Application Note

Environmental

MEE Method HJ605 Using the Teledyne Tekmar Atomx XYZ P&T and Agilent GC/MSD to Determine Volatile Organic Compound Concentration in Soil Matrices

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Abstract

Ministry of Ecology and Environment (MEE) Method HJ605 was used to determine the concentration of volatile organic compounds (VOCs) in soil matrices. The Teledyne Tekmar Atomx XYZ purge and trap (P&T) system coupled with the Agilent 7890B GC and 5977B MS was used to create a working linear calibration curve, method detection limits (MDLs), and a midpoint calibration check for target compounds.

Introduction

The Atomx XYZ is the most advanced Teledyne Tekmar 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 life span. In addition to other refinements, the Atomx XYZ incorporates a precision-machined valve manifold block to reduce potential leak sources and ensure that the system is both reliable and robust. Furthermore, the Agilent 7890B GC is the world's most widely used GC system, featuring accurate temperature controls, precise injection systems, and high-performance electronic pneumatic control (EPC) modules for good retention time and area count repeatability. The Agilent 5977B GC/MSD is the latest in the series of most trusted single quadrupole GC/MS instruments. It is ideal for labs that focus on applications such as environmental, chemical, petrochemical, food, forensic, pharmaceutical, and material testing.

Experimental

Sample preparation

A working 50 parts per million (ppm) calibration standard was prepared in methanol from commercially available 8260B mix, VOA (ketones), and 502.2 calibration mix standards. In total, the standard contained 66 compounds. The calibration standard did not include 1,1,2-trichloropropane and 1-chloro-2,3-epoxypropane from the MEE HJ639 method compound list, because they were not readily available. Also, m- and p-xylene were listed as one compound, as they commonly co-elute.

The soil calibration curve was prepared from 2 to 200 parts per billion (ppb) for all compounds. The relative response factor (RRF) was calculated for each compound using one of the three internal standards: fluorobenzene, chlorobenzene-d5, and 1,4-dichlorobenzene-d4. Surrogate standards consisted of dibromofluoromethane, toluene-d8, and 4-bromofluorobenzene. Internal and surrogate standards were prepared together in methanol from commercially available standards at a concentration of 25 ppm. After, 5 µL was mixed with each 5 mL sample, for a resulting concentration of 25 ppb.

Seven 2 ppb soil standards were prepared for MDL and precision calculations. Seven 20 ppb soil standards were prepared for the midpoint calibration check, precision, and accuracy. All calibration, MDL, and midpoint calibration check samples were analyzed using the Atomx XYZ conditions in Table 1, and the Agilent GC/MS conditions in Table 2.

Instrument conditions

Table 1. Teledyne Tekmar Atomx XYZ soil method conditions for MEE HJ605.

Standby	Variable
Valve Oven Temperature	140 °C
Transfer Line Temperature	140 °C
Sample Mount Temperature	90 °C
Water Heater Temperature	90 °C
Sample Vial Temperature	40 °C
Soil Valve Temperatur	100 °C
Standby Flow	10 mL/min
Purge Ready Temperature	40 °C
Purge	**Variable**
Prepurge Time	0.00 min
Prepurge Flow	0 mL/min
Preheat Mix Speed	Slow
Sample Preheat Time	0.00 min
Presweep Time	0.25 min
Water Volume	10.00 mL
Sweep Water Time	0.25 min
Sweep Water Flow	100 mL/min
Sparge Vessel Heater	Off
Purge Mix Speed	Medium
Purge Time	11.00 min
Purge Flow	40 mL/min
Purge Temperature	20 °C
MCS Purge Temperature	20 °C
Dry Purge Time	2.00 min
Dry Purge Flow	100 mL/min
Dry Purge Temperature	20 °C

Desorb	Variable
Methanol Needle Rinse	Off
Water Needle Rinse Volume	7.00 mL
Standby	**Variable**
Sweep Needle Time	0.25 min
Desorb Preheat Temperature	245 °C
GC Start Signal	Begin Desorb
Desorb Time	2.00 min
Drain Flow	300 mL/min
Desorb Temperature	250 °C
Bake	**Variable**
Bake Time	2.00 min
Bake Flow	400 mL/min
Bake Temperature	280 °C
MCS Bake Temperature	180 °C
Trap Number	9
Purge Gas	Nitrogen

Table 2. Agilent 7890B GC and 5977B MS system conditions for MEE Method HJ605.

Agilent 7890B GC Conditions	
Column	Agilent DB-VRX – equivalent, 20 m × 0.18 mm, 1 µm 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	220 °C; 80:1 split; septum purge flow: 0.5 mL/min
Agilent 5977B MS Conditions	
Temperature	Transfer line: 225 °C; source: 250 °C; quad: 200 °C
Scan	Range: 35 to 270 amu; solvent delay: 0.50 minutes, dwell/scan time: 0.15 seconds
Gain	Gain factor: 1.00

Results and discussion

The relative standard deviation (%RSD) of the average of the RRFs for the calibration curve, MDL, precision, and midpoint calibration check accuracy and precision data are shown in Table 3. Figure 1 displays a 10 ppb standard, indicating excellent peak resolution with minimal water inference for all VOCs.

Table 3. MEE Method HJ605 soil calibration, MDL, and midpoint calibration check data.

	Calibration (2 to 200 ppb)					Method Detection Limit (n = 7, 2 ppb)		Midpoint Calibration Check (n = 7, 20 ppb)	
Compound	Retention Time (min)	Quant Ion	IS	Average RRF	RRF (≤20% RSD R^2≥0.99)	MDL (ppb)	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
Dichlorodifluoromethane	1.06	85	1	0.445	5.2	0.64	10.2	9.1	89
Chloromethane	1.19	50	1	0.328	8.2	0.71	10.6	9.5	92
Vinyl Chloride	1.24	62	1	0.329	8.3	0.91	15.0	8.7	97
Bromomethane[1,4]	1.46	94	1	0.271	0.991	1.64	10.4	19.0	109
Chloroethane	1.55	64	1	0.160	18.3	0.89	10.6	5.5	115
Trichlorofluoromethane	1.66	101	1	0.539	12.0	0.35	5.0	2.7	97
1,1-Dichloroethene	2.04	61	1	0.668	7.8	0.29	4.2	2.9	100
Iodomethane[1,4]	2.13	142	1	0.745	0.996	2.28	16.6	19.7	90
Carbon Disulfide	2.44	76	1	0.359	5.4	0.23	3.5	3.1	97
Methylene Chloride[1,4]	2.53	49	1	0.710	0.997	0.99	11.9	3.2	100
Acetone[1,2,5]	2.57	58	1	0.055	0.999	2.50	6.6	8.8	88
trans-1,2-Dichloroethene	2.70	61	1	0.717	6.9	0.30	4.4	2.8	100
1,1-Dichloroethane	3.34	63	1	1.12	11.3	0.34	4.9	2.3	102
2-Chloro-1,3-Butadiene	3.37	53	1	0.900	5.8	0.29	4.6	1.9	99
cis-1,2-Dichloroethene	3.92	61	1	0.702	5.8	0.25	4.1	2.3	101
2,2-Dichloropropane	4.04	77	1	0.879	10.7	0.31	4.6	2.2	92
Bromochloromethane	4.12	130	1	0.441	6.7	0.23	3.6	2.7	97
Chloroform	4.24	83	1	1.03	9.0	0.30	4.4	2.5	94
Carbon Tetrachloride	4.38	117	1	0.841	5.7	0.27	4.2	2.6	93
Dibromofluoromethane (SURR)	4.42	111	1	0.562	5.4		6.2	2.6	103
1,1,1-Trichloroethane	4.44	97	1	0.921	3.7	0.25	3.9	2.6	95
2-Butanone[2,4]	4.56	43	1	0.101	6.2	1.17	6.5	5.4	89
1,1-Dichloropropene	4.58	75	1	0.758	2.1	0.26	4.4	2.3	95
Benzene	4.82	78	1	2.37	3.5	0.26	4.3	2.4	91
1,2-Dichloroethane	5.02	62	1	0.655	8.8	0.29	4.2	2.5	101
Fluorobenzene (IS 1)	5.28	96							
Trichloroethene	5.46	130	1	0.785	2.8	0.31	5.1	2.4	92
Dibromomethane	5.86	174	1	0.331	8.5	0.22	3.7	2.4	89
1,2-Dichloropropane	5.97	63	1	0.534	5.6	0.30	4.7	2.2	101
Bromodichloromethane	6.07	83	1	0.701	8.5	0.29	3.8	2.4	94

Compound	Calibration (2 to 200 ppb)					Method Detection Limit (n = 7, 2 ppb)		Midpoint Calibration Check (n = 7, 20 ppb)	
	Retention Time (min)	Quant Ion	IS	Average RRF	RRF (≤20% RSD R^2≥0.99)	MDL (ppb)	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
4-Methyl-2-Pentanone[2,4]	6.30	100	1	0.045	19.8	0.46	3.9	1.9	85
cis-1,3-Dichloropropene	6.74	75	1	0.792	1.8	0.29	4.7	2.0	96
Toluene-d8 (SURR)	6.95	98	1	1.75	2.2		0.6	0.6	100
Toluene	7.00	91	1	2.38	9.2	0.24	4.4	2.2	94
Tetrachloroethylene	7.40	166	1	1.04	11.7	0.37	6.8	3.4	88
trans-1,3-Dichloropropene	7.44	75	1	0.717	3.0	0.24	3.9	2.1	97
1,1,2-Trichloroethane	7.58	97	1	0.488	3.4	0.33	5.4	2.5	96
Dibromochloromethane	7.74	129	2	0.316	6.9	0.31	4.9	1.6	96
1,3-Dichloropropane	7.82	76	1	0.793	4.1	0.25	3.9	2.5	97
1,2-Dibromoethane	7.92	107	2	0.278	3.8	0.26	4.4	1.5	93
2-Hexanone2,4	8.18	43	2	0.058	9.3	1.32	8.1	2.4	87
Chlorobenzene-d5 (IS 2)	8.39	117							
Chlorobenzene	8.41	112	2	0.941	2.8	0.26	4.3	1.9	96
Ethylbenzene	8.46	91	2	1.26	12.8	0.22	4.5	1.9	99
1,1,1,2-Tetrachloroethane	8.47	131	2	0.338	4.4	0.28	4.6	1.8	90
m,p-Xylene[3]	8.59	91	2	1.02	15.0	0.45	4.8	1.6	105
o-Xylene	8.91	91	2	1.06	16.0	0.20	4.4	1.7	96
Bromoform	8.95	173	2	0.203	11.7	0.20	3.8	1.6	94
Styrene	8.96	104	2	0.826	17.0	0.25	5.9	1.6	98
Isopropylbenzene	9.17	105	2	1.29	15.9	0.21	4.7	1.9	98
4-Bromofluorobenzene (SURR)	9.35	95	2	0.455	3.5		0.8	0.9	103
Bromobenzene	9.41	77	2	0.592	6.8	0.30	4.6	1.8	93
n-Propylbenzene	9.47	91	2	1.46	17.3	0.24	5.4	2.2	98
1,1,2,2-Tetrachloroethane	9.51	83	3	0.580	18.0	0.42	5.0	4.6	97
2-Chlorotoluene	9.56	91	3	2.07	7.2	0.33	5.7	2.8	100
1,2,3-Trichloropropane[1,4]	9.59	75	3	0.676	0.997	1.03	15.0	4.4	106
1,3,5-Trimethylbenzene	9.63	105	3	2.63	8.1	0.27	5.0	3.2	102
4-Chlorotoluene	9.68	91	3	2.19	8.0	0.30	5.3	2.9	98
tert-Butylbenzene	9.85	119	3	2.29	5.3	0.24	4.4	3.5	99
1,2,4-Trimethylbenzene	9.89	105	3	2.41	13.0	0.30	6.2	2.8	103
sec-Butylbenzene	9.97	105	3	3.29	9.0	0.28	5.1	3.2	103
p-Isopropyltoluene	10.08	119	3	2.53	10.4	0.31	6.3	3.3	101
1,3-Dichlorobenzene	10.09	146	3	1.59	9.3	0.36	5.4	3.1	82
1,4-Dichlorobenzene-d4 (IS 3)	10.15	152							
1,4-Dichlorobenzene	10.16	146	3	1.52	7.1	0.55	8.2	2.7	84
n-Butylbenzene1	10.37	91	3	1.88	0.996	0.89	9.4	3.5	81
1,2-Dichlorobenzene	10.44	146	3	1.41	9.3	0.30	4.3	3.4	85
1,2-Dibromo-3-chloropropane	10.97	157	3	0.150	11.2	0.30	4.5	4.3	90
Hexachlorobutadiene	11.44	180	3	0.707	19.5	0.50	10.9	3.7	86
1,2,4-Trichlorobenzene	11.44	225	3	0.569	10.1	0.35	5.3	4.5	83
Naphthalene	11.65	128	3	1.31	17.5	0.48	9.3	4.7	89
1,2,3-Trichlorobenzene	11.77	180	3	0.693	19.1	0.35	7.3	4.6	87

[1] Compound calibrated by linear regression
[2] Calibration curve 2.5 to 500 ppb
[3] Calibration curve 2 to 400 ppb
[4] MDL calculated using 5 ppb
[5] MDL calculated using 25 ppb

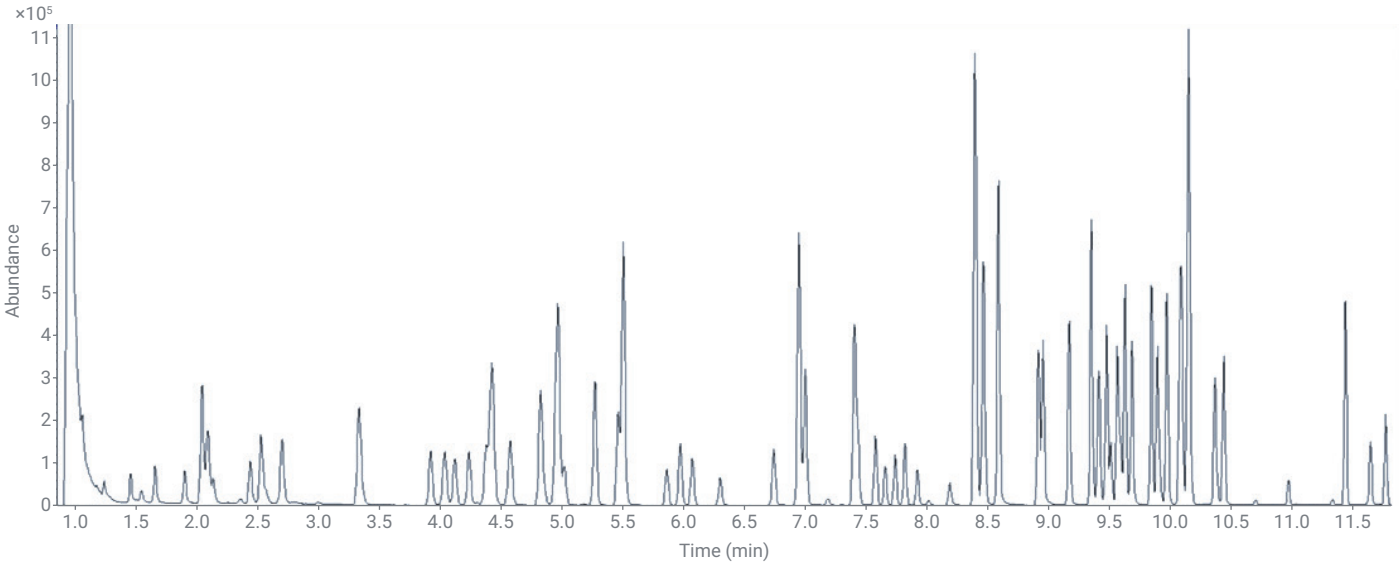

Figure 1. Total ion chromatogram of MEE Method HJ605 10 ppb VOC standard.

Conclusion

This study demonstrates the capability of the Teledyne Tekmar Atomx XYZ P&T system to process VOCs in soil samples following MEE Method HJ605 with detection by the Agilent 7890B GC and 5977B MS. The %RSDs of the calibration curve passed all method requirements. Furthermore, MDL and precision for seven 2 ppb standards showed minimal interference from excessive water. The average MDL analysis was 0.47 ppb with a 5.9% RSD. The mid-point calibration check for seven 20 ppb water standards displayed an average of less than 4% RSD, and an average recovery of 95% for the compounds of interest.

By making additional, appropriate changes to the P&T method and GC oven temperature program, the sample cycle time and moisture conveyed to the GC column may also be reduced. This can increase laboratory throughput in a 12-hour period and improve sensitivity.

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