APPLICATION NOTE: AN2103
TECHNOLOGY: P+T VOC
INDUSTRY: ENV

MEE Method HJ605 Using the Teledyne Tekmar Atomx XYZ and Thermo Scientific™ TRACE™ 1310 GC and ISQ™ 7000 MS with an ExtractaBrite Source

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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 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 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 life span. 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™, VOA (Ketones), and 502.2 Calibration Mix. In total, the standard contained 62 compounds.

The soil calibration curve was prepared from 2 parts per billion (ppb) to 200 ppb. The relative response factor (RF) 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 Restek standards at a concentration of 25 parts per million (ppm), after which 10 microliters (µL) was then mixed with each 5 milliliter (mL) sample for a resulting concentration of 50 ppb.

Seven 2 ppb soil standards were prepared for MDL and precision calculations. Seven 20 ppb soil standards were prepared for the mid-point calibration check, precision and accuracy. All calibration, MDL and mid-point calibration check samples were analyzed using the Atomx XYZ conditions in Table I and the GC/MS conditions in Table II

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Experimental Instrument Conditions

Table I Teledyne Tekmar Atomx XYZ Soil Method Conditions for HJ605			
Standby	**Variable**	**Purge**	**Variable**
Valve Oven Temp	140 ºC	MCS Purge Temp	20 ºC
Transfer Line Temp	140 ºC	Dry Purge Time	0.5 min
Sample Mount Temp	90 ºC	Dry Purge Flow	100 mL/min
Water Heater Temp	90 ºC	Dry Purge Temp	20 ºC
Sample Vial Temp	40 ºC	**Desorb**	**Variable**
Soil Valve Temp	100 ºC	Methanol Needle Rinse	Off
Standby Flow	10 mL/min	Water Needle Rinse Volume	7.00 mL
Purge Ready Temp	40 ºC	Sweep Needle Time	0.25 min
Purge	**Variable**	Desorb Preheat Temp	245 ºC
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 ºC
Pre-sweep Time	0.25 min	**Bake**	**Variable**
Water Volume	10.00 mL	Bake Time	2.00 min
Sweep Water Time	0.25 min	Bake Flow	400 mL/min
Sweep Water Flow	100 mL/min	Bake Temp	280 ºC
Sparge Vessel Heater	Off	MCS Bake Temp	180 ºC
Purge Mix Speed	Medium		
Purge Time	11.00 min		
Purge Flow	40 mL/min	**Trap**	#9
Purge Temp	20 ºC	**Purge Gas**	Nitrogen

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Table II Thermo Scientific TRACE 1310 GC and ISQ 7000 MS System Conditions for HJ605	
Thermo Scientific TRACE 1310 GC Conditions	
Column	TraceGOLD TG-VMS, 20 m x 0.18 mm, 1µm Film, Helium – 1 mL/min
Oven Profile	38 ºC, 1.80 min, 10 ºC/min to 120 ºC, 15 ºC/min to 240 ºC, 2 min Hold, Run Time 20 min
Inlet	200 ºC, 24:1 Split, Purge Flow 0.5 mL/min
Thermo Scientific ISQ 7000 MS Conditions	
Temp	Transfer Line 280 ºC; Ion Source 280 ºC
Scan	Range 35 *amu* to 270 *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 mid-point calibration check accuracy and precision data are shown in Table III. Figure 1 displays a 20 ppb standard, indicating excellent peak resolution with minimal water inference for all VOCs.

Table III HJ605 Soil Calibration, Method Detection Limit and Mid-Point Calibration Check Data

Compound	Calibration (2 ppb – 200 ppb)			Method Detection Limit (n=7, 2 ppb)		Mid-Point Calibration Check (n=7, 20 ppb)	
	Retention Time	Quant Ion	Linearity RF (≤20% RSD r^2≥0.99)	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
Dichlorodifluoromethane	1.11	85	6.1	0.43	5.0	5.9	128
Chloromethane	1.24	50	7.4	0.35	3.9	4.2	121
Vinyl Chloride	1.28	62	5.1	0.31	3.7	5.4	127
Bromomethane	1.49	94	16.9	0.62	5.0	8.6	130
Chloroethane	1.58	64	15.0	0.58	6.5	8.3	122
Trichlorofluoromethane	1.67	101	14.4	0.49	4.9	4.3	126
1,1-Dichloroethene	2.04	96	4.0	0.53	6.3	4.3	130
Carbon Disulfide	2.04	76	6.2	0.52	6.3	4.8	114
Iodomethane	2.13	142	10.7	0.26	5.6	7.3	118
Methylene Chloride	2.45	49	18.2	0.69	5.0	4.2	117
Acetone[123]	2.51	58	0.998	6.37	11.0	14.3	185
trans-1,2-Dichloroethene	2.57	61	2.8	0.55	6.9	3.5	126
1,1-Dichloroethane	3.01	63	3.9	0.46	5.2	2.8	130
cis-1,2-Dichloroethene	3.43	61	4.5	0.42	5.3	3.0	122
2,2-Dichloropropane	3.51	77	9.1	0.45	6.6	3.4	128
Bromochloromethane	3.58	128	6.5	0.52	6.0	3.8	130
Chloroform	3.65	83	3.9	0.36	4.3	3.0	130
Carbon Tetrachloride	3.75	117	15.6	0.26	5.6	4.8	99
Dibromofluoromethane (SURR)	3.80	111	2.4		1.9	1.6	126
1,1,1-Trichloroethane	3.81	87	11.0	0.32	5.2	4.2	127

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	Retention Time	Quant Ion	Linearity RF (≤20% RSD r^2≥0.99)	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
1,1-Dichloropropene[1]	3.91	75	0.997	0.28	1.5	8.9	121
2-Butanone[2]	3.91	43	12.7	1.38	5.5	3.4	101
Benzene	4.12	78	6.2	0.26	3.9	3.2	112
1,2-Dichloroethane	4.29	62	5.8	0.38	5.1	3.8	126
Fluorobenzene (IS)	4.48	96					
Trichloroethene	4.63	130	14.4	0.19	3.1	3.8	103
Dibromomethane	5.00	93	3.4	0.35	4.5	3.4	126
1,2-Dichloropropane	5.10	63	16.1	0.37	6.4	5.0	108
Bromodichloromethane	5.18	83	12.5	0.42	6.7	4.6	120
Toluene-d8 (SURR)	6.00	98	1.3		1.0	0.7	91
Toluene	6.05	91	4.6	0.38	5.5	3.6	99
Tetrachloroethene	6.45	166	5.9	0.45	5.4	7.8	117
1,1,2-Trichloroethane	6.67	100	8.4	0.38	6.0	4.6	107
4-Methyl-2-Pentanone[2]	6.67	83	11.9	2.68	12.7	9.1	103
Dibromochloromethane	6.85	129	11.9	0.34	5.5	4.5	110
1,3-Dichloropropane	6.96	76	10.6	0.42	6.4	3.8	109
1,2-Dibromoethane	7.08	107	11.9	0.33	6.4	5.7	84
2-Hexanone[2]	7.43	43	5.7	1.18	7.5	7.2	90
Chlorobenzene-d5 (IS)	7.69	117					
Chlorobenzene	7.71	112	6.5	0.43	6.1	3.8	100
Ethylbenzene	7.79	91	6.1	0.44	6.3	4.1	109
1,1,1,2-Tetrachloroethane	7.81	131	6.3	0.38	7.1	4.9	86
m-,p-Xylene	7.98	106	8.3	0.90	6.3	4.0	112
o-Xylene	8.48	106	4.1	0.55	8.0	4.2	109
Bromoform	8.54	173	9.9	0.27	3.8	4.7	83
Styrene	8.55	104	4.0	0.45	6.8	4.5	110
Isopropylbenzene	8.90	105	4.2	0.44	6.5	4.1	111
4-Bromofluorobenzene (SURR)	9.21	95	4.2		2.3	1.7	94
Bromobenzene	9.32	77	3.1	0.37	6.2	4.3	92
n-Propylbenzene	9.44	91	2.2	0.44	6.9	4.0	101
1,1,2,2-Tetrachloroethane	9.53	83	13.4	0.38	5.3	5.2	80
2-Chlorotoluene	9.59	91	3.5	0.42	6.6	4.2	99
1,3,5-Trimethylbenzene	9.72	105	3.5	0.51	8.0	4.1	102
4-Chlorotoluene	9.81	91	3.8	0.43	6.6	3.6	99
tert-Butylbenzene	10.12	119	2.7	0.45	7.1	4.0	102
1,2,4-Trimethylbenzene	10.22	105	2.8	0.47	7.4	4.2	102
sec-Butylbenzene	10.36	105	2.7	0.50	7.6	4.1	103
1,3-Dichlorobenzene	10.56	146	6.2	0.51	7.4	4.4	101
p-Isopropyltoluene	10.57	119	3.9	0.55	8.3	3.8	103
1,4-Dichlorobenzene-d4 (IS)	10.67	152					
1,4-Dichlorobenzene	10.68	146	6.2	0.47	6.7	3.8	98

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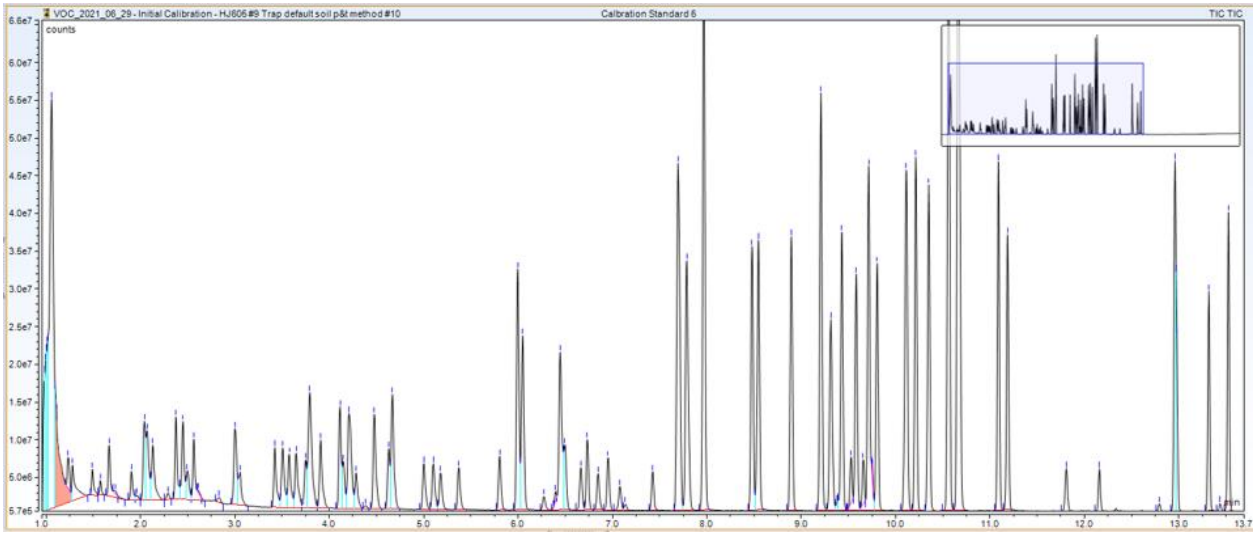
	Retention Time	Quant Ion	Linearity RF (≤20% RSD r^2≥0.99)	MDL	Precision (≤20%)	Precision (≤20%)	Accuracy (±30%)
n-Butylbenzene	11.10	91	5.1	0.56	8.1	4.0	104
1,2-Dichlorobenzene	11.19	146	3.1	0.42	6.3	4.0	99
1,2-Dibromo-3-Chloropropane	12.16	157	5.6	0.49	7.9	4.4	92
1,2,4-Trichlorobenzene	12.96	180	8.5	0.79	10.4	3.8	102
Hexachlorobutadiene	12.98	225	9.9	0.71	9.1	3.9	99
Naphthalene	13.32	128	4.1	0.62	9.2	3.6	101
1,2,3-Trichlorobenzene	13.53	180	5.6	0.76	10.2	3.8	101

1. Linear calibration
2. Calibration range from 2.5-500 ppb
3. Acetone contamination in the lab led to high recovery of the mid-point calibration check

Figure 1 Total Ion Chromatogram (TIC) of MEE Method HJ605 20 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 a Thermo Scientific TRACE 1310 GC and ISQ 7000 MS with an ExtractaBrite Source. The %RSD of the calibration curve passed all method requirements. Furthermore, MDL and precision for seven 2 ppb standards showed minimal interference from excessive water. For most compounds, MDL analysis resulted in values of <0.50 ppb. The mid-point calibration check for seven 20 ppb soil standards displayed all compounds, except for one, having <9% RSD with an average recovery of 109%. During the analysis, the laboratory had an acetone contamination which led to a high recovery for the mid-point calibration check.

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 be reduced, increasing laboratory throughput in a 12-hour period and improving sensitivity.

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