APPLICATION NOTE 000377

Analysis of volatile organic compounds in soil using purge and trap coupled to single quadrupole GC-MS

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Goal

To demonstrate the compliance of the Teledyne Tekmar Atomx XYZ purge and trap (P&T) system along with a Thermo Scientific™ ISQ™ 7610 single quadrupole mass spectrometer coupled to a Thermo Scientific™ TRACE™ 1610 gas chromatograph (GC) and Thermo Scientific™ Chromeleon™ Chromatography Data System (CDS) for the determination of volatile organic compounds (VOCs) in soil according to the Chinese Method HJ 605. Linearity, detection limit, precision, and accuracy were assessed to evaluate the method performance. A long-term study was also performed to demonstrate the robustness of the method.

Introduction

Analytical testing laboratories must monitor the environment to ensure that the public are not exposed to elevated levels of volatile organic compounds (VOCs). VOCs are generated by humans and produced in the processing of, or as, adhesives, petroleum products, paints, refrigerants, and pharmaceuticals. These compounds are of major concern globally, as they can contaminate the environment and cause negative health effects in humans who are exposed to elevated levels. There are several worldwide regulations in place to monitor the levels of VOCs in soil. The United States Environmental Protection Agency (U.S. EPA) has defined Method 8260 to monitor the levels of VOCs in soil.[1]

The Chinese Ministry of Ecology and Environment (MEE) has developed a standard method, HJ 605, to monitor VOCs in soil.[2] To perform HJ 605, method acceptance criteria must be achieved. These criteria include demonstrating the method detection limit (MDL) for all compounds in soil. The linear calibrations are required to have R^2 values of >0.99 or Relative Response Factor (RRF) RSD values of <20%. To ensure the sampling with the purge and trap is satisfactory, the recovery must be between 70 to 130% for all compounds. To assess the entire reproducibility of the method, the precision must be less than 25% for two consecutive injections for every compound. Meeting these criteria is critical, but analytical testing laboratories must also produce reproducible results day in and day out.

In this application note, purge and trap sampling was coupled to GC-MS to demonstrate HJ 605 for the analysis of VOCs in soil.

Experimental

An ISQ 7610 single quadrupole mass spectrometer equipped with a Thermo Scientific™ ExtractaBrite™ ion source was coupled to a TRACE 1610 GC and a Teledyne Tekmar Atomx XYZ P&T and used to assess the system suitability for the determination on VOCs in soil according to the Chinese MEE HJ 605 method. The Thermo Scientific™ NeverVent™ vacuum probe interlock (VPI) technology of the ISQ 7610 system allows users perform ion source maintenance and service the analytical columns without venting the mass spectrometer, significantly reducing instrument downtime and allowing continuous sample throughput. The Atomx XYZ concentrator's efficient trap cooling design reduces sample cycle time and allows for increased sample throughput. The moisture control system improves water vapor removal, thereby reducing peak interference and increasing GC column life span. Experimental conditions for the GC-MS system and the purge and trap are displayed in Tables 1 and 2.

Table 1. Teledyne Tekmar Atomx XYZ operating conditions for analysis of VOCs in soil

Standby	Variable	Purge	Variable
Valve oven temp.	140 ˚C	Purge temp.	20 ˚C
Transfer line temp.	140 ˚C	MCS purge temp.	20 ˚C
Sample mount temp	90 ˚C	Dry purge time	2.00 min
Water heater temp.	90 ˚C	Dry purge flow	100 mL/min
Sample cup temp.	40 ˚C	Dry purge temp.	20 ˚C
Soil valve temp.	100 ˚C	**Desorb**	**Variable**
Standby flow	10 mL/min	Methanol needle rinse	Off
Purge ready temp.	40 ˚C	Water needle rinse volume	7.00 mL
Purge	**Variable**	Sweep needle time	0.25 min
Pre-purge time	0.00 min	Desorb pre-heat temp.	245 ˚C
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 ˚C
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.	260 ˚C
Purge mix speed	Medium	MCS bake temp.	180 ˚C
Purge time	11.00 min	Trap	#9
Purge flow	40 mL/min	Purge gas	Nitrogen

Table 2. GC-MS operating conditions for analysis of VOCs in soil and sediment

TRACE 1610 GC conditions	
Column	TraceGOLD TG-VMS, 20 m x 0.18 mm, 1 µm film (P/N 26080-4950)
Carrier gas	Helium, 0.8 mL/min
Oven temp. 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	SSL, 200 ˚C, 30:1 split, purge flow 0.5 mL/min
ISQ 7610 MS conditions	
Temp.	Transfer line 230 ˚C; EI ion source 280 ˚C
Scan	Range 40 amu to 270 amu, solvent delay 0.50 min, dwell/scan time 0.20 s
Current	Emission current 25 µA, gain 3.00E+005

Data acquisition, processing, and reporting

Data was acquired, processed, and reported using Chromeleon CDS software, version 7.3. The integrated instrument control of the Atomx XYZ P&T ensures full automation from sequence set-up to data reporting, simplifying the instrument operation. Figure 1 shows the Atomx XYZ P&T, which is fully controllable using the Chromeleon CDS e-panel. The fully optimized method used within this application note and the eWorkflow™ are available for download in the Thermo Scientific™ AppsLab application note repository (www.appslab.com)[3]. Chromeleon CDS eWorkflows can be easily imported into the CDS, allowing the user to start a sequence, process the data, and generate results with only few clicks.

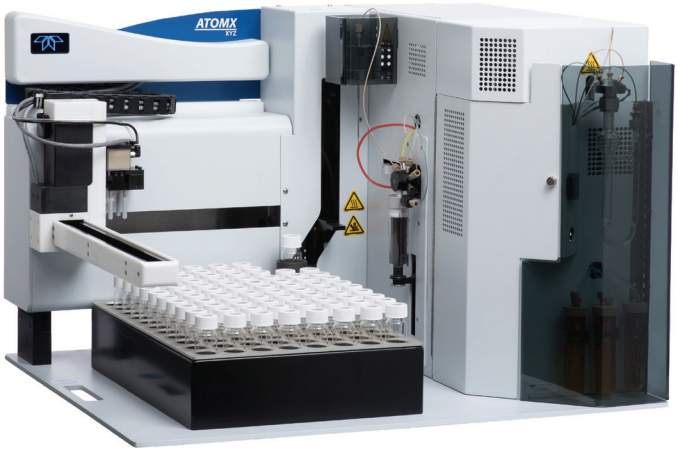


Figure 1. The Atomx XYZ P&T system fully controllable by Chromeleon CDS

Standard and sample preparation

To produce standards and simulated soil samples, standard mixes were spiked into reagent water. 8260B MegaMix® (P/N 30633), VOA (Ketones) (P/N 30006), and 502.2 Calibration Mix (P/N 30042) were purchased from Restek. The three mixes were combined using purge and trap grade methanol (Honeywell/Burdick & Jackson, P/N 232-1L) to obtain one working solution at 50 ppm. The full list of analyzed compounds is reported in Appendix 1.

Internal standards, fluorobenzene (P/N 30030), chlorobenzene-d_5 (P/N 30074) and 1,4-dichlorobenzene-d_4 (P/N 30074) as well as surrogates standards dibromofluoromethane (P/N 30240), toluene-d_8 (P/N 30240), and 4-bromofluorobenzene (P/N 30240) were purchased from Restek and diluted in methanol to a final concentration of 25 ppm.

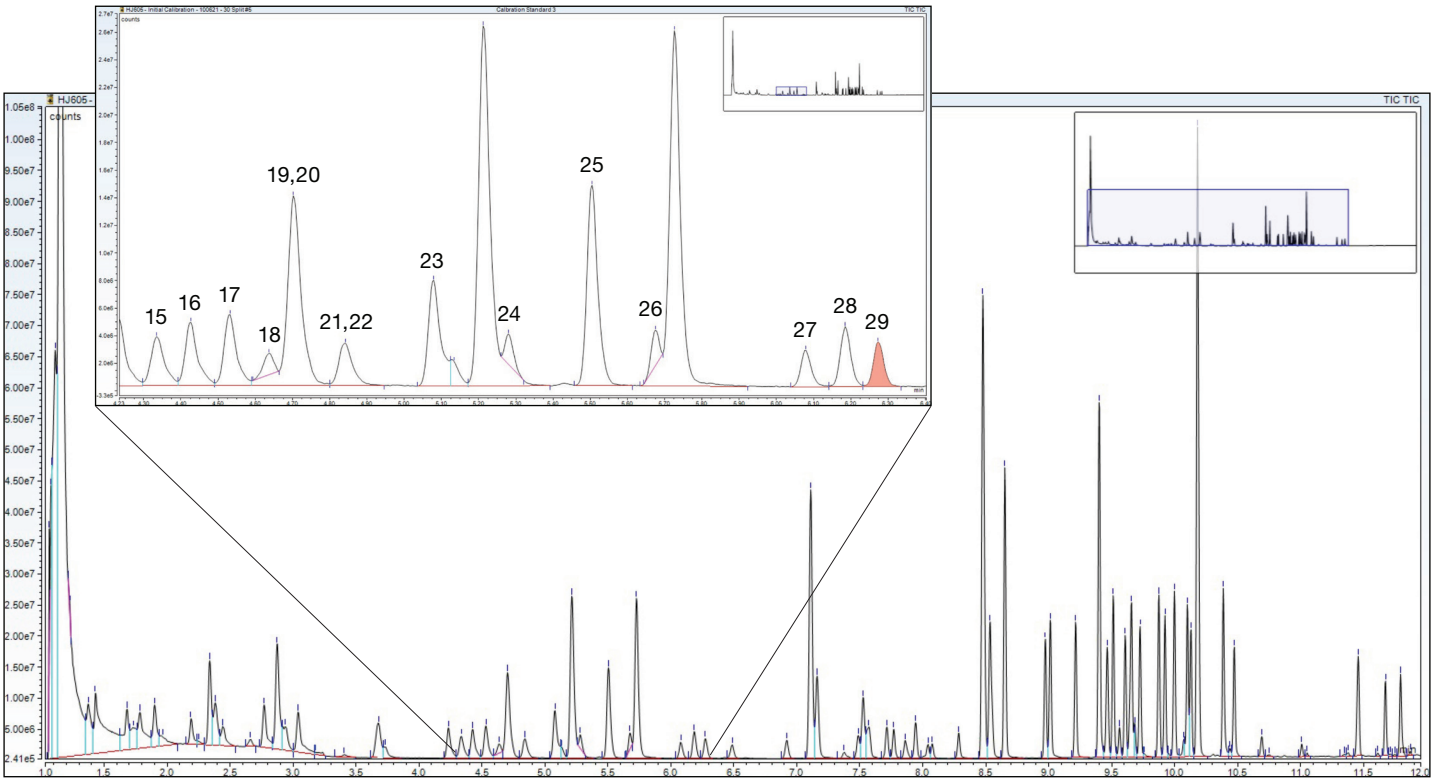
A calibration curve was prepared by diluting the working solutions with reagent water to obtain eight calibration levels ranging from 1 to 200 ppb. Each calibration level (5 mL) was spiked with ISTD and surrogate standards (final concentration 25 ppb). The ISTD and surrogate standards were used to calculate the relative response factor (RRF) for each compound.

Method detection limit (MDL) and peak area repeatability were assessed by using n=7 standards at 1 ppb. Soil standards (n=7) were prepared for the instrument check (20 ppb) and used to assess method precision and accuracy.

Results and discussion

Chromatography

Chromatographic separation was achieved for the 66 target compounds in 12 minutes. The effective water removal of the Atomx XYZ P&T combined with the superior inertness of the Thermo Scientific™ TraceGOLD™ TG-VMS column allowed for reduced moisture entering the GC system resulting in good peak shape and resolution. An example of chromatography for a 5 ppb VOC standard in water is reported in Figure 2.



Peaks:

15. 2,2-Dichloropropane
16. Bromochloromethane
17. Chloroform
18. Carbon tetrachloride
19. Dibromofluoromethane (surr)
20. 1,1,1-Trichloroethane
21. 1,1-Dichloropropene
22. 2-Butanone
23. Benzene
24. 1,2-Dichloroethane
25. Fluorobenzene (ISTD)
26. Trichloroethene
27. Dibromomethane
28. 1,2-Dichloropropane
29. Bromodichloromethane

Figure 2. Total ion chromatogram (TIC) obtained for reagent water spiked at 5 ppb. The inset highlights an example of peak shapes and resolution with minimal water interference.

Linearity and sensitivity

Linearity was assessed by injecting eight calibration levels ranging from 1 ppb to 200 ppb. Good linearity was obtained with coefficient of determination (R^2) of >0.99 and average response factor %RSD (AvCF %RSD) <20, confirming the linear trend across the specified concentration range and meeting the method requirements. The MDL and precision were assessed using n=7 reagent water samples spiked at 1 ppb. The MDLs had an average of 0.2 ppb and 3.5% RSD. Figure 3 shows a subset of the MDL data with calculated MDLs and precision for 30 compounds. R^2 as well as AvCF %RSD, calculated MDL, and amounts are reported in Appendix 1.

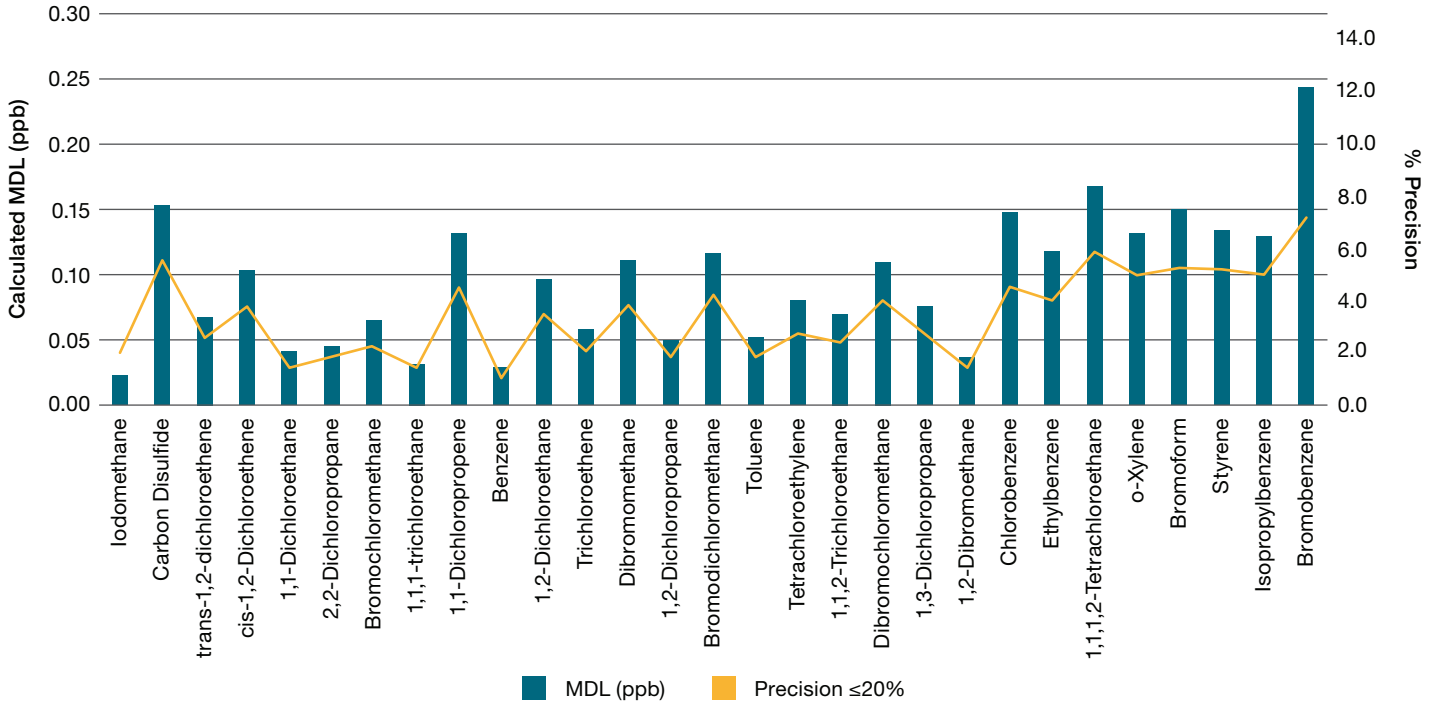


Figure 3. Calculated MDLs and precision for 30 analytes in soil

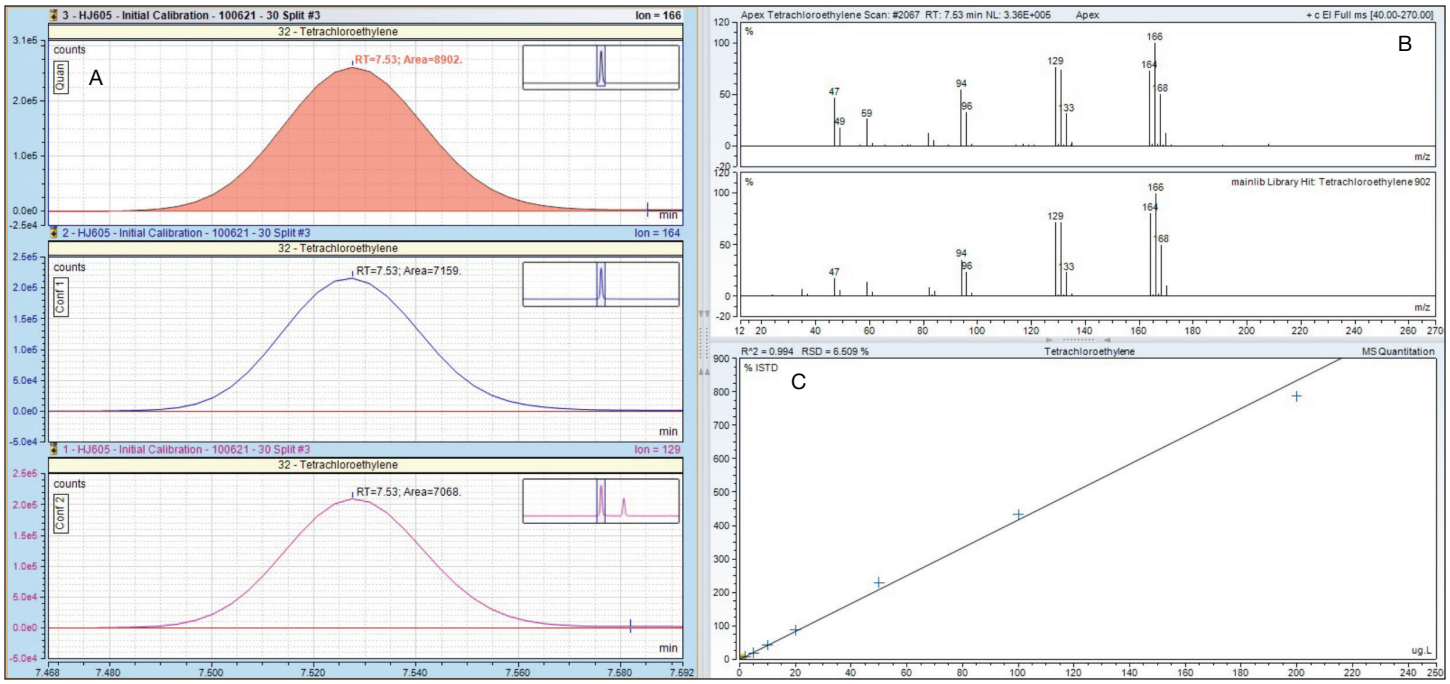


Figure 4. Chromeleon CDS results browser showing extracted ion chromatograms for tetrachloroethylene in the 1 ppb soil standard, quantitation ion (*m/z* = 166) and two confirming ions (*m/z* = 164, 129) (A), a matching measured spectrum to the NIST library (B), and a linear calibration over a concentration range of 1 ppb to 200 ppb (C)

As an example, the Chromeleon CDS results browser showing extracted ion chromatograms for tetrachloroethylene and 1,2-dibromoethane at 1 ppb, as well as the measured vs. NIST 20 library spectrum comparison and the calibration curve over a concentration range of 1 ppb to 200 ppb, are shown in Figures 4 and 5. Excellent linearities were obtained with R^2 values above 0.99 and average response factor RSDs <20%. The Thermo Scientific™ XLXR™ detection system on the ISQ 7610 GC-MS provides an extended linear dynamic range allowing for extended calibration curves. The detector also has an extended lifetime, which significantly reduces the need for replacement.

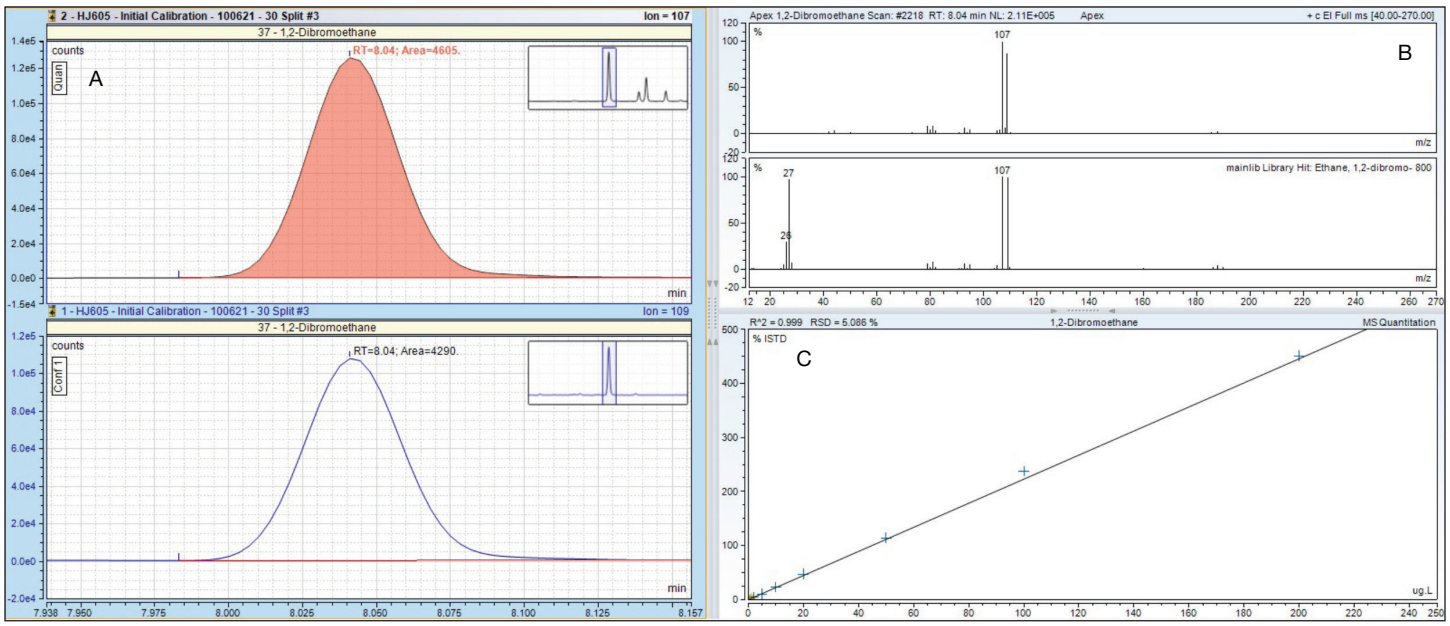


Figure 5. Chromeleon CDS results browser showing extracted ion chromatograms for 1,2-dibromoethane at 1 ppb in a soil standard, quantitation ion (*m/z* = 107) and one confirming ions (*m/z* = 109) (A), a matching measured spectrum to the NIST library (B), and a linear calibration over a concentration range of 1 ppb to 200 ppb (C)

Precision and accuracy

Precision and accuracy were assessed by using n=7 replicates of soil spiked at 20 ppb. For all compounds, the calculated amount was within 20% the spiked concentration with mean recovery within ±30% of the true value, which is an acceptance criterion for HJ 605. Results are detailed in Appendix 1. Figure 6 shows a cross section of compounds in the soil standard at 20 ppb, demonstrating good accuracy and precision.

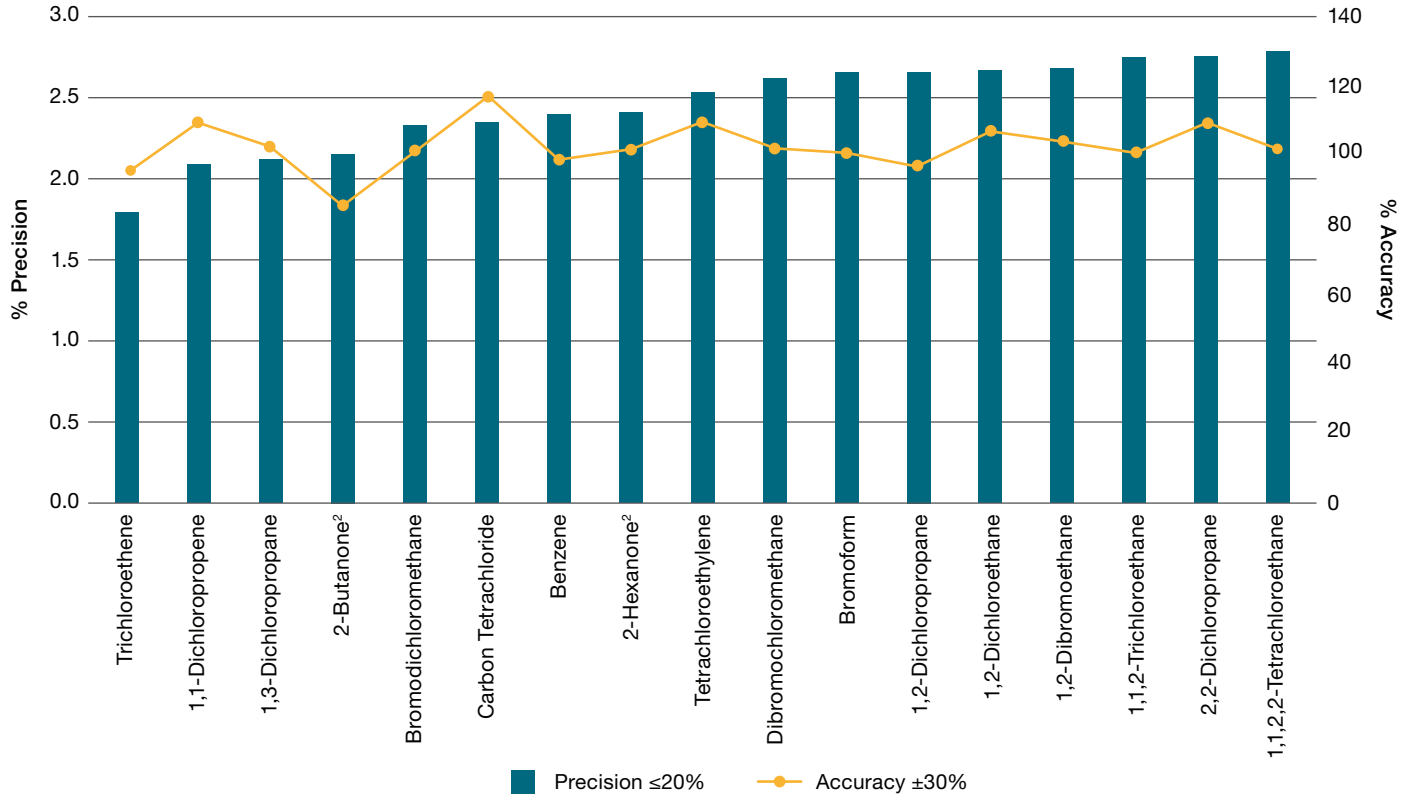


Figure 6. Accuracy (% recovery) and precision obtained by analyzing n=7 replicates of water standard at 20 ppb

Method robustness

To demonstrate the robustness of the analytical method, the reproducibility of the system was assessed by injecting 20 ppb soil QC samples at intervals over a 77-sample injection sequence that ran over two days. The samples were acquired with no user intervention at all on the P&T, GC, or MS system, and the absolute peak areas were plotted to demonstrate the stability of the results. Figure 7 shows the repeatability of ten of the compounds of the 12 QC standards injected over the sequence with excellent percentage RSDs. The accuracy and precision for every compound in the 12 QCs ran over 77 injections are shown in Appendix 2.

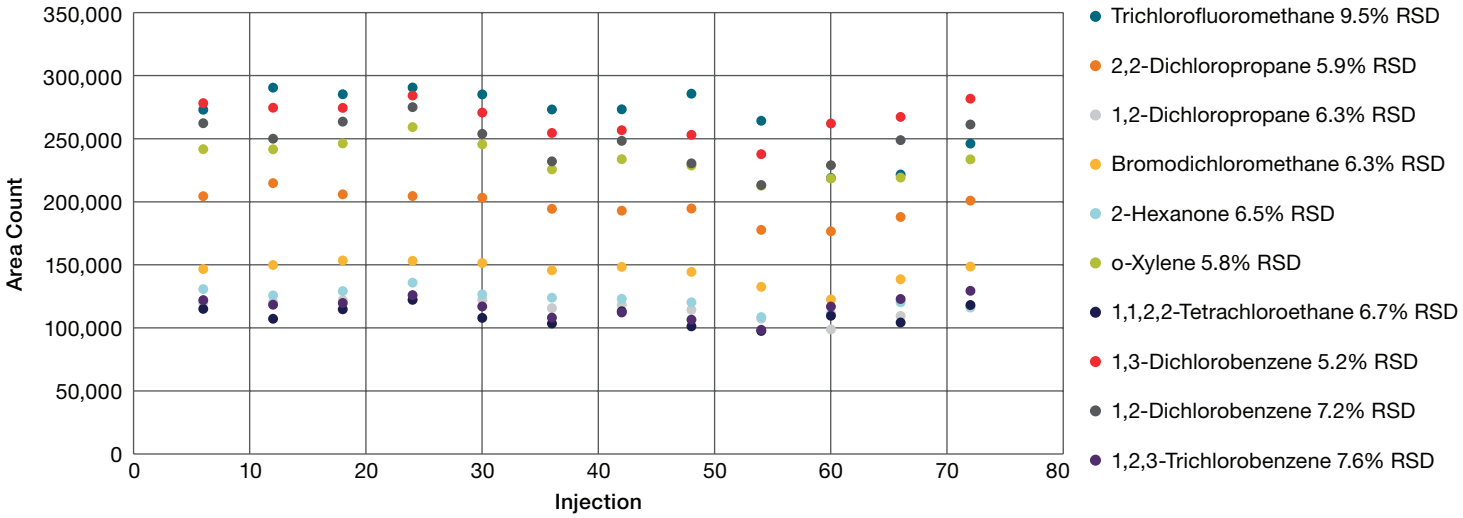


Figure 7. Repeatability (absolute peak area) of 12 QC 20 ppb soil standards assessed at regular intervals over n=77 consecutive sample injections corresponding to two days of analysis

Conclusion

The results shown in this study demonstrate that the TRACE 1610 GC coupled to the ISQ 7610 single quadrupole MS and the Atomx XYZ P&T provides a suitable tool for analytical testing laboratories analyzing environmental soil samples in compliance with the HJ 605 method.

- The ISQ 7610 VPI coupled with the Teledyne Tekmar Atomx XYZ P&T exceeds all the requirements outlined in HJ 605 method for analysis of VOCs in soil and sediment.
- Exceptional chromatography was obtained with good peak shape and baseline resolution for most of the compounds due to the effective water management of the Atomx XYZ P&T and the high inertness of the TraceGOLD TG-VMS column.
- The XLXR electron multiplier detector on the ISQ 7610 provided a wider linear dynamic range allowing for extended calibration curves (1–200 ppb) and excellent linearity for all compounds with $R^2 > 0.99$ and AvCF %RSD <20, confirming a good linear trend.
- Calculated MDLs for n=7 regent soil samples spiked at 1 ppb had an average of 0.2 ppb and 5.2% RSD.
- Precision and accuracy for n=7 reagent soil samples spiked at 20 ppb showed excellent results with calculated amounts ±20% the spiked concentrations and mean recovery of 103% for the compounds.
- System robustness was excellent showing reproducible results over two days of continuous analysis without user intervention.

References

1. Method 8260 Measurement of Volatile Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry https://www.epa.gov/sites/production/files/2017-04/documents/method_8260d_update_vi_final_03-13-2017.pdf
2. Summary of HJ 605 https://www.chinesestandard.net/PDF/English.aspx/HJ605-2011
3. Thermo Scientific AppsLab Library (https://appslab.thermofisher.com/)

Appendix 1 – Calibration, MDL, and midlevel check results for soil standard

Compound	Calibration (1 ppb–200 ppb)				Method detection limit (n=7, 1 ppb)		Midpoint check (n=7, 20 ppb)	
	Ret.Time	Quant Ion	Avg. RF	Linarity (%RSD)	MDL	Precision ≤20%	Precision ≤20%	Accuracy ±30%
Dichlorodifluoromethane	1.23	85	1.31	9.4	0.05	1.9	3.9	97
Chloromethane	1.37	50	1.87	8.3	0.12	4.1	5.6	97
Vinyl Chloride	1.44	62	1.93	8.4	0.04	1.5	3.8	100
Bromomethane	1.68	94	1.15	19.6	0.09	2.1	4.5	95
Chloroethane	1.79	64	1.06	8.7	0.12	4.4	4.0	88
Trichlorofluoromethane	1.90	101	1.92	6.6	0.06	2.4	4.3	94
1,1-Dichloroethene	2.34	96	1.34	8.9	0.06	2.3	4.9	98
Iodomethane	2.45	142	1.74	18.4	0.02	2.0	5.7	107
Carbon Disulfide	2.78	76	1.08	9.2	0.15	5.6	4.6	102
Methylene Chloride[1, 4]	2.88	49	6.23	0.996	0.59	9.3	6.0	121
Acetone[1, 2, 5]	2.95	58	0.251	0.995	2.06	16.2	5.8	77
trans-1,2-dichloroethene	3.04	61	2.29	8.3	0.07	2.6	3.9	104
2-Chloro-1,3-Butadiene	3.65	53	1.18	14.1	0.07	2.5	3.9	103
cis-1,2-Dichloroethene	3.68	61	0.230	7.6	0.10	3.8	3.4	102
1,1-Dichloroethane	3.69	63	2.47	8.2	0.04	1.4	3.6	103
2,2-Dichloropropane	4.34	77	1.41	13.6	0.04	1.8	2.8	110
Bromochloromethane	4.43	128	0.389	7.6	0.06	2.3	2.9	106
Chloroform	4.53	83	1.97	6.9	0.04	1.4	3.2	106
Carbon Tetrachloride	4.64	117	0.798	10.2	0.29	10.9	2.4	117
Dibromofluoromethane (surr)	4.71	111	0.539	3.4	-	1.9	1.8	105
1,1,1-trichloroethane	4.71	97	1.21	17.2	0.03	1.4	3.0	105
1,1-Dichloropropene	4.84	75	0.996	9.9	0.13	4.6	2.1	110
2-Butanone[2]	4.85	43	0.346	11.3	0.32	3.5	2.2	86
Benzene	5.08	78	3.33	6.7	0.03	1.0	2.4	98
1,2-Dichloroethane	5.28	62	1.32	8.4	0.10	3.5	2.7	108
Fluorobenzene (ISTD)	5.50	96	-	-	-	-	-	-
Trichloroethene	5.68	130	0.585	11.7	0.06	2.0	1.8	95
Dibromomethane	6.08	93	0.559	6.3	0.11	4.0	3.3	106
1,2-Dichloropropane	6.19	63	0.894	9.3	0.05	1.8	2.7	97
Bromodichloromethane	6.28	83	1.07	13.4	0.12	4.4	2.3	101
cis-1,3-Dichloropropene	6.92	75	1.11	6.8	0.06	2.2	3.5	102
Toluene-d8 (surr)	7.11	98	2.06	4.7	-	1.9	1.6	109
Toluene	7.16	91	4.19	8.0	0.05	1.8	3.1	109
Tetrachloroethylene	7.53	166	1.05	6.5	0.08	2.7	2.5	110
4-methyl-2-pentanone[2]	7.56	100	0.036	14.2	0.40	5.5	3.5	114
trans-1,3-Dichloropropene	7.58	75	1.110	9.8	0.06	2.4	2.6	94
1,1,2-Trichloroethane	7.71	83	0.563	5.5	0.07	2.3	2.8	101

Appendix 1 (continued) – Calibration, MDL, and midlevel check results for soil standard

Compound	Calibration (1 ppb–200 ppb)				Method detection limit (n=7, 1 ppb)		Midpoint check (n=7, 20 ppb)	
	Ret.Time	Quant Ion	Avg. RF	Linarity (%RSD)	MDL	Precision ≤20%	Precision ≤20%	Accuracy ±30%
Dibromochloromethane	7.86	129	0.493	9.3	0.11	4.0	2.6	102
1,3-Dichloropropane	7.95	76	1.20	7.0	0.08	2.7	2.1	103
1,2-Dibromoethane	8.04	107	0.558	5.1	0.04	1.3	2.7	104
2-Hexanone[2]	8.29	43	0.420	8.9	0.40	4.8	2.4	102
Chlorobenzene-d5 (ISTD)	8.48	117	-	-	-	-	-	-
Chlorobenzene	8.49	112	1.29	7.5	0.15	4.6	3.6	95
Ethylbenzene	8.53	91	2.52	9.4	0.12	4.0	3.0	101
1,1,1,2-Tetrachloroethane	8.55	131	0.269	7.1	0.17	5.9	2.9	98
m,p-Xylene[3]	8.65	106	0.972	13.8	0.23	4.4	2.9	100
o-Xylene	8.97	106	0.857	16.8	0.13	4.9	3.2	106
Bromoform	9.01	173	0.184	8.7	0.15	5.2	2.7	101
Styrene	9.01	104	1.43	14.3	0.13	5.2	3.5	109
Isopropylbenzene	9.21	105	2.22	17.0	0.13	5.0	2.8	105
4-Bromofluorobenzene (surr)	9.40	95	1.17	3.7	-	2.7	1.1	98
Bromobenzene	9.47	77	2.62	11.0	0.24	7.2	3.7	93
n-Propylbenzene	9.51	91	6.15	14.3	0.21	7.3	3.6	97
1,1,2,2-Tetrachloroethane	9.56	83	0.863	8.1	0.23	7.9	2.8	103
2-Chlorotoluene	9.61	91	3.80	12.5	0.25	8.1	3.6	97
1,2,3-Trichloropropane	9.64	75	1.03	10.1	0.21	6.6	4.2	96
1,3,5-Trimethylbenzene	9.66	105	3.76	13.6	0.25	8.5	3.9	124
4-Chlorotoluene	9.72	91	3.86	12.7	0.23	7.7	4.1	98
tert-Butylbenzene	9.87	119	2.85	17.3	0.24	9.3	4.6	104
1,2,4-Trimethylbenzene	9.93	105	3.47	18.3	0.24	10.3	4.6	106
sec-Butylbenzene	10.00	105	4.99	13.0	0.24	8.0	4.2	120
p-Isopropyltoluene	10.10	119	3.39	17.3	0.28	9.8	6.1	124
1,3-Dichlorobenzene	10.13	146	1.97	9.1	0.22	6.76	4.9	93
1,4-Dichlorobenzene-d4 (ISTD)	10.18	152	-	-	-	-	-	-
1,4-Dichlorobenzene	10.19	146	2.03	10.9	0.31	9.0	4.6	94
n-Butylbenzene	10.39	91	4.25	12.6	0.28	9.1	4.6	121
1,2-Dichlorobenzene	10.47	146	1.75	10.4	0.24	7.3	4.2	98
1,2-Dibromo-3-chloropropane	11.01	157	0.149	15.7	0.20	6.9	3.8	99
Hexachlorobutadiene	11.45	225	0.078	14.5	0.23	7.6	5.3	97
1,2,4-Trichlorobenzene	11.46	180	0.877	12.2	0.40	11.7	4.8	97
Naphthalene	11.67	128	2.32	14.9	0.39	12.0	3.4	105
1,2,3-Trichlorobenzene	11.79	180	0.861	12.0	0.31	8.65	4.9	99

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

Appendix 2 – Repeatability of a 20 ppb VOC soil standard assessed at intervals over n=77 consecutive injections for n=12 injections

Compound	Analyte recover n=12, 77 injections	
	Precision ≤20%	Accuracy ±30%
Dichlorodifluoromethane	10.3	93
Chloromethane	10.3	91
Vinyl Chloride	9.3	92
Bromomethane	8.7	93
Chloroethane	9.2	89
Trichlorofluoromethane	8.2	94
1,1-Dichloroethene	9.4	96
Iodomethane	10.4	101
Carbon Disulfide	7.0	100
Methylene Chloride	6.8	124
Acetone	6.1	189
trans-1,2-dichloroethene	8.3	103
cis-1,2-Dichloroethene	5.1	102
1,1-Dichloroethane	5.0	102
2,2-Dichloropropane	4.5	103
Bromochloromethane	4.9	104
Chloroform	4.5	105
Carbon Tetrachloride	8.5	119
Dibromofluoromethane (surr)	2.4	106
1,1,1-trichloroethane	7.5	107
1,1-Dichloropropene	7.6	107
2-Butanone	2.9	79
Benzene	5.3	99
1,2-Dichloroethane	4.5	104
Fluorobenzene (ISTD)	-	-
Trichloroethene	6.2	97
Dibromomethane	4.7	103
1,2-Dichloropropane	3.7	96
Bromodichloromethane	4.0	101
Toluene-d8 (surr)	2.0	111
Toluene	5.3	106
Tetrachloroethylene	7.2	115
4-methyl-2-pentanone	4.7	103
1,1,2-Trichloroethane	4.2	101
Dibromochloromethane	4.0	101
1,3-Dichloropropane	4.0	101
1,2-Dibromoethane	4.4	103

Appendix 2 (continued) – Repeatability of a 20 ppb VOC soil standard assessed at intervals over n=77 consecutive injections for n=12 injections

Compound	Analyte recover n=12, 77 injections	
	Precision ≤20%	Accuracy ±30%
2-Hexanone	3.7	87
Chlorobenzene-d5 (ISTD)	-	-
Chlorobenzene	6.0	89
Ethylbenzene	6.4	93
1,1,1,2-Tetrachloroethane	5.2	96
m,p-Xylene	5.8	93
o-Xylene	5.8	96
Bromoform	4.7	97
Styrene	5.9	101
Isopropylbenzene	6.6	97
4-Bromofluorobenzene (surr)	2.2	96
Bromobenzene	7.0	86
n-Propylbenzene	8.2	88
1,1,2,2-Tetrachloroethane	7.8	88
2-Chlorotoluene	7.4	88
1,2,3-Trichloropropane	6.9	88
1,3,5-Trimethylbenzene	6.8	114
4-Chlorotoluene	7.9	88
tert-Butylbenzene	7.3	96
1,2,4-Trimethylbenzene	6.7	96
sec-Butylbenzene	7.9	112
p-Isopropyltoluene	8.1	119
1,3-Dichlorobenzene	7.8	85
1,4-Dichlorobenzene-d4 (ISTD)	-	-
1,4-Dichlorobenzene	8.3	83
n-Butylbenzene	10.5	104
1,2-Dichlorobenzene	7.3	88
1,2-Dibromo-3-chloropropane	7.0	86
Hexachlorobutadiene	7.8	88
1,2,4-Trichlorobenzene	13.9	79
Naphthalene	6.8	89
1,2,3-Trichlorobenzene	9.2	85

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