Validation of USEPA Method 8260C Using Teledyne Tekmar Atomx, and Perkin-Elmer Clarus 600 GC/MS

Application Note

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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 samples were analyzed in this study. A working linear calibration curve and Method Detection Limits (MDLs) will be demonstrated for the target compound list. The water study used a 5mL sample volume, while the soil study utilized an in-vial purge. Following the conditions of USEPA Method 8260C, an Atomx Automated VOC Sample Prep system in conjunction with a GC/MS was used to validate the method.

Introduction

Teledyne Tekmar has developed the Atomx, an Automated 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 and efficiency through the features that it provides, such as the three standard addition vessels and 80-postion autosampler tray. Also, the Atomx allows for sampling of multiple matrices, water, soil and methanol extraction in a single run.

Using the PerkinElmer Clarus 600/600T GCMS, a linear calibration curve and MDLs were performed for both water and soil matrices. The water samples employed a calibration range from 0.5-200ppb, while the soil calibration curve ranged from 1.0-200ppb. Both water and soil methods require either a 5mL or 5g sample size. Analysis strictly followed the criteria outlined in the USEPA Method 8260C.

Experimental-Instrument Conditions

The Atomx, equipped with a #9 adsorbent trap, and a PerkinElmer Clarus 600 GC with a Clarus 600T MS were utilized for this study. **Tables 1-4** show the CG/MS and purge and trap conditions for both water and soil applications.

GC Parameters	
GC:	Perkin-Elmer Clarus 600 Gas Chromatograph
Column	Restek RTX-VMS 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:	18.4psi
Inlet:	Split/Split less

MSD Parameters	
MSD:	Clarus 600T Quadrupole Mass Spectrometer
Source:	200 °C
Transfer Line Temp:	200 °C
Solvent Delay:	0.5 min
Scan Range:	35-270 m/z
Scan Time:	0.2 sec
Inter-scan Delay:	0.1 sec
Ionization Mode:	EI+

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	Desorb Preheat Time	245 °C
Standby Flow	10mL/min	GC Start Signal	Start of Desorbs
Purge Ready Temp	40 °C	Desorb Time	2.00 min
Condensate Ready Temp	45 °C	Drain Flow	300mL/min
Presweep Time	0.25 min	Desorb 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	0 mL/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	10 mL/min	Sweep Needle Time	0.25 min
Purge Ready Temp	40 °C	Desorb Preheat Time	245 °C
Condensate Ready Temp	45 °C	GC Start Signal	Start of Desorbs
Presweep Time	0.25 min	Desorb Time	2.00 min
Water Volume	10 mL	Drain Flow	300 mL/min
Sweep Water Time	0.25 min	Desorb Temp	250 °C
sweep Water Flow	100 mL/min	Bake Time	2.00 min
Sparge Vessel Heater	Off	Bake Flow	400 mL/min
Sparge Vessel Temp	20 °C	Bake Temp	280 °C
Purge Mix Speed	Fast	Condensate Bake Temp	200 °C

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

Calibration and Minimum Detection Limits

A 50ppm working calibration standard was prepared in methanol. Calibration standards were made in volumetric flasks using de-ionized water. 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 IS in 5µL aliquots to the sample providing a constant 25ppb final concentration in 5mL.

TurboMass software was used to process the calibration and MDL data. The relative response factors (RRF) of all target analytes were evaluated for average Response Factor (RF) and calibration percent relative standard deviation (%RSD). Both water and soil calibration curves met the USEPA 8260C[1] performance criteria with results listed in **Table 5**. Total Ion Chromatograms (TIC) for 25ppb water and soil standard can be found in **Figures 1-2**.

Method detection limits (MDLs) were established for all compounds by analyzing seven replicates at 1.0ppb for water and 5.0ppb for soil. The MDLs for each matrix can be found in **Table 5**.

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Compound	Water				Soil			
	Spike Level (ppb)	MDL	Avg. RF	Calibration %RSD	Spike Level (ppb)	MDL	Avg. RF	Calibration %RSD
Pentafluorobenzene (IS)	25				25			
Dichlorodifluoromethane	1	0.361	0.237	8.6	5	0.748	0.595	6
Chloromethane	1	0.138	0.662	9.6	5	0.631	0.828	9.4
Vinyl Chloride	1	0.194	0.443	5.1	5	0.757	0.726	6.6
Bromomethane*	1	0.132	0.354	0.9989	5	1.342	0.438	17
Chloromethane	1	0.242	0.277	15	5	0.728	0.428	18.3
Trichlorofluoromethane	1	0.194	0.408	7.8	5	0.6	0.654	8.3
Diethyl Ether	1	0.177	0.450	3.4	5	0.677	0.485	5.6
1,1-Dichloroethene	1	0.341	0.355	4.7	5	0.712	0.38	7.2
Carbon Disulfide	1	0.450	1.34	8.1	5	0.774	1.54	16
1,1,2-Trichlorofluoromethane	1	0.208	0.340	7.2	5	0.957	0.36	4.7
Iodomethane*	1	1.222	0.292	0.9992	5	1.597	0.313	0.9954
Allyl Chloride	1	0.118	0.925	9.7	5	0.866	0.985	5.9
Methylene Chloride	1	0.085	0.517	11.7	5	0.538	0.53	9.1
Acetone*	1	0.157	0.426	0.9988	5	1.214	0.28	0.9989
Trans-1,2-Dichloroethene	1	0.350	0.406	3.6	5	0.747	0.471	9.1
Methyl Acetate	1	0.111	0.968	15.8	5	0.833	0.36	8.4
Methyl-tert-butyl-ether	1	0.099	1.313	5.3	5	0.626	1.275	2.3
Tert-butyl Alcohol	5	1.503	0.098	7.5	25	3.937	0.057	11.3
Chloroprene	1	0.303	0.459	5.5	5	0.733	0.453	2.7
1,1-Dichloroethane	1	0.110	0.875	4.5	5	0.582	0.995	5
Acrylonitrile*	1	0.070	0.593	0.9989	5	0.784	0.163	11.3
Ethyl Acetate	1	0.223	0.403	8.8	5	1.031	0.398	3.2
Vinyl Acetate*	1	0.000	0.168	10.6	5	1.065	0.096	0.9999
Ethyl-tert-butyl Ether	1	0.096	1.453	5.5	5	0.600	1.395	0.8
Cis-1,2-Dichloroethene	1	0.129	0.550	3.1	5	0.622	0.517	4.3
2,2-Dichloropropane	1	0.185	0.602	3.9	5	0.609	0.666	4
Bromochloromethane	1	0.246	0.235	15.3	5	1.138	0.235	9.1
Chloroform	1	0.158	0.811	4.5	5	0.735	0.857	2.2
Carbon Tetrachloride	1	0.354	0.413	8.8	5	0.688	0.59	3.1
1,1,1-Trichloroethane	1	0.234	0.561	3.6	5	0.720	0.632	3
Tetrahydrofuran	1	0.188	0.729	3.3	5	0.720	0.273	5.9
Dibromofluoromethane (Surr)	25		0.461	1.7	25		0.522	0.7
Methyl Acrylate	1	0.043	1.019	7	5	0.265	0.394	5.6
1,1-Dichloropropene	1	0.275	0.719	4.1	5	0.720	0.734	5.1
2-Butanone (MEK)	1	0.199	0.827	6.9	5	0.829	0.249	2.7
Benzene	1	0.133	2.510	5.7	5	0.662	2.257	4.2
Methacrylonitrile	1	0.081	0.451	6.9	5	0.850	0.2	7
Tert-amyl-methyl Ether	1	0.054	1.568	5.8	5	0.503	1.34	1.5

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Table 5 Continued	Water				Soil			
Compound	Spike Level (ppb)	MDL	Avg. RF	Calibration %RSD	Spike Level (ppb)	MDL	Avg. RF	Calibration %RSD
1,2-Dichloroethane	1	0.046	0.587	5	5	0.523	0.681	3.9
Isopropyl Acetate	1	0.061	1.790	5.8	5	0.482	0.989	4.3
Trichloroethene	1	0.229	0.314	3.6	5	0.599	0.276	6.6
1,4-Difluorobenzene (IS)	25				25			
Dibromomethane	1	0.284	0.168	9.2	5	0.759	0.118	7.5
1,2-Dichloropropane	1	0.107	0.305	3.9	5	0.687	0.265	2.7
Bromodichloromethane	1	0.080	0.382	4.4	5	0.742	0.328	4.4
Methyl Methacrylate	1	0.077	0.393	6.8	5	0.513	0.185	5.4
n-Propyl Acetate	1	0.077	1.004	7	5	0.403	0.383	6.4
2-Chloroethyl Vinyl Ether	1	0.210	0.260	8.4	5	0.655	0.148	6
cis-1,3-Dichloropropene	1	0.158	0.530	6.7	5	0.629	0.424	6.6
Toluene-D8 (Surr)	25		1.339	0.7	25		1.044	1
Toluene	1	0.116	1.314	4.4	5	0.702	1.042	3.5
2-Nirtopropane	1	0.177	0.162	6.4	5	1.311	0.055	17.3
Tetrachloroethene	1	0.387	0.302	10	5	0.786	0.273	10.7
4-methyl-2-pentanone	1	0.087	0.781	6.7	5	0.586	0.258	6.9
1,1,2-Trichloroethane	1	0.208	0.311	2	5	0.478	0.197	3.5
Ethyl Methacrylate	1	0.051	0.597	9.9	5	0.604	0.351	8.5
Dibromochloromethane	1	0.115	0.293	3.6	5	0.593	0.242	6.4
Trans-1,3-Dichloropropene	1	0.153	0.473	9.2	5	0.693	0.368	8.8
1,3-Dichloropropane	1	0.040	0.668	4.5	5	0.487	0.441	3.2
1,2,3-Trichloropropane	1	0.194	0.370	2.8	5	0.702	0.21	5.2
1,2-Dibromoethane	1	0.186	0.352	3.4	5	0.667	0.216	6
n-butyl acetate	1	0.134	0.439	14.9	5	0.617	0.163	13.5
2-Hexanone	1	0.127	0.694	5.8	5	0.813	0.183	8.1
Chlorobenzene-D5 (IS)	25				25			
Chlorobenzene	1	0.175	0.955	5.4	5	0.881	0.898	4.8
Ethylbenzene	1	0.233	0.523	4.8	5	0.760	0.442	5
1,1,1,2-Tetrachloroethane	1	0.175	0.283	3.9	5	0.873	0.259	5.8
M&P-Xylene	2	0.517	0.647	5.4	10	1.619	0.54	5
O-Xylene	1	0.149	0.623	5.9	5	0.814	0.513	6.5
Styrene	1	0.109	1.066	7	5	0.768	0.851	4.8
Bromoform	1	0.254	0.228	8.2	5	0.691	0.138	7.1
Isopropylbenzene	1	0.171	1.517	5.9	5	0.898	1.264	5.8
n-Amyl Acetate	1	0.062	0.884	17.2	5	0.707	0.442	18.1
4-Bromo-1-fluorobenzene (Surr)	25		0.569	2.5	25		0.417	1.4
n-Propylbenzene	1	0.248	1.716	5.7	5	0.730	1.375	4.6
trans-1,4-Dichloro-2-Butene	1	0.153	0.404	9.7	5	1.102	0.183	14.4
Bromobenzene	1	0.182	0.350	4.7	5	0.933	0.314	4.7
1,1,2,2-Tetrachloroethane	1	0.076	1.061	4.7	5	0.789	0.531	10

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Table 5 Continued	Water				Soil			
Compound	Spike Level (ppb)	MDL	Compound	Spike Level (ppb)	MDL	Compound	Spike Level (ppb)	MDL
1,3,5-Trimethylbenzene	1	0.171	2.357	4.9	5	1.290	2.154	5.4
2-Chlorotoluene	1	0.191	1.878	3.7	5	0.942	1.644	5.5
Cis-1,4-Dichloro-2-Butene	1	0.174	0.290	13.8	5	1.197	0.117	20
4-Chlorotoluene	1	0.209	2.010	4.6	5	1.071	1.798	7.3
Tertbutylbenzene	1	0.222	1.943	7.6	5	1.084	2.187	7.2
1,2,4-Trimethylbenzene	1	0.169	2.412	6.2	5	0.917	2.218	4.7
sec-Butylbenzene	1	0.320	2.933	6	5	1.143	2.683	6.4
p-Isopropyl toluene	1	0.333	2.228	7.4	5	1.091	2.468	5.2
1,3-Dichlorobenzene	1	0.199	1.359	5.3	5	0.799	1.326	11.6
1,4-Dichlorobenzene-D4 (IS)	25				25			
1,4-Dichlorobenzene	1	0.298	1.290	2.8	5	0.939	1.219	9
n-Butylbenzene	1	0.396	2.142	7	5	0.984	1.929	8.4
1,2-Dichlorobenzene	1	0.262	1.264	5.2	5	1.170	1.215	9.5
1,2-dibromo-3-Chloropropane	1	0.555	0.305	7.5	5	0.590	0.114	8.7
Hexachlorobutadiene	1	0.430	0.324	5.6	5	0.908	0.358	7.3
1,2,4-Trichlorobenzene	1	0.436	0.892	7.2	5	1.577	0.724	12
Napthalene	1	0.344	3.268	9.1	5	1.197	2.06	7.8
1,2,3-Trichlorobenzene	1	0.524	0.838	8.4	5	1.241	0.68	8.6

Table 5: Experimental Results For USEPA Method 8260C Water and Soil

* Compound was linear regressed

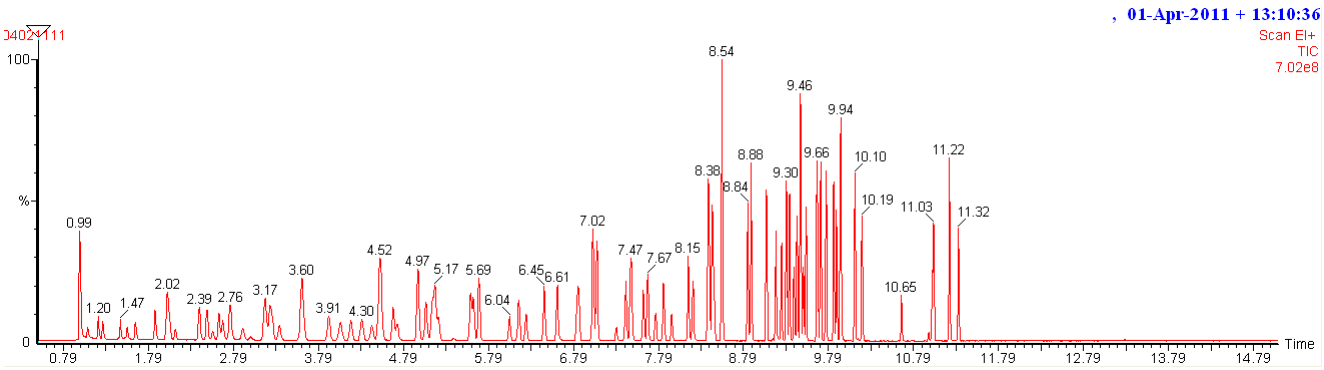

Figure 1: TIC of a 25ppb Water Standard for Method 8260C

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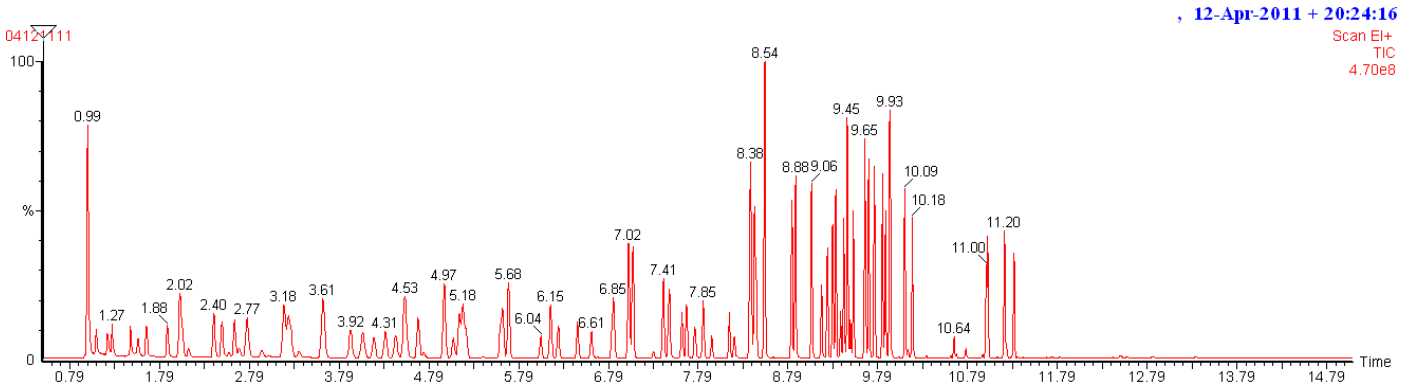

Figure 2: TIC of a 25ppb Soil Standard for Method 8260C

Conclusions

This study demonstrates the capability of the Atomx Automated VOC Sample Prep system in conjunction with a PerkinElmer Clarus 600 GC/MS in regards to USEPA 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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