signal	Begin Desorb
Condensate Ready Temp	45 ºC	Desorb Time	2.00 min
Purge Ready Temp	40 ºC	Drain Flow	300 mL/min
Purge	**Variable**	Desorb Temp	250 ºC
Sample Equilibrate Time	0.00 min	**Bake**	**Variable**
Pre-sweep Time	0.25 min	Methanol Glass Rinse	Off
Prime Sample Fill Volume	3.00 mL	Number of Methanol Glass Rinses	0
Sample Volume	5.00 mL	Methanol Glass Rinse Volume	0.00 mL
Sweep Sample Time	0.25 min	Water Bake Rinses	1
Sweep Sample Flow	100 mL/min	Water Bake Rinse Volume	7.00 mL
Sparge Vessel Heater	Off	Bake Rinse Sweep Time	0.25 min
Sparge Vessel Temp	20 ºC	Bake Rinse Sweep Flow	100 mL/min
Pre-purge Time	0.00 min	Bake Rinse Drain Time	0.40 min
Pre-purge Flow	0 mL/min	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	Condensate Bake Temp	200 ºC
Condensate Purge Temp	20 ºC		
Dry Purge Time	1.00 min	**Trap**	#9
Dry Purge Flow	100 mL/min	**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	Condensate Purge Temp	20 ºC
Sample Mount Temp	90 ºC	Dry Purge Time	2.00 min
Water Heater Temp	90 ºC	Dry Purge Flow	100 mL/min
Sample Vial Temp	40 ºC	Dry Purge Temp	20 ºC
Soil Valve Temp	100 ºC	**Desorb**	**Variable**
Standby Flow	10 mL/min	Methanol Needle Rinse	Off
Condensate Ready Temp	45 ºC	Methanol Needle Rinse Volume	0.00 mL
Purge Ready Temp	40 ºC	Water Needle Rinse Volume	7.00 mL
Purge	**Variable**	Sweep Needle Time	0.25 min
Pre-purge Time	0.00 min	Desorb Preheat Temp	245 ºC
Pre-Purge Flow	0 mL/min	GC Start Signal	Begin Desorb
Pre-heat Mix Speed	Slow	Desorb Time	2.00 min
Sample Pre-heat Time	0.00 min	Drain Flow	300 mL/min
Pre-sweep 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	400 mL/min
Sparge Vessel Heater	Off	Bake Temp	280 ºC
Purge Mix Speed	Medium	Condensate Bake Temp	200 ºC
Purge Time	11.00 min	**Trap**	#9
Purge Flow	40 mL/min	**Purge Gas**	Nitrogen

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Table III Thermo Scientific TRACE 1310 GC and ISQ 7000 MS System Conditions	
Thermo Scientific TRACE 1310 GC	
Column	Rtx® VMS, 20 m x 0.18 mm, 1µm Film, Helium – 0.8 mL/min
Oven Profile	35 ºC, 3 min, 12 ºC/min to 85 ºC, 25 ºC/min to 225 ºC, 2 min Hold, Run Time 14.767 min
Inlet	200 ºC, 50:1 Split, Purge Flow 0.5 mL/min
Thermo Scientific ISQ 7000 MS	
Temp	Transfer Line 230 ºC; Ion Source 280 ºC
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 RFs for the calibration curve, MDL, precision and IDC accuracy and precision data are shown in Table IV (water) and Table V (soil). Figure 1 (water) and Figure 2 (soil) display a 5 ppb standard, indicating excellent peak resolution with minimal water inference for all VOCs. An example of the linearity of the water calibration curve for chloromethane at the lowest point in the curve (0.2 ppb) is shown in Figure 3. The linearity of the soil calibration curve for dibromomethane at the lowest point in the curve (1 ppb) is displayed in Figure 4.

Table IV US EPA Method 8260 - Water Calibration, Method Detection Limit and Initial Demonstration of Capability Data

Compound	Retention Time	Calibration (0.5 ppb – 200 ppb)		Method Detection Limit (n=7, 0.5 ppb)		Initial Demonstration of Capability (n=7, 20 ppb)	
		Linearity RF (≤20% RSD r^2≥0.99)	Average RF	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
Dichlorodifluoromethane[1,9]	1.39	0.998	0.266	0.10	8.9	7.8	66
Chloromethane[1,5,7]	1.57	0.994	0.628	0.20	9.2	6.3	84
Vinyl Chloride	1.65	8.76	0.218	0.13	9.9	6.1	79
Bromomethane[4,7]	1.95	19.0	0.268	0.18	7.7	4.9	78
Chloroethane	2.08	4.62	0.182	0.07	5.0	5.1	75
Trichlorofluoromethane	2.21	16.7	0.478	0.10	7.1	6.4	82
Diethyl Ether	2.50	8.11	0.203	0.08	5.6	5.7	83
1,1-Dichloroethene	2.66	5.71	0.304	0.09	6.2	6.8	80
1,1,2-Trichlorotrifluoroethane	2.71	18.1	0.305	0.11	8.3	7.0	82
Iodomethane[1,6,8]	2.78	0.999	0.267	6.73	11.6	11.6	92
Allyl Chloride	3.14	12.9	0.188	0.08	5.8	4.9	80
Carbon Disulfide	3.14	12.2	0.187	0.08	5.6	4.8	81
Methylene Chloride	3.24	19.0	0.506	0.10	6.3	3.4	79
trans-1,2-Dichloroethene	3.41	5.30	0.365	0.13	8.4	6.5	86
Methyl Acetate	3.46	8.65	0.144	0.25	18.0	5.5	81
Methyl-tert-butyl Ether (MTBE)	3.56	14.0	0.39	0.11	9.7	6.3	85

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Compound	Retention Time	Calibration (0.5 ppb – 200 ppb)		Method Detection Limit (n=7, 0.5 ppb)		Initial Demonstration of Capability (n=7, 20 ppb)	
		Linearity RF (≤20% RSD r^2≥0.99)	Average RF	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
tert-Butyl Alcohol (TBA)	3.72	7.07	0.013	0.47	7.2	5.9	73
Acetonitrile[1,4,7]	3.78	0.999	0.047	0.74	4.3	7.9	94
Diisopropyl Ether	3.96	8.89	0.388	0.09	7.5	5.0	83
Acrylonitrile	4.01	15.0	0.296	0.12	10.2	6.3	90
Chloroprene	4.01	15.2	0.296	0.12	9.9	6.3	90
Propionitrile	4.01	18.7	0.380	0.12	10.0	7.2	97
1,1-Dichloroethane	4.03	10.0	0.483	0.09	6.1	5.4	86
Ethyl-tert-butyl- Ether (ETBE)	4.32	17.2	0.425	0.10	8.0	5.3	102
Vinyl Acetate	4.33	15.4	0.283	0.07	10.3	8.9	90
cis-1,2-Dichloroethene	4.57	6.15	0.464	0.08	5.2	5.6	92
2,2-Dichloropropane	4.67	11.3	0.415	0.10	7.2	6.0	84
Bromochloromethane	4.76	3.47	0.213	0.08	6.4	4.8	75
Chloroform	4.86	2.42	0.606	0.07	5.1	5.3	74
Carbon Tetrachloride	4.96	15.5	0.469	0.10	7.8	5.9	87
Methyl Acrylate	5.02	16.8	0.143	0.08	7.1	5.9	83
Ethyl Acetate	5.03	19.1	0.174	0.06	5.7	7.9	80
Dibromofluoromethane (SURR)	5.03	6.38	0.260		2.1	9.2	96
1,1,1-Trichloroethane	5.04	11.8	0.509	0.09	6.9	15.1	90
Tetrahydrofuran	5.04	15.5	0.100	0.08	7.1	6.8	77
1,1-Dichloropropene	5.16	14.6	0.324	0.09	7.5	5.7	90
2-Butanone (MEK)[3]	5.19	9.14	0.034	0.17	4.0	4.6	76
Benzene	5.40	4.39	1.14	0.08	6.4	6.1	85
Methacrylonitrile	5.48	6.31	0.139	0.10	8.9	5.4	81
Pentafluorobenzene (IS)	5.53						
1,2-Dichloroethane-d4 (SURR)	5.54	10.4	0.088		3.0	1.8	102
tert-Amyl Methyl Ether (TAME)	5.56	9.82	0.396	0.08	6.2	4.6	8
1,2-Dichloroethane	5.61	7.29	0.482	0.12	8.2	5.3	91
Isobutanol[4,7]	5.68	13.3	0.008	2.1	14.3	7.2	77
Isopropyl Acetate	5.93	17.9	0.354	0.06	4.1	5.8	103
Trichloroethene	5.99	10.6	0.438	0.11	8.8	7.4	90
1,4-Diflourobenzene (IS)	6.04						
Dibromomethane	6.39	5.00	0.289	0.09	6.4	5.5	90
1,2-Dichloropropane	6.49	8.26	0.312	0.07	5.2	5.9	94
Bromodichloromethane	6.58	4.05	0.557	0.11	7.9	5.6	91
Methyl Methacrylate	6.78	17.6	0.217	0.14	11.1	6.5	94
Propyl Acetate	6.94	15.9	0.284	0.15	13.1	6.1	83
2-Chloroethyl Vinyl Ether	7.18	18.2	0.112	0.08	5.3	5.7	110
cis-1,3-Dichloropropene	7.21	9.53	0.434	0.04	3.8	4.8	88
Toluene-d8 (SURR)	7.39	2.69	0.345		1.9	1.1	106
Toluene	7.43	5.30	1.77	0.07	4.9	6.9	88

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Compound	Retention Time	Calibration (0.5 ppb – 200 ppb)		Method Detection Limit (n=7, 0.5 ppb)		Initial Demonstration of Capability (n=7, 20 ppb)	
		Linearity RF (≤20% RSD r^2≥0.99)	Average RF	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
2-Nitropropane	7.66	11.6	0.085	0.10	6.1	5.7	90
Tetrachloroethene	7.78	19.5	0.822	0.15	11.1	15.5	106
4-Methyl-2-Pentanone	7.82	13.6	0.082	0.30	9.2	5.4	81
trans-1,3-Dichloropropene	7.83	17.0	0.494	0.09	7.2	6.0	94
1,1,2-Trichloroethane	7.96	11.7	0.277	0.08	6.4	6.1	92
Ethyl Methacrylate	8.00	12.2	0.355	0.07	6.3	5.9	88
Dibromochloromethane	8.10	13.0	0.449	0.09	7.6	5.6	90
1,3-Dichloropropane	8.18	10.9	0.553	0.04	3.3	5.1	91
1,2-Dibromoethane	8.28	11.7	0.386	0.05	4.0	6.0	91
Butyl Acetate	8.45	12.8	0.324	0.09	6.9	6.5	85
2-Hexanone	8.51	12.0	0.052	0.35	10.7	5.6	86
Chlorobenzene-d5 (IS)	8.69						
Chlorobenzene	8.70	4.14	1.25	0.09	6.0	6.2	88
Ethylbenzene	8.74	5.39	1.83	0.07	4.8	6.5	91
1,1,1,2-Tetrachloroethane	8.76	5.23	0.312	0.09	7.0	5.1	80
m-,p-Xylene	8.85	7.21	0.634	0.18	6.9	7.4	90
o-Xylene	9.17	6.84	0.613	0.06	4.4	7.0	84
Styrene	9.21	6.48	0.996	0.09	7.7	6.3	87
Bromoform	9.22	9.40	0.290	0.08	7.3	7.1	81
Isopropylbenzene	9.40	9.90	1.48	0.07	5.8	7.4	86
Amyl Acetate	9.51	7.03	0.329	0.06	5.4	7.8	80
4-Bromofluorobenzene (SURR)	9.59	4.64	0.525		2.0	1.9	100
Bromobenzene	9.66	6.00	0.942	0.10	7.4	6.8	87
1,3,5-Trimethylbenzene	9.69	13.7	0.092	0.19	16.0	7.5	92
n-Propylbenzene	9.69	12.0	2.30	0.08	6.8	6.6	90
1,1,2,2-Tetrachloroethane	9.75	7.56	0.444	0.12	9.4	5.6	83
2-Chlorotoluene	9.79	6.88	1.60	.09	7.1	6.3	85
1,2,3-Trichloropropane	9.83	7.57	0.372	0.12	9.3	6.5	86
cis-1,4-Dichloro-2-Butene	9.87	9.99	0.164	0.09	7.7	4.5	90
trans-1,4-dichloro-2-butene	9.87	11.2	0.124	0.13	10.1	8.5	85
4-Chlorotoluene	9.91	8.64	1.56	0.09	7.6	6.7	89
Pentachloroethane	10.04	17.0	0.160	0.11	9.2	7.5	95
tert-Butylbenzene	10.05	12.9	1.69	0.14	11.5	6.1	92
1,2,4-Trimethylbenzene	10.09	18.8	1.61	0.10	7.6	6.6	113
sec-Butylbenzene	10.17	16.0	2.12	0.09	8.0	6.8	94
p-Isopropyltoluene[2]	10.26	17.3	1.76	0.09	7.5	6.6	115
1,3-Dichlorobenzene	10.31	5.59	1.47	0.08	5.9	6.6	84
1,4-Dichlorobenzene-d4 (IS)	10.36						
1,4-Dichlorobenzene	10.37	6.68	1.57	0.09	6.9	6.2	82
n-Butylbenzene	10.55	17.7	1.48	0.11	7.9	6.8	113

Table IV US EPA Method 8260 - Water Calibration, Method Detection Limit and Initial Demonstration of Capability Data							
Compound	Retention Time	Calibration (0.5 ppb – 200 ppb)		Method Detection Limit (n=7, 0.5 ppb)		Initial Demonstration of Capability (n=7, 20 ppb)	
		Linearity RF (≤20% RSD r^2≥0.99)	Average RF	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
Hexachloroethane	10.63	11.9	0.549	0.07	11.6	7.0	81
1,2-Dichlorobenzene	10.65	5.56	1.47	0.07	5.3	5.5	82
1,2-Dibromo-3-Chloropropane	11.18	8.11	0.172	0.14	10.1	7.6	81
Nitrobenzene	11.56	12.0	0.022	0.14	9.0	7.2	71
Hexachlorobutadiene	11.61	8.19	0.064	0.14	10.8	7.6	84
1,2,4-Trichlorobenzene	11.63	8.94	0.972	0.12	8.1	6.5	85
Naphthalene	11.85	6.32	1.95	0.07	5.6	8.4	89
1,2,3-Trichlorobenzene	11.97	9.86	0.961	0.10	7.2	7.4	86

1. Linear calibration
2. Calibration range 0.5-200 ppb
3. Calibration range 1.25-500 ppb
4. Calibration range 2-200 ppb
5. Calibration range 5-200 ppb
6. Calibration range 10-200 ppb
7. 5 ppb MDL
8. 20 ppb MDL
9. Compound displayed interference with the CO_2 peak during desorb

Table V US EPA Method 8260 Soil - Calibration, Method Detection Limit and Initial Demonstration of Capability Data							
Compound	Retention Time	Calibration (1 ppb – 200 ppb)		Method Detection Limit (n=7, 1 ppb)		Initial Demonstration of Capability (n=7, 20 ppb)	
		Linearity RF (≤20% RSD r^2≥0.99)	Average RF	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
Dichlorodifluoromethane[1]	1.39	0.999	0.192	0.19	5.9	4.4	92
Chloromethane[1,3]	1.56	0.993	0.471	0.59	3.2	5.1	117
Vinyl Chloride	1.64	8.84	0.180	0.17	5.4	4.3	91
Bromomethane[1,3]	1.94	0.996	0.266	0.48	2.8	5.6	110
Chloroethane	2.06	7.97	0.142	0.23	6.6	4.9	97
Trichlorofluoromethane	2.20	6.73	0.444	0.17	5.9	4.6	87
Diethyl Ether	2.49	4.17	0.156	0.22	6.7	4.4	97
1,1-Dichloroethene	2.64	6.85	0.270	0.11	3.8	3.6	91
1,1,2-Trichlorotrifluoroethane	2.69	4.88	0.292	0.14	5.2	4.0	87
Iodomethane[1,4,5]	2.77	0.996	0.467	3.39	4.6	4.6	116
Allyl Chloride	3.12	5.09	0.156	0.11	3.3	4.1	96
Carbon Disulfide	3.12	5.53	0.156	0.13	3.8	4.0	97
Methylene Chloride	3.23	18.4	0.398	0.25	5.5	2.8	85
trans-1,2-Dichloroethene	3.39	3.73	0.344	0.09	2.9	4.0	95
Methyl Acetate	3.46	7.91	0.135	0.51	13.3	3.0	92

Table V US EPA Method 8260 Soil - Calibration, Method Detection Limit and Initial Demonstration of Capability Data							
Compound	Retention Time	Calibration (1 ppb – 200 ppb)		Method Detection Limit (n=7, 1 ppb)		Initial Demonstration of Capability (n=7, 20 ppb)	
		Linearity RF (≤20% RSD r²≥0.99)	Average RF	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
Methyl-tert-butyl Ether (MTBE)	3.55	2.27	0.333	0.17	5.8	4.0	101
tert-Butyl Alcohol (TBA)	3.78	7.40	0.028	1.95	9.0	5.6	95
Acetonitrile[1,2]	3.83	0.999	0.024	1.16	6.6	5.0	105
Diisopropyl Ether	3.96	2.94	0.319	0.15	5.0	4.2	106
Acrylonitrile	4.00	11.5	0.296	0.14	5.7	4.4	100
Chloroprene	4.00	12.0	0.297	0.13	5.4	4.5	100
Propionitrile	4.00	17.0	0.399	0.12	5.4	4.0	98
1,1-Dichloroethane	4.03	5.72	0.407	0.14	4.0	3.9	100
Ethyl-tert-butyl- Ether (ETBE)	4.33	16.7	0.368	0.20	8.2	4.3	96
Vinyl Acetate	4.33	1.92	0.285	0.10	3.6	9.5	103
cis-1,2-Dichloroethene	4.57	4.53	0.413	0.11	3.3	3.8	103
2,2-Dichloropropane	4.67	6.35	0.388	0.15	4.8	4.2	101
Bromochloromethane	4.76	5.22	0.163	0.09	2.9	3.3	87
Chloroform	4.86	5.48	0.493	0.19	5.7	3.6	94
Carbon Tetrachloride	4.96	5.87	0.437	0.12	4.2	3.9	99
Methyl Acrylate	5.02	7.60	0.144	0.31	9.2	5.6	111
1,1,1-Trichloroethane	5.03	4.64	0.463	0.12	3.9	3.7	102
Dibromofluoromethane (SURR)	5.03	4.02	0.283		4.9	3.9	96
Ethyl Acetate	5.03	8.35	0.202	0.32	9.7	4.1	101
Tetrahydrofuran	5.04	16.6	0.105	0.14	4.2	3.8	95
1,1-Dichloropropene	5.16	3.69	0.320	0.15	5.6	3.7	107
2-Butanone (MEK)[1]	5.20	1.00	0.042	0.99	12.7	7.0	108
Benzene	5.40	4.13	0.984	0.12	4.0	3.6	102
Methacrylonitrile	5.48	3.25	0.119	0.14	4.5	4.8	102
Pentafluorobenzene (IS)	5.53						
1,2-Dichloroethane-d4 (SURR)	5.54	10.4	0.076		1.5	2.3	108
tert-Amyl Methyl Ether (TAME)	5.57	3.89	0.307	0.13	4.6	4.1	96
1,2-Dichloroethane	5.61	5.19	0.370	0.15	4.5	2.9	97
Isobutanol[2]	5.75	9.98	0.012	1.26	6.8	7.9	97
Isopropyl Acetate	5.93	13.2	0.368	0.14	5.1	5.0	101
Trichloroethene	5.98	8.32	0.413	0.13	4.5	3.4	106
1,4-Diflourobenzene (IS)	6.03						
Dibromomethane	6.39	4.87	0.222	0.16	4.8	3.6	99
1,2-Dichloropropane	6.50	6.98	0.272	0.16	4.9	3.3	111
Bromodichloromethane	6.58	5.23	0.459	0.13	4.2	3.5	101
Methyl Methacrylate	6.78	16.9	0.199	0.30	9.9	5.6	108
Propyl Acetate	6.94	6.94	0.293	0.17	5.7	5.8	102
2-Chloroethyl Vinyl Ether	7.18	14.6	0.064	0.17	8.9	5.1	76
cis-1,3-Dichloropropene	7.21	3.76	0.358	0.16	5.8	3.5	108
Toluene-d8 (SURR)	7.39	3.72	0.346		1.2	1.5	97
Toluene	7.44	3.80	1.59	0.14	4.5	3.3	101

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Table V US EPA Method 8260 Soil - Calibration, Method Detection Limit and Initial Demonstration of Capability Data							
Compound	Retention Time	Calibration (1 ppb – 200 ppb)		Method Detection Limit (n=7, 1 ppb)		Initial Demonstration of Capability (n=7, 20 ppb)	
		Linearity RF (≤20% RSD r^2≥0.99)	Average RF	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
2-Nitropropane	7.66	13.5	0.094	0.24	7.1	5.4	94
Tetrachloroethene	7.78	9.44	0.614	0.09	3.2	10.8	95
4-Methyl-2-Pentanone	7.82	7.08	0.084	0.78	10.6	5.5	99
trans-1,3-Dichloropropene	7.83	15.0	0.424	0.07	2.8	3.7	98
1,1,2-Trichloroethane	7.96	3.07	0.226	0.16	5.2	4.2	105
Ethyl Methacrylate	8.00	16.1	0.28	0.21	8.9	5.3	94
Dibromochloromethane	8.10	3.39	0.371	0.12	4.0	3.7	99
1,3-Dichloropropane	8.19	2.20	0.446	0.12	4.1	4.3	104
1,2-Dibromoethane	8.28	2.15	0.317	0.11	3.8	4.0	99
Butyl Acetate	8.45	7.13	0.294	0.17	6.4	4.8	99
2-Hexanone	8.51	10.5	0.060	0.53	7.2	5.5	98
Chlorobenzene-d5 (IS)	8.69						
Chlorobenzene	8.71	3.53	1.08	0.10	3.4	3.3	96
Ethylbenzene	8.74	5.06	1.64	0.08	2.8	2.8	98
1,1,1,2-Tetrachloroethane	8.76	5.11	0.250	0.15	5.4	3.3	95
m-,p-Xylene	8.85	6.01	0.584	0.19	3.5	3.4	102
o-Xylene	9.17	4.85	0.549	0.12	4.5	4.1	95
Styrene	9.21	2.86	0.884	0.10	3.9	3.6	97
Bromoform	9.22	2.67	0.239	0.15	5.4	3.8	98
Isopropylbenzene	9.40	7.12	1.41	0.11	4.5	4.3	97
Amyl Acetate	9.51	3.44	0.263	0.20	7.8	5.3	105
4-Bromofluorobenzene (SURR)	9.60	3.38	0.543		1.8	2.2	99
Bromobenzene	9.66	0.990	0.887	0.22	7.0	5.9	98
1,3,5-Trimethylbenzene	9.69	4.69	0.100	0.29	10.8	6.5	98
n-Propylbenzene	9.69	4.08	2.46	0.19	6.9	6.2	106
1,1,2,2-Tetrachloroethane	9.75	2.50	0.409	0.22	6.8	7.3	99
2-Chlorotoluene	9.80	3.58	1.56	0.20	6.5	5.4	102
1,2,3-Trichloropropane	9.84	2.30	0.350	0.25	7.7	6.4	103
cis-1,4-Dichloro-2-Butene	9.87	8.91	0.129	0.24	8.9	5.8	108
trans-1,4-dichloro-2-butene	9.87	4.15	0.116	0.23	8.1	4.6	105
4-Chlorotoluene	9.91	4.34	1.55	0.19	6.6	6.0	101
Pentachloroethane	10.05	8.18	0.169	0.16	6.2	7.3	104
tert-Butylbenzene	10.05	7.37	1.89	0.19	7.1	8.3	106
1,2,4-Trimethylbenzene	10.10	16.5	1.67	0.15	7.0	5.6	93
sec-Butylbenzene	10.17	7.73	2.37	0.17	6.8	6.5	112
p-Isopropyltoluene	10.27	15.2	1.94	0.13	6.5	5.5	97
1,3-Dichlorobenzene	10.31	1.63	1.39	0.22	7.2	5.1	92
1,4-Dichlorobenzene-d4 (IS)	10.36						
1,4-Dichlorobenzene	10.37	4.68	1.44	0.23	7.6	5.8	86
n-Butylbenzene	10.55	14.5	1.63	0.16	6.9	6.1	97
Hexachloroethane	10.63	6.17	0.549	0.08	5.3	5.8	101

Table V US EPA Method 8260 Soil - Calibration, Method Detection Limit and Initial Demonstration of Capability Data							
Compound	Retention Time	Calibration (1 ppb – 200 ppb)		Method Detection Limit (n=7, 1 ppb)		Initial Demonstration of Capability (n=7, 20 ppb)	
		Linearity RF (≤20% RSD r^2≥0.99)	Average RF	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
1,2-Dichlorobenzene	10.65	1.25	1.29	0.21	7.0	5.5	93
1,2-Dibromo-3-Chloropropane	11.19	2.25	0.154	0.27	7.9	6.1	94
Nitrobenzene	11.57	13.9	0.020	0.27	8.6	8.1	66
Hexachlorobutadiene	11.61	4.69	0.077	0.23	8.0	6.1	84
1,2,4-Trichlorobenzene	11.63	8.02	0.824	0.22	7.5	5.8	79
Naphthalene	11.85	10.1	1.57	0.23	7.5	5.8	94
1,2,3-Trichlorobenzene	11.97	8.09	0.783	0.18	5.8	6.2	84

1. Linear calibration
2. Calibration range 2-200 ppb
3. Calibration range 5-200 ppb
4. Calibration range 10-200 ppb
5. 20 ppb MDL

Figure 1 Total Ion Chromatogram (TIC) of a Water Method, 5 ppb VOC Standard with an Inset Indicating Consistent Peak Shapes for all Compounds with Minimal Water Interference

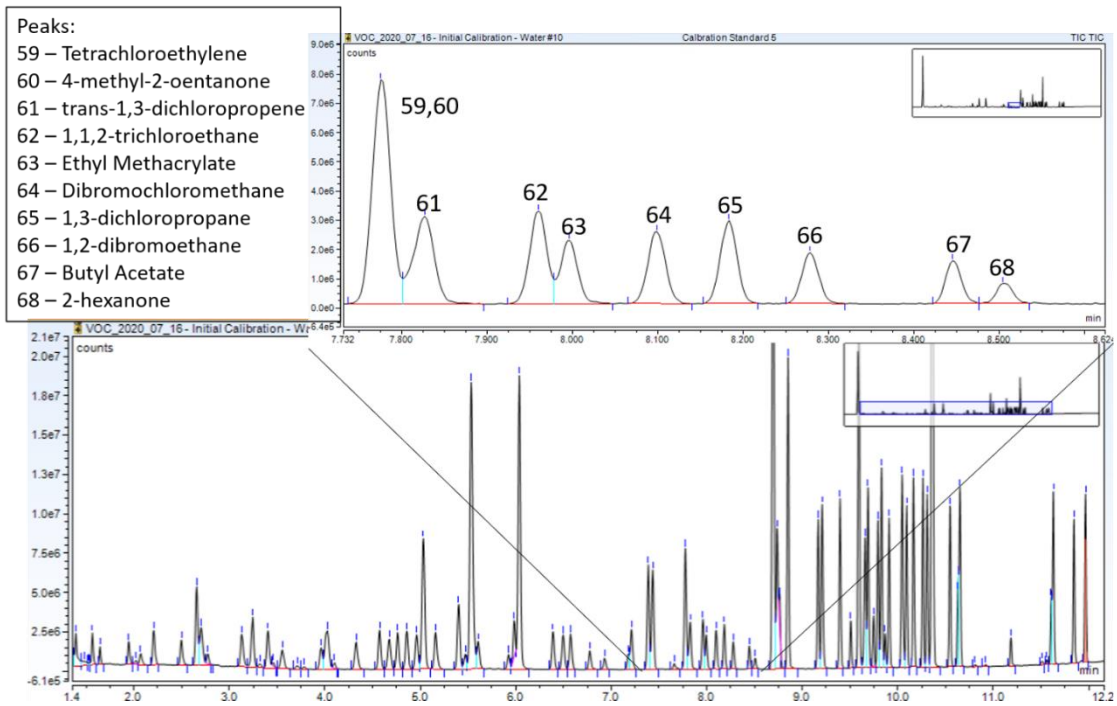

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Figure 2 TIC of a Soil Method, 5 ppb VOC Standard with an Inset Indicating Consistent Peak Shapes for all Compounds with Minimal Water Interference

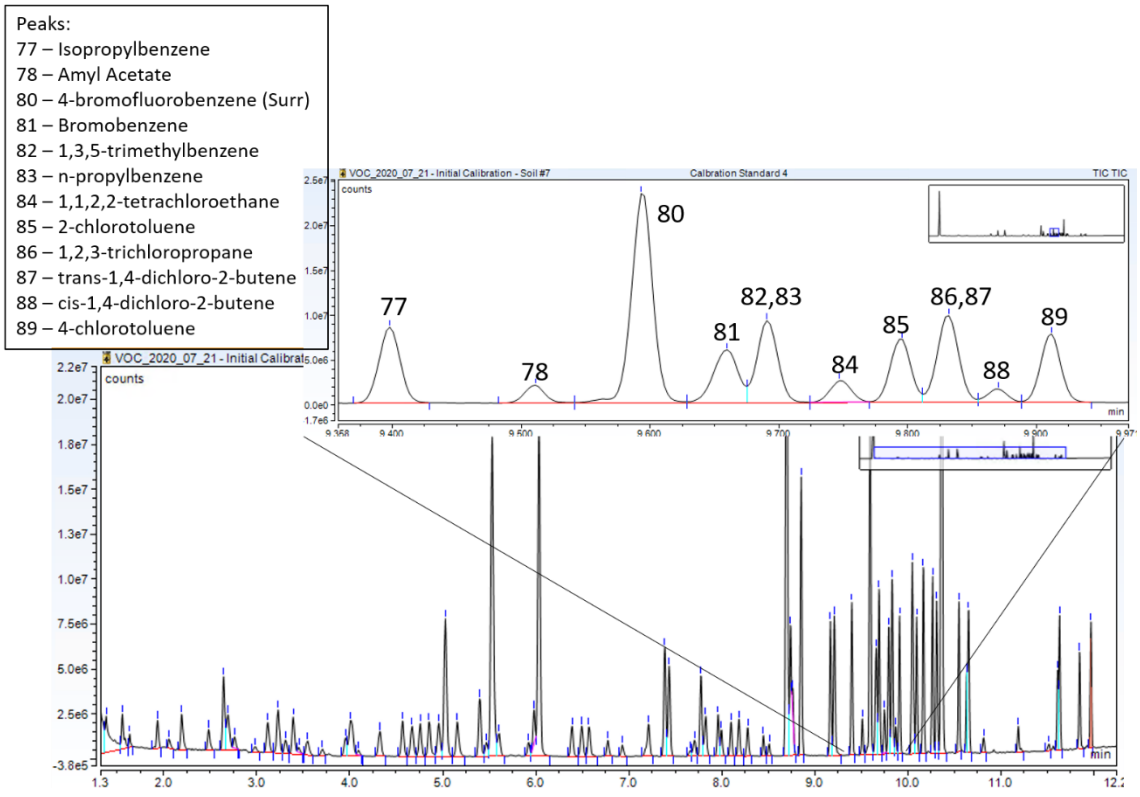

Figure 3 Extracted Ion Chromatograms for Chloromethane in the 0.2 ppb Water Standard: (A) Quantitation Ion and a Confirming Ion, (B) Matching Measured Spectrum to the NIST Library and (C) Linear Calibration Over a Concentration Range of 0.2 ppb to 200 ppb

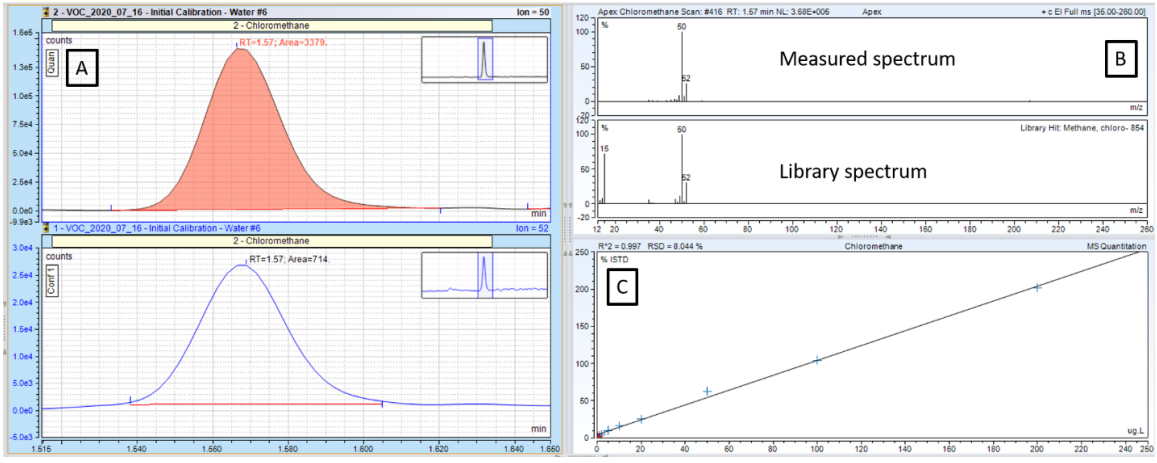

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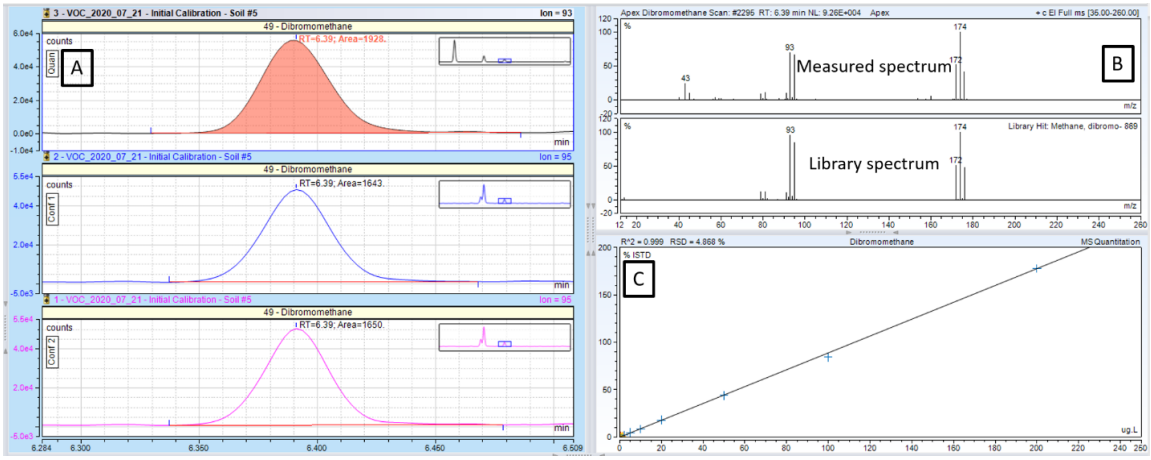
Figure 4 Extracted Ion Chromatograms for Dibromomethane in the 1 ppb Soil Standard: (A) Quantitation Ion and Two Confirming Ions, (B) Matching Measured Spectrum to the NIST Library and (C) Linear Calibration Over a Concentration Range of 1 ppb to 200 ppb



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. Detection was performed by a Thermo Scientific TRACE 1310 GC and ISQ 7000 MS with an ExtractaBrite source.

The %RSD of the calibration curve passed all method requirements. MDL and precision for seven 0.5 ppb standards for the water method, and seven 1 ppb standards for the soil method, showed minimal interference from excessive water and resulted in values <0.25 ppb for most compounds.

The IDC with precision and accuracy for seven 20 ppb water standards displayed less than 10% RSD for 100 of 103 compounds and an average recovery of 88% for the compounds of interest. The IDC with precision and accuracy for seven 20 ppb soil standards displayed all compounds having <11% RSD with an average recovery of 98%.

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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