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Validation of Volatile Organic Compound by USEPA Method 8260C

Application Note

By Tyler Trent

Abstract

In order to determine the concentration of volatile organic compounds (VOCs) in water and soil matrices the USEPA developed Method 8260C[1] in conjunction with preparative Methods 5030[2] and 5035[3]. Water and soil sample analysis were analyzed in this study. A working linear calibration curve and Method Detection Limits (MDLs) will be demonstrated for various target compounds from the approved list. The water study used a 5mL sample volume, while the soil study utilized a 5 gram mass under in vial purge conditions. Following the guidelines of USEPA Method 8260C, an Atomx automated VOC sample prep system in conjunction with a GCMS was used to validate the method.

Introduction

Teledyne Tekmar has developed the Atomx, a VOC sample prep system that integrates a Purge and Trap Concentrator with a Multi-Matrix Autosampler. This “all-in-one” set up allows for increased throughput by incorporating an 80 position vial sampler capable of processing water, soil and automated methanol extractions. This integrated platform also brings advances in communication allowing faster analysis time by preparing one sample while the other is running. In addition the ability to program internal standard and surrogate volumes automatically and new clean up techniques further improve sample capacity so very critical to today’s environmental testing laboratories.

Using an Agilent 7890/5975 GCMS with a 6mm draw out plate installed, a linear calibration curve and MDL study were performed for both water and soil matrices. The water samples analyzed a calibration range from 0.5-200ppb, while the soil calibration curve was from 1.0-200ppb. Water samples were analyzed using a 5mL volume, while soil analysis required a 5 gram sample. Analysis strictly followed the criteria outlined in the USEPA Method 8260C.

Experimental-Instrument Conditions

The Atomx, equipped with a #9 adsorbent trap, and an Agilent 7890A GC with a 5975C inert XL MSD were utilized for this study. **Tables 1-4** show the CGMS and purge and trap conditions for both water and soils applications.

GC Parameters	
GC:	Agilent 7890A
Column	Restek RTX-624 20m x 0.18mmID x 1um
Oven Program:	40°C for 4 min; 16°C/min to 100°C for 0 min; 30°C /min to 200°C for 4 min, 15.083 min runtime
Inlet:	220°C
Column Flow	0.9mL/min
Gas:	Helium
Split:	80:1
Pressure:	21.542 psi
Inlet:	Split/Split less

MSD Parameters	
MSD:	5975C inert XL
Source:	230°C
Quad:	150°C
Solvent Delay:	0.5 min
Scan Range:	m/z 35-270
Scans:	5.76 scans/sec
Threshold:	150
MS Transfer Line Temp:	230°C

Tables 1 & 2: GC and MSD Parameters

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Atomx Water Parameters			
Variable	Value	Variable	Value
Valve oven Temp	140°C	Dry Purge Flow	100mL/min
Transfer Line Temp	140°C	Dry Purge Temp	20°C
Sample Mount Temp	90°C	Methanol Needle Rinse	Off
Water Heater Temp	90°C	Methanol Needle Rinse Volume	3.0mL
Sample Vial Temp	20°C	Water Needle Rinse Volume	7.0mL
Sample Equilibrate Time	0.00 min	Sweep Needle Time	0.50min
Soil Valve Temp	100°C	Desorbs Preheat Time	245°C
Standby Flow	10mL/min	GC Start Signal	Start of Desorbs
Purge Ready Temp	40°C	Desorbs Time	2.00 min
Condensate Ready Temp	45°C	Drain Flow	300mL/min
Presweep Time	0.25 min	Desorbs Temp	250°C
Prime Sample Fill Volume	3.0mL	Methanol Glass rinse	Off
Sample Volume	5.0mL	Number of Methanol Glass Rinses	1
Sweep Sample Time	0.25 min	Methanol Glass Rinse Volume	3.0mL
Sweep Sample Flow	100mL/min	Number of Bake Rinses	1
Sparge Vessel Heater	On	Water Bake Rinse Volume	7.0mL
Sparge Vessel Temp	40°C	Bake Rinse Sweep Time	0.25 min
Prepurge Time	0.00 min	Bake Rinse Sweep Flow	100mL/min
Prepurge Flow	0mL/min	Bake Rinse Drain Time	0.40 min
Purge Time	11.00 min	Bake Time	4.00 min
Purge Flow	40mL/min	Bake Flow	200mL/min
Purge Temp	20°C	Bake Temp	280°C
Condensate Purge Temp	20°C	Condensate Bake Temp	200°C
Dry Purge Time	2.00 min		

Table 3: Atomx Water Parameters (Parameters highlighted in yellow were not used.)

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Atomx Soil Parameters			
Variable	Value	Variable	Value
Valve oven Temp	140°C	Purge Time	11.00 min
Transfer Line Temp	140°C	Purge Flow	40mL/min
Sample Mount Temp	90°C	Purge Temp	20°C
Water Heater Temp	90°C	Condensate Purge Temp	20°C
Sample Vial Temp	40°C	Dry Purge Time	2.00 min
Prepurge Time	0.00 min	Dry Purge Flow	100mL/min
Prepurge Flow	0mL/min	Dry Purge Temp	20°C
Preheat Mix Speed	Medium	Methanol Needle Rinse	Off
Sample Preheat Time	0.00 min	Methanol Needle Rinse Volume	3.0mL
Soil Valve Temp	100°C	Water Needle Rinse Volume	7.0mL
Standby Flow	10mL/min	Sweep Needle Time	0.25 min
Purge Ready Temp	40°C	Desorbs Preheat Time	245°C
Condensate Ready Temp	45°C	GC Start Signal	Start of Desorbs
Presweep Time	0.25 min	Desorbs Time	2.00 min
Water Volume	10mL	Drain Flow	300mL/min
Sweep Water Time	0.25 min	Desorbs Temp	250°C
sweep Water Flow	100mL/min	Bake Time	2.00 min
Sparge Vessel Heater	Off	Bake Flow	400mL/min
Sparge Vessel Temp	20°C	Bake Temp	280°C
Purge Mix Speed	Slow	Condensate Bake Temp	200°C

Table 4: Atomx Soil Parameters (Parameters highlighted in yellow were not used.)

Calibration

A 50ppm working calibration standard was prepared in methanol. Calibration standards were then serially diluted with de-ionized water to the final calibration concentration level. The water calibration ranged from 0.5-200ppb, while the soil ranged from 1-200ppb. A 25ppm internal standard (IS) was prepared in methanol and transferred to one of the three standard addition vessels on the Atomx. Using the standard addition feature, the Atomx transferred the internal standard in 5µL aliquots to the sample providing a constant 25ppb final concentration.

Aglient Chemstation software was used to process the calibration and MDL data. The relative response factors (RRF) of all target analytes were evaluated for average RRF and percent relative standard deviation (%RSD) over the calibrated range. Both water and soil calibration curves met the USEPA 8260C[1] performance criteria. With results listed in Table 5.

Minimum Detection Limits

Method detection limits were established for all compounds by analyzing seven replicates at a 0.5ppb concentration for water and 1.0ppb level for soil. The detection limits for each matrix can be found in **Table 5**.

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	Water					Soil				
Compound	Spike Level	MDL	Minimum RF[1]	Avg. RF	Calibration %RSD	Spike Level	MDL	Minimum RF[1]	Avg. RF	Calibration %RSD
Pentafluorobenzene (IS)	25					25				
Dichlorodifluoromethane	0.5	0.193	0.1	0.212	6.96	1	0.298	0.1	0.138	11.58
Chloromethane	0.5	0.188	0.1	0.458	2.98	1	0.419	0.1	0.355	6.7
Vinyl Chloride	0.5	0.145	0.1	0.389	4.83	1	0.201	0.1	0.316	6.73
Bromomethane	0.5	0.322	0.1	0.201	12.56	1	0.446	0.1	0.181	13.97
Chloroethane	0.5	0.267	0.1	0.198	15.88	1	0.421	0.1	0.21	13.67
Trichloromonofluoromethane	0.5	0.119	0.1	0.434	16.44	1	0.171	0.1	0.366	7.5
Diethyl Ether	0.5	0.329		0.204	6.56	1	0.173		0.139	2.68
1,1,2-Trichloro-1,2,2-trifluoroethane	0.5	0.286	0.1	0.285	3.31	1	0.276	0.1	0.209	11.59
1,1-Dichloroethene	0.5	0.441	0.1	0.271	4.67	1	0.43	0.1	0.216	5.56
Carbon disulfide	0.5	0.264	0.1	0.735	5.59	1	0.234	0.1	0.701	14.83
Iodomethane	0.5	0.119		0.499	7.47	1	0.127		0.429	12.11
Acetone*	0.5	0.593	0.1	0.179	0.999	1	1.657	0.1	0.175	0.995
Allyl chloride	0.5	0.35		0.175	7.93	1	0.499		0.151	11.08
Methyl Acetate	0.5	0.202	0.1	0.833	7.79	1	0.28	0.1	0.239	15.61
Acetonitrile	0.5	0.33		0.212	13.84	1	0.547		0.65	18.92
Methylene Chloride	0.5	0.197	0.1	0.329	9.68	1	0.216	0.1	0.273	9
Tert-Butyl Alcohol (TBA)	5	0.102		1.133	3.19	5	0.227		0.263	9.99
Methyl-tert-butyl Ether (MTBE)	0.5	0.115	0.1	1.033	9.46	1	0.269	0.1	0.7	7.44
trans-1,2-Dichloroethene	0.5	0.197	0.1	0.324	8.05	1	0.173	0.1	0.285	14.13
Acrylonitrile	0.5	0.126		0.511	18.86	1	0.749		0.13	7.17
1,1-Dichloroethane	0.5	0.155	0.2	0.726	7.74	1	0.233	0.2	0.634	9.15
Chloroprene	0.5	0.08		0.761	4.67	1	0.172		0.66	9.05
Vinyl Acetate	0.5	0.246		1.211	8.31	1	0.282		0.657	3.18
Ethyl-tert-butyl Ether (ETBE)	0.5	0.107		1.645	5.4	1	0.145		1.237	2.86
2,2-Dichloropropane	0.5	0.145		0.523	8.94	1	0.157		0.449	5.92
cis-1,2-Dichloroethene	0.5	0.117	0.1	0.396	6.32	1	0.169	0.1	0.334	7.63
Ethyl Acetate	0.5	0		0.034	8.57	1	0		0.011	14.22
Methyl Acrylate	0.5	0.191		1.018	6.57	1	0.233		0.313	1.57
Propionitrile	0.5	0.192		0.235	9.87	1	1.149		0.052	11.45
Bromochloromethane	0.5	0.191		0.467	6.63	1	0.16		0.346	2.06
Tetrahydrofuran	0.5	0.159		0.459	8.9	1	0.194		0.1	6.12
Chloroform	0.5	0.163	0.2	0.641	7.61	1	0.185	0.2	0.557	10.24
Methacrylonitrile	0.5	0.096		0.576	4.75	1	0.401		0.199	5.06
1,1,1-Trichloroethane	0.5	0.169	0.1	0.594	5.36	1	0.152	0.1	0.525	13.4
Dibromofluoromethane (Surr)	25	2.196		0.386	2.81	25	1.69		0.368	1.42
Carbon Tetrachloride	0.5	0.062	0.1	0.548	3.68	1	0.212	0.1	0.45	4.91
1,1-Dichloropropene *	0.5	0.23		.981	.9986	1	0.309		.676	.9999
Benzene	0.5	0.136	0.5	1.442	3.65	1	0.142	0.5	1.261	15.48
1,2-Dichloroethane	0.5	0.112		0.602	4.59	1	0.133		0.456	2.86
tert-Amyl Methyl Ether (TAME)	0.5	0.079		1.099	5.34	1	0.099		0.762	2.91
Isopropyl Acetate	0.5	0.131		0.58	10.17	1	0.238		0.373	7.43
1,4-Diflourobenzene (IS)	25					25	0			
Trichloroethylene	0.5	0.145		0.296	8.57	1	0.105		0.237	7.74
1,2-Dichloropropane	0.5	0.167	0.1	0.32	5.39	1	0.199	0.1	0.264	9
Dibromomethane	0.5	0.187		0.179	3.54	1	0.236		0.118	4.42
Methyl Methacrylate	0.5	0.124		2.92	4.87	1	0.754		0.112	3.67
n-Propyl acetate	0.5	0.057		0.948	3.74	1	0.583		3.12	5.77
Bromodichloromethane	0.5	0.09	0.2	0.34	3.79	1	0.293	0.2	0.272	4.34
2 Nitropropane *	0.5	ND		0.008	0.993	1	ND		0.002	0.998

Table 5: Experimental Results

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1. Recommended minimum relative response factor criteria from Method 8260C

* Compound was linear regressed

	Water					Soil				
Compound	Spike Level	MDL	Minimum RF[1]	Ave. RF	Calibration %RSD	Spike Level	MDL	Minimum RF[1]	Ave. RF	Calibration %RSD
2-Chloroethyl Vinyl Ether	0.5	0.11		0.28	3.98	1	0.22		0.101	3.97
cis-1,3-Dichloropropene	0.5	0.08	0.2	0.39	2.89	1	0.14	0.2	0.301	5.62
Toluene-d8 (Surr)	25	0.76		1.03	1.35	25	1.2		1.003	0.98
Toluene	0.5	0.1	0.4	1.14	4.87	1	0.17	0.4	0.968	13.31
trans-1,3-Dichloropropene	0.5	0.1	0.1	3.54	6.66	1	0.23	0.1	0.254	8.85
Ethyl Methacrylate	0.5	0.1		0.42	7.56	1	0.17		0.22	8.42
Tetrachloroethylene	0.5	0.17	0.2	0.39	6.43	1	0.26	0.2	0.288	11.85
1,1,2-Trichloroethane	0.5	0.07	0.1	0.22	1.92	1	0.27	0.1	0.142	1.21
1,3-Dichloropropane	0.5	0.13		0.44	2.1	1	0.27		0.299	2.97
Chlorobenzene-d5 (IS)	25					25	0			
2-Hexanone	0.5	0.06	0.1	0.67	7.65	1	0.83	0.1	0.177	10.85
Dibromochloromethane	0.5	0.11	0.1	0.38	5.26	1	0.12	0.1	0.261	8.34
Butyl Acetate	0.5	0.08		0.94	11.61	1	0.13		0.365	11.76
1,2-Dibromoethane	0.5	0.08	0.1	0.35	3.59	1	0.28	0.1	0.21	1.72
Chlorobenzene	0.5	0.07	0.5	0.93	3.78	1	0.32	0.5	0.768	5.37
1,1,1,2-Tetrachloroethane	0.5	0.14		0.37	5.09	1	0.1		0.305	5.49
Ethylbenzene	0.5	0.1	0.1	1.42	3.03	1	0.24	0.1	1.226	9.92
m-,p-Xylene	1	0.29	0.1	0.59	2.5	2	0.44	0.1	0.494	8.67
o-Xylene	0.5	0.06	0.3	0.59	8.17	1	0.22	0.3	0.491	9.48
Styrene	0.5	0.06	0.3	1	3.18	1	0.31	0.3	0.791	2.95
Bromoform	0.5	0.25	0.1	0.29	9.04	1	0.36	0.1	0.156	10.61
Isopropylbenzene (Cumene)	0.5	0.06	0.1	1.5	4.27	1	0.23	0.1	1.28	9.02
Bromofluorobenzene (BFB, Surr)	25	1.43		0.46	2.24	25	1.17		0.44	0.96
Bromobenzene	0.5	0.22		0.44	4.59	1	0.27		3.43	3.98
n-Propylbenzene	0.5	0.12		1.64	3.38	1	0.28		1.369	9.12
1,4-Dichlorobenzene-d4 (IS)	25	0				25	0			
1,1,2,2-Tetrachloroethane	0.5	0.08	0.3	0.71	7	1	0.19	0.3	0.357	9.5
1,2,3-Trichloropropane	0.5	0.2		0.69	2.15	1	0.16		0.331	5.48
trans-1,4-dichloro-2-Butene	0.5	0.11		0.35	15.24	1	0.37		0.147	17.63
2-Chlorotoluene	0.5	0.19		1.68	2.62	1	0.35		1.439	6.28
1,3,5-Trimethylbenzene	0.5	0.11		2.08	3.53	1	0.32		1.829	10.87
4-Chlorotoluene	0.5	0.1		1.93	2.16	1	0.35		1.691	12.43
tert-Butylbenzene	0.5	0.09		1.95	3.36	1	0.31		1.762	8.54
1,2,4-Trimethylbenzene	0.5	0.03		2.18	4.3	1	0.36		1.876	8.16
sec-Butylbenzene	0.5	0.12		2.67	4.07	1	0.32		2.361	9.85
1,3-Dichlorobenzene	0.5	0.21	0.6	1.4	3.55	1	0.35	0.6	1.139	9.18
p-Isopropyltoluene (p-Cymene)	0.5	0.13		2.45	5.79	1	0.4		2.032	7.99
1,4-Dichlorobenzene	0.5	0.11	0.5	1.45	3.48	1	0.38	0.5	1.164	8.52
n-Butylbenzene	0.5	0.18		1.89	6.87	1	0.38		1.521	10.66
1,2-Dichlorobenzene	0.5	0.12	0.4	1.37	5.92	1	0.38	0.4	1.065	4.6
1,2-Dibromo-3-chloropropane	0.5	0.28	0.05	0.24	9.38	1	0.71	0.05	0.071	16.44
Nitrobenzene	0.5	0.42		0.85	16.62	1	0.57		0.011	17.17
1,2,4-Trichlorobenzene	0.5	0.3	0.2	0.92	7.22	1	0.82	0.2	0.615	9.29
Hexachlorobutadiene	0.5	0.37		0.37	6.38	1	0.39		0.299	9.09
Naphthalene	0.5	0.08		3.62	9.29	1	0.84		1.663	6.54
1,2,3-Trichlorobenzene	0.5	0.11		0.89	8.32	1	0.74		0.592	8.73

Table 5: Experimental Results Continued

1. Recommended minimum relative response factor criteria from Method 8260C

* Compound was linear regressed

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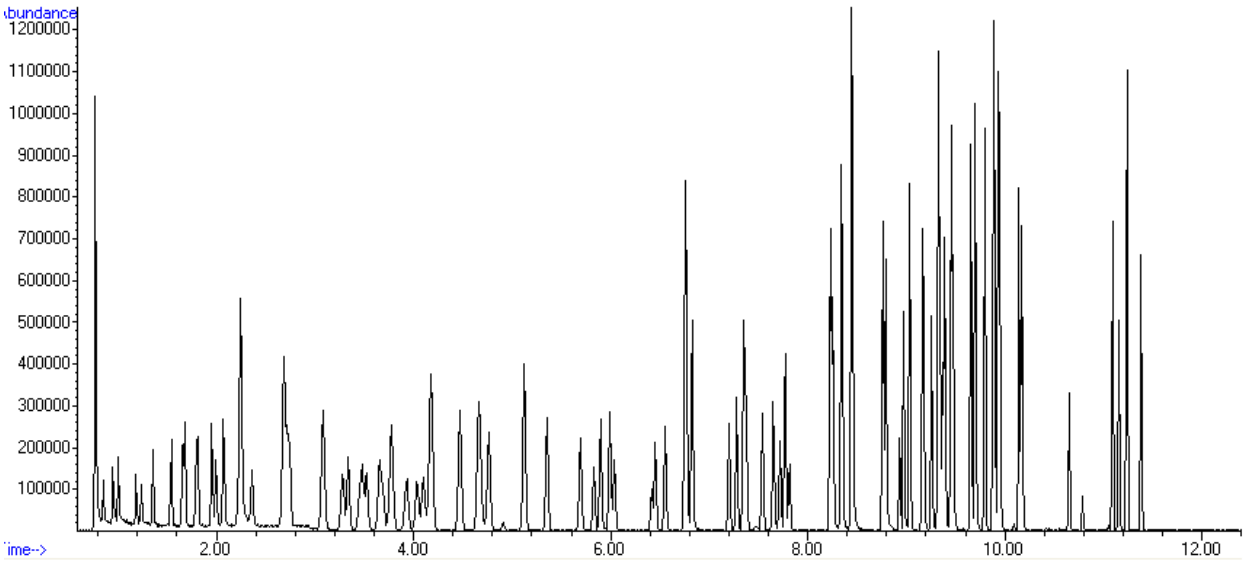

Figure 1: TIC of a 20ppb Water Chromatogram for Method 8260C

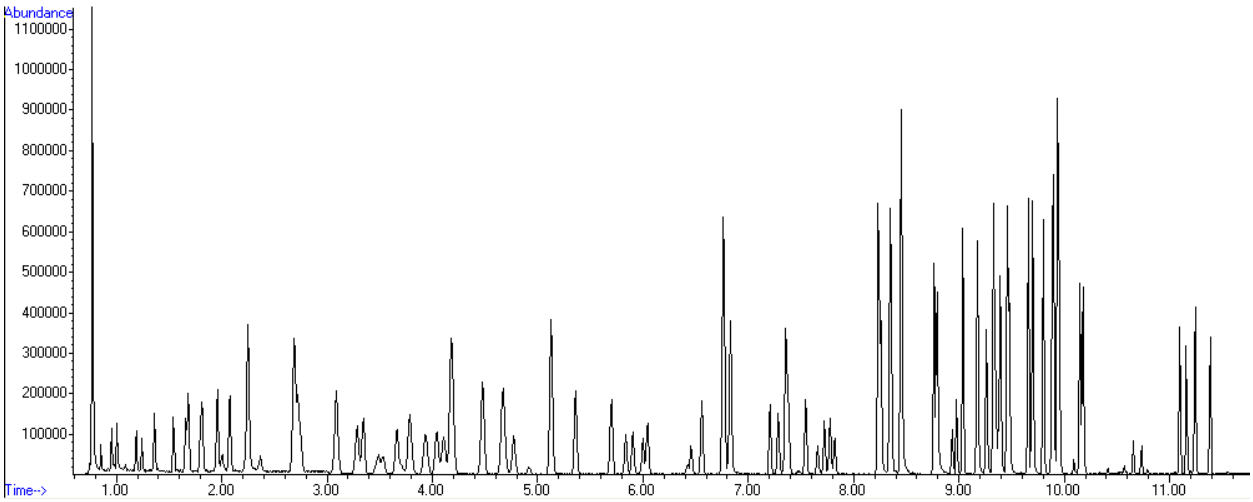

Figure 2: TIC of a 20ppb Soil Chromatogram for Method 8260C

Conclusions

This study demonstrates the capability of the Atomx automated sample prep system in conjunction with an Agilent 7890/5975 GCMS in regards to USEAP Method 8260C. Calibration and MDL data met all performance criteria of the method. By completely automating the sample preparation of multiple matrices, efficiency and throughput can be greatly increased saving time and money.

References

1. USEPA Method 8260C Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Revision 3, August 2006
2. USEPA Method 5030 Purge-And-Trap For Aqueous Samples Revision 3, May 2003
3. USEPA Method 5035 Closed-System Purge-And Trap and Extractions For Volatile Organics In Soil and Waste Samples Revision 1, July 2002

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