

Analysis of VOCs and Petroleum Hydrocarbons (C₆ to C₉) in Soil and Sediment with a P&T-8890 GC/FID/5977 MSD System

Method HJ 605-2011 and HJ 1020-2019

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Abstract

HJ 605-2011 is primarily used by environmental labs in China for the analysis of volatile organic compounds (VOCs) in soil and sediment, while HJ 1020-2019 is used for the determination of petroleum hydrocarbons (C₆ to C₉) in the same matrix. This application note is targeted at labs that analyze both VOCs and petroleum hydrocarbons (C₆ to C₉) in soil and sediment. The Teledyne Tekmar Atomx XYZ purge and trap (P&T) system and an Agilent 8890 gas chromatograph (GC) configured with a flame ionization detector (FID) and a mass spectrometric detector (MSD) enable a unified GC configuration that provides the flexibility to run HJ 605 and HJ 1020 on a single instrument. The results of this application note meet all performance criteria outlined by HJ 605-2011 and HJ 1020-2019 methods.

Introduction

Soil contamination has been a growing concern because it can be a source of groundwater (drinking water) contamination and contaminated soils can also reduce the usability of land for development. In support of its waste management program, the Chinese Ministry of Environmental Protection has developed various methods to test for contaminants in environmental samples. VOCs and petroleum hydrocarbons are frequently detected pollutants in soil and sediments. HJ 605-2011 details the determination of VOCs in soil and sediment using P&T and GC/MS systems.¹ HJ 1020-2019 describes the analysis of petroleum hydrocarbons (C₆ to C₉) in the same matrix using P&T and GC/FID.² Many environmental labs follow these two standards for the detection of those types of compounds, using two separate GC systems. The two standards use the same sample pretreatment equipment and target the same matrix with different analytes, using different GC detectors. An Agilent 8890 GC configured with two detectors (FID and MSD) was used to measure VOCs and petroleum hydrocarbons. A purged two-way CFT device was used to split the column effluent 1:1 between the MSD and FID, forming a versatile system suited for routine analysis of the two types of compounds in soil samples.

This application note describes a well established analytical method for VOCs and petroleum hydrocarbon (C₆ to C₉) analysis, respectively, with one GC system configured for both FID and MSD. The linearity, repeatability, method detection limit (MDL), and recovery for the targeted 65 VOCs and petroleum hydrocarbons were tested on the new, combined platform.

Experimental

An Agilent 8890 GC, an Agilent 5977B MSD equipped with FID, and a Teledyne Tekmar Atomx XYZ sampler were used for this series of experiments. A purged two-way CFT device was used

to split the column effluent 1:1 between the MSD and FID. Figure 1 shows the configuration of the system. Tables 1 and 2 show the parameter details. P&T, GC, and MSD parameters are compatible with HJ 605 and HJ 1020 method guidelines.

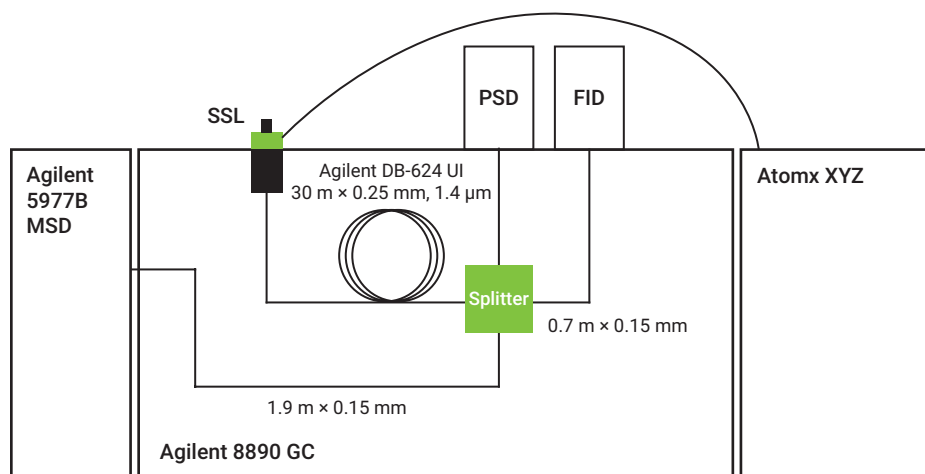


Figure 1. Configuration of the system.

Table 1. Teledyne Tekmar Atomx XYZ soil method conditions.

Parameter	Value	Parameter	Value
Standby		Desorb	
Valve Oven Temperature	140 °C	Water Needle Rinse Volume	7 mL
Transfer Line Temperature	140 °C	Sweep Needle Time	0.25 min
Sample Mount Temperature	90 °C	Desorb Preheat Temperature	245 °C
Water Heater Temperature	90 °C	GC Start Signal	Begin desorb
Sample Cup Temperature	20 °C	Desorb Time	2 min
Soil Valve Temperature	100 °C	Drain Flow	300 mL/min
Standby Flow	10 mL/min	Desorb Temperature	250 °C
Purge Ready Temperature	40 °C	Bake	
Purge		Bake Time	2 min
Water Volume	10 mL	Bake Flow	200 mL/min
Purge Mix Speed	Medium	Bake Temperature	280 °C
Purge Time	11 min	MCS Bake Temperature	180 °C
Purge Flow	40 mL/min	Trap	No. 9
Purge Temperature	20 °C		
MCS Purge Temperature	20 °C		
Dry Purge Time	2 min		
Dry Purge Flow	100 mL/min		

Chemicals, standards and sample preparation

65 VOCs and petroleum hydrocarbon (C₆ to C₉) standard mixtures were purchased from ANPEL Scientific Instrument Co. Ltd. (Shanghai, China). The single standards of fluorobenzene, chlorobenzene-d₅, 1,4-dichlorobenzene-d₄, dibromofluoromethane, toluene-d₈, 4-bromofluorobenzene, 2-methylpentane, and *n*-decane were also purchased from ANPEL. Three soil samples (sample 1, sample 2, and sample 3) were obtained from one customer's lab for tests. Samples 1 and 2 were used for recovery testing, while sample 3 was used for real sample analysis.

The stock solution of 65 VOCs at a concentration of 10 µg/mL was prepared in methanol. An internal standard (IS) mixture of fluorobenzene (ISTD 1), chlorobenzene-d₅ (ISTD 2), and 1,4-dichlorobenzene-d₄ (ISTD 3) was diluted with methanol at 25 µg/mL. A surrogate mixture of dibromofluoromethane, toluene-d₈, and 4-bromofluorobenzene at 10 µg/mL was prepared in methanol. The 10 µg/mL stock solution and 10 µg/mL surrogate solution were diluted with water to create 5, 10, 20, 50, 100, and 200 µg/L calibration solutions. Six vials (40 mL) were prepared at each calibration level by filling each vial with 5 mL of calibration solutions. A 10 µL amount of internal standard (25 µg/mL) was introduced into each vial automatically using Atomx XYZ.

A stock solution of petroleum hydrocarbons at a concentration of 500 µg/mL was prepared in methanol. The stock solution was diluted with water to create 0, 0.1, 0.2, 1, 2, and 6 µg/mL calibration solutions. Six vials (40 mL) were prepared at each calibration level by filling each vial with 5 mL of calibration solutions.

Table 2. Agilent 8890 GC and 5977B MSD system conditions.

Agilent 8890 GC Conditions	
Inlet	SSL, 250 °C, split 15:1
Liner	Straight, deactivated, 2 mm id (p/n 5181-8818)
CFT Device	Purged two-way splitter (p/n G3180-60501), split ratio 1:1 MSD:FID
PSD	3.8 psi constant pressure
Column	Agilent DB-624 Ultra Inert, 30 m × 0.25 mm, 1.4 µm (p/n 122-1334UI)
Carrier	Helium, 1.5 mL/min, constant flow
Oven	1. 38 °C (1.8 min), then 10 °C/min to 120 °C, then 15 °C/min to 240 °C (2 min), recommended by HJ605 method 2. 38 °C (1 min), then 3.8 °C/min to 80 °C (1 min), then 10 °C/min to 105 °C (5 min), then 10 °C/min to 150 °C (1 min), then 10 °C/min to 180 °C (5 min), recommended by HJ1020 method
FID Restrictor	0.7 m × 0.15 mm id deactivated fused silica tubing (p/n 160-2625-10)
MSD Restrictor	1.9 m × 0.15 mm id deactivated fused silica tubing (p/n 160-2625-10)
FID	Temperature: 250 °C, hydrogen: 30 mL/min, air: 300 mL/min Column + make-up gas (N ₂) = constant: 25 mL/min
MSD Transfer Line Temperature	250 °C
Agilent 5977B MSD	
Source Type	EI
Source Temperature	230 °C
Quad Temperature	150 °C
Drawout Plate	3 mm
Tune File	Atune.u
Acquisition Type	SCAN

Results and discussion

Volatile organic compounds analysis

The MSD data for VOCs were collected in SCAN mode and analyzed using Agilent MassHunter software. Figure 2 shows a chromatogram of 65 target compounds at a concentration of 50 µg/L. Excellent peak shape was obtained for most analytes. The six early eluting compounds are extremely volatile and coelute with water and

methanol. Therefore, working solutions for these compounds should be freshly prepared before injection. The amount of methanol in the working solution may affect the peak shape and response of the analytes. To obtain a stable and reliable response, the volume of methanol in each working solution should be kept constant.

The ISTD quantitative method is used in the HJ 605-2011 method, and the instrument repeatability and linearity

performance were verified based on concentration quantitation results instead of the absolute response of targeted analytes. Seven consecutive analyses of calibration solution were run at 20 µg/L. The %RSD of detected concentrations for 65 VOCs were in the range of 0.6 to 5.5%, which demonstrated excellent quantitation precision. Table 3 and Figure 3 show the details for all compounds, which met the criteria of less than 20% RSD required in the HJ 605 method.

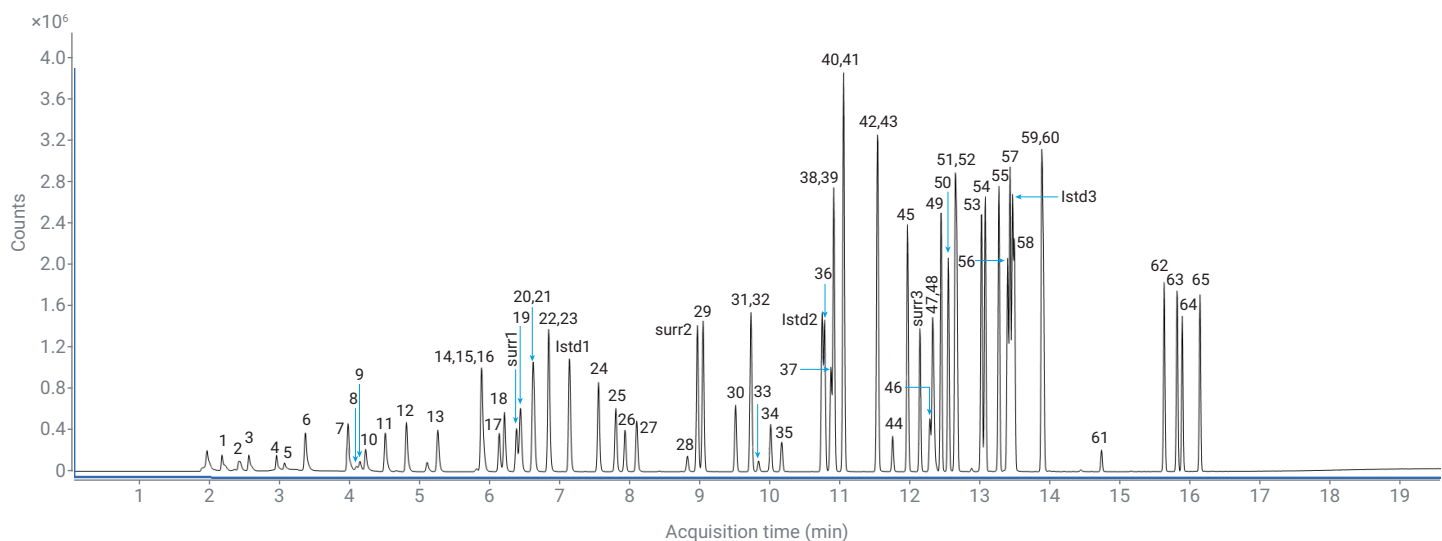


Figure 2. Total ion chromatogram of the 65 target compounds (50 µg/L).

Table 3. Analysis results for VOCs.

No.	Name	RT	m/z	CF R ²	MDL (µg/kg)	Concentration RSD% (n = 7)	Mean Recovery % (n = 4)	
							Sample 1	Sample 2
1	Dichlorodifluoromethane	2.182	85.0	0.99984	0.7	2	91.1	90.6
2	Chloromethane	2.429	50.0	0.99987	1.4	4.7	90.9	87.1
3	Chloroethene	2.564	62.0	0.99989	0.6	1.6	89.6	88.0
4	Bromomethane	2.964	94.0	0.99955	0.6	1.8	85.3	90.2
5	Chloroethane	3.076	64.0	0.99810	1.1	5.5	87.1	90.7
6	Trichloromonofluoromethane	3.376	101.0	0.99987	0.2	4.7	87.2	88.7
7	1,1-Dichloroethene	3.982	96.0	0.99997	0.2	2.5	90.5	90.6
8	Acetone	4.100	58.0	0.99955	0.5	2.1	90.6	119.4
9	Iodomethane	4.152	142.0	0.99902	1.0	1.3	96.6	93.7
10	Carbon disulfide	4.235	76.0	0.99934	0.3	3.5	92.2	89.6
11	Methylene chloride	4.517	84.0	0.99999	0.2	0.7	94.6	92.1
12	trans-1,2-Dichloroethene	4.817	96.0	0.99974	0.4	0.7	96.2	91.7
13	1,1-Dichloroethane	5.264	63.0	0.99998	0.3	1.2	95.0	94.3
14	cis-1,2-Dichloroethene	5.888	95.9	0.99995	0.6	0.6	96.4	91.7
15	2,2-Dichloropropane	5.894	77.0	0.99998	0.5	1.1	93.9	95.1
16	2-Butanone	5.923	72.0	0.99921	0.8	3.8	101.0	109.3
17	Bromochloromethane	6.141	130.0	1.00000	0.4	1.2	96.9	93.0
18	Chloroform	6.217	83.0	0.99986	0.2	1.4	93.5	92.9
Surr 1	Dibromofluoromethane	6.388	111.0	0.99999	0.4	2	94.8	93.2
19	1,1,1-Trichloroethane	6.441	97.0	0.99998	0.3	1.4	92.1	92.7
20	1,1-Dichloropropene	6.623	75.0	0.99988	0.4	1.3	95.4	92.2
21	Carbon tetrachloride	6.635	119.0	0.99999	0.5	2.1	93.0	93.3
22	Benzene	6.846	78.0	0.99998	0.4	1.2	95.1	94.5
23	1,2-Dichloroethane	6.852	62.0	0.99948	0.2	2	94.8	96.6
24	Trichloroethylene	7.558	132.0	0.99985	0.3	0.6	96.3	93.6
25	1,2-Dichloropropane	7.805	63.0	0.99997	0.4	1.2	97.0	97.0
26	Dibromomethane	7.935	174.0	0.99981	0.3	1.1	97.8	93.7
27	Bromodichloromethane	8.105	83.0	0.99994	0.4	1.6	95.3	94.8
28	4-Methyl-2-pentanone	8.829	85.0	0.99446	3.6	4	113.6	117.7
Surr 2	Toluene-D8	8.970	98.0	0.99979	0.5	1.1	93.5	91.3
29	Toluene	9.052	92.0	0.99995	0.5	1.2	93.2	91.4
30	1,1,2-Trichloroethane	9.517	83.0	0.99998	0.3	1.1	97.1	97.9
31	Tetrachloroethylene	9.735	166.0	0.99974	0.2	1.6	97.1	97.7
32	1,3-Dichloropropane	9.735	76.0	0.99989	0.3	0.9	91.1	87.0
33	2-Hexanone	9.846	43.0	0.99751	1.0	4.4	103.7	117.6
34	Dibromochloromethane	10.017	129.0	0.99934	0.6	2	95.1	93.7
35	1,2-Dibromoethane	10.176	107.0	0.99960	0.3	1	98.3	96.3
36	Chlorobenzene	10.787	112.0	0.99998	0.3	1.5	93.0	89.2
37	1,1,1,2-Tetrachloroethane	10.876	131.0	0.99984	0.5	1.6	92.6	90.4
38	Ethylbenzene	10.917	91.0	0.99866	0.4	1.4	91.7	89.7
39	1,1,2-Trichloropropane	10.923	63.0	0.99897	0.3	1.4	94.5	96.2
40,41	m,p-Xylene	11.058	106.0	0.99913	0.3	1.3	91.1	88.5
42	o-Xylene	11.534	91.1	0.99899	0.5	1.2	92.5	89.6
43	Styrene	11.546	104.0	0.99889	0.4	1.5	92.5	87.9
44	Bromoform	11.758	173.0	0.99858	0.6	1.8	97.6	94.9
45	Isopropylbenzene	11.970	105.0	0.99986	0.8	1.6	96.1	91.9

No.	Name	RT	m/z	CF R ²	MDL (µg/kg)	Concentration RSD% (n = 7)	Mean Recovery % (n = 4)	
							Sample 1	Sample 2
Surr 3	4-Bromofluorobenzene	12.146	174.0	0.99924	0.7	1.7	97.9	89.6
46	1,1,2,2-Tetrachloroethane	12.287	83.0	0.99912	0.4	1.2	101.1	107.7
47	Bromobenzene	12.328	156.0	0.99912	0.5	1	98.2	90.9
48	1,2,3-Trichloropropane	12.346	75.0	0.99986	0.4	1.2	98.8	102.6
49	n-Propylbenzene	12.452	91.0	0.99931	0.7	1.3	93.9	90.1
50	2-Chlorotoluene	12.552	91.0	0.99984	0.5	1.1	94.6	89.9
51	1,3,5-Trimethylbenzene	12.652	105.0	0.99959	0.6	1.2	94.4	90.0
52	4-Chlorotoluene	12.676	91.1	0.99973	0.3	0.8	93.7	89.4
53	tert-Butylbenzene	13.028	119.0	0.99994	0.8	1.4	94.3	89.3
54	1,2,4-Trimethylbenzene	13.081	105.0	0.99967	0.5	1.1	94.5	89.2
55	sec-Butylbenzene	13.275	105.0	0.99891	0.6	1.4	92.7	87.3
56	1,3-Dichlorobenzene	13.399	146.0	0.99996	0.4	1.2	94.5	85.8
57	p-Isopropyltoluene	13.434	119.0	0.99901	0.4	1.2	92.4	86.4
58	1,4-Dichlorobenzene	13.499	146.0	0.99998	0.2	0.9	94.4	85.6
59	n-Butylbenzene	13.887	91.0	0.99922	0.4	1	90.5	86.0
60	1,2-Dichlorobenzene	13.911	146.0	1.00000	0.2	0.8	95.5	87.3
61	1,2-Dibromo-3-chloropropane	14.740	157.0	0.99577	0.6	3.4	105.9	106.9
62	1,2,4-Trichlorobenzene	15.634	180.0	0.99871	0.7	1.6	93.5	76.7
63	Hexachlorobutadiene	15.822	260.0	0.99789	1.0	2.3	85.7	71.0
64	Naphthalene	15.893	128.0	0.99991	1.1	2	100.2	91.9
65	1,2,3-Trichlorobenzene	16.146	182.0	0.99870	0.6	1.1	92.8	74.4

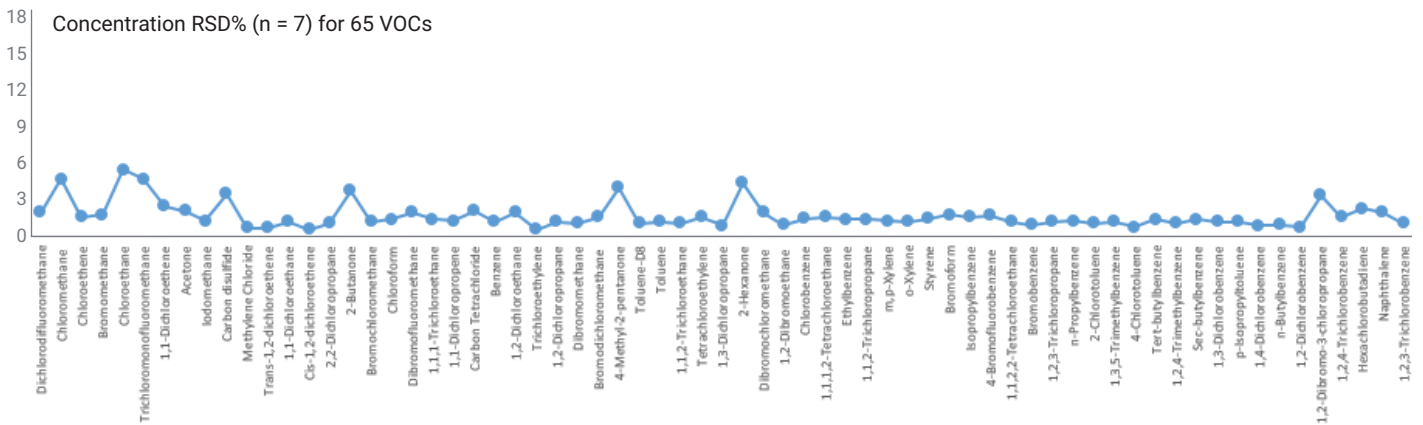


Figure 3. The concentration of RSD% for 65 VOCs at 20 µg/L.

The calibration curve was developed based on an ISTD method for six concentration levels from 5 µg/L to 200 µg/L. The correlation coefficient R² values for 65 VOCs are shown in Table 3. Excellent linearity was demonstrated for all compounds with an R² range of 0.99446 to 1. The method detection limit (MDL) for 65 VOCs was calculated from the standard deviation of seven replicate runs of the lowest calibration standard at 5 µg/L, then translated into MDL (µg/kg) followed by the HJ 605-2011 method requirement. Note that all MDLs are

below 1.1 µg/kg, except four compounds (chloromethane, chloroethane, 4-methyl-2-pentanone, and naphthalene), which have MDLs below 3.6 µg/kg .

The method recovery was measured by spiking 25 µL of 10 mg/L calibration standard into 5 g real soil samples (corresponding to 50 µg/kg VOCs in the soil sample). For this work, two soil samples (samples 1 and 2) were obtained from the customer's lab. Four runs of each sample's duplicates with spiking were tested and the average recovery rates were calculated, shown

in Figure 4 and Table 3. As Table 3 illustrates, the recovery rate for sample 1 was between 85.3 and 113.6%, and for sample 2 was between 71.0 and 119.4%. Considering the different matrix in samples 1 and 2, the difference in recovery rate is acceptable. Sample 2 has a more complicated matrix, and the recovery results of late effluent components in this sample were relatively low because those compounds have a high boiling point and are hard to purge from real samples.

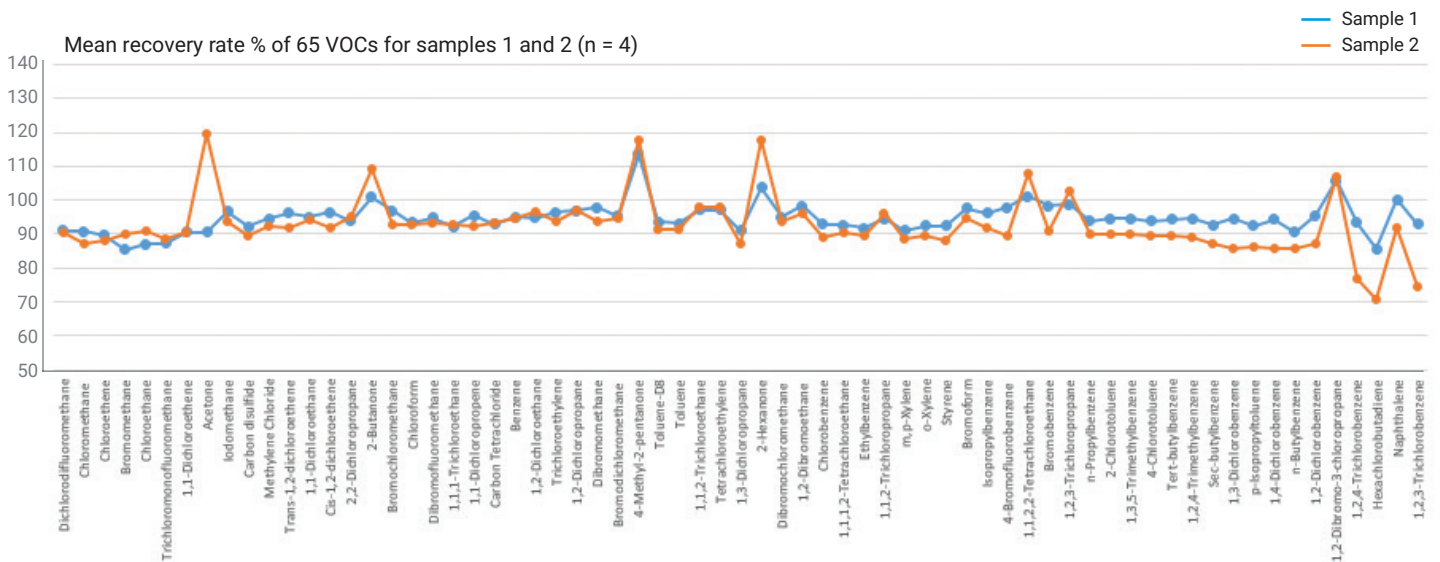


Figure 4. The mean recovery rates for 65 VOCs for sample 1 and sample 2.

Petroleum hydrocarbons (C₆ to C₉) analysis

The HJ 1020-2019 method specifies organic compounds with a retention time between 2-methylpentane (included) and *n*-decane (not included) on the FID signal as our target. It identified these peaks as the overall integral as C₆ to C₉. Before proceeding with the calibration and analysis, a qualitative test was performed

by injecting the single standard solutions of 2-methylpentane (C₆H₁₄) and *n*-decane (C₁₀H₂₂) to identify the retention time range for C₆ to C₉. The integration starts at the beginning of 2-methylpentane and ends before the beginning of *n*-decane. The *Area Sum ON/OFF* function in the MassHunter software was used for integration.

Figure 5 shows an overlay of three chromatograms (2-methylpentane single standard, *n*-decane single standard and C₆ to C₉ standard mixture) obtained from FID signals using the same oven program as the HJ 605 method. The retention time range of C₆ to C₉ could easily be identified by comparing the three chromatograms.

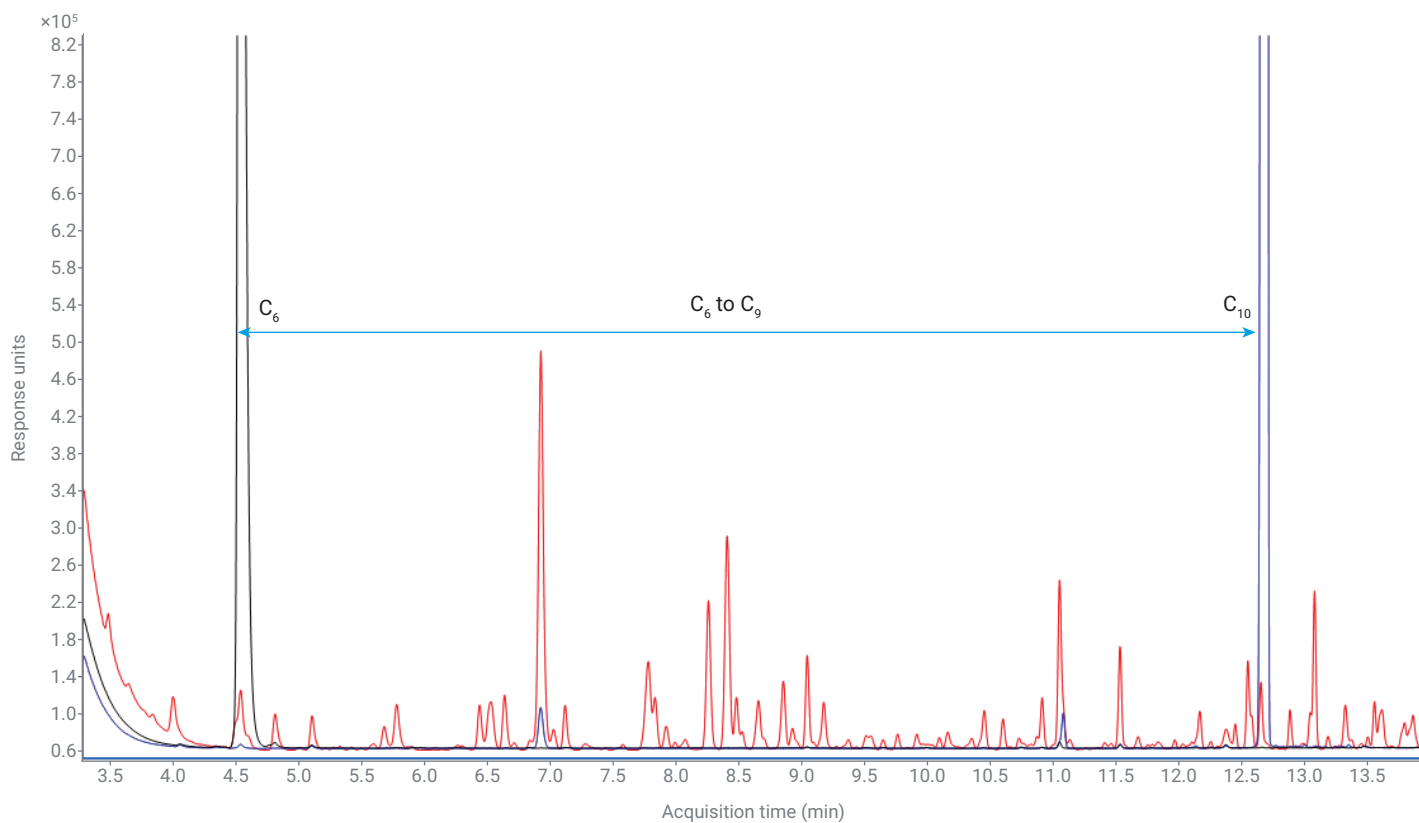


Figure 5. An overlay of chromatograms obtained from the system using the HJ 605 oven program. The black line represents a single standard solution of 2-methylpentane (C₆); the blue line represents a single standard solution of *n*-decane (C₁₀); and the red line represents a calibration solution of petroleum hydrocarbons. The peaks between C₆ (included) and C₁₀ (not included) were named C₆ to C₉.

Figure 6 shows the calibration performance. The correlation coefficient of 0.9997 indicates a good linear fit across the full calibration range. A repeatability test was done by measuring the detector response of seven sequential injections at the concentration of 0.1 µg/mL and 1 µg/mL. Table 4 and Figure 7 show the results. The measured area relative standard deviation (RSD) of 3.7% and 0.8% were far below the required maximum of 25% RSD described in the HJ 1020 method. The MDL for petroleum hydrocarbons was also calculated from the standard deviation of seven replicate runs of the lowest calibration standard at 0.1 µg/mL. To fit with the HJ 1020-2019 method requirements, 5 g of sample was used for MDL calculation, and the MDL corresponded to 0.02 mg/kg. The method recovery was measured by spiking 10 µL of 500 mg/L calibration standard into 5 g of soil samples (corresponding to 1 mg/kg petroleum

hydrocarbons in the soil). Samples 1 and 2 were used for the recovery tests. Three runs of each sample's duplicates with spiking were tested and the average recovery rates were calculated. Table 4 shows that the recovery rate

for sample 1 was 91.8%, and 89.4% for sample 2. The better recovery rate for sample 1 also shows that the matrix of sample 2 is more complicated than sample 1.

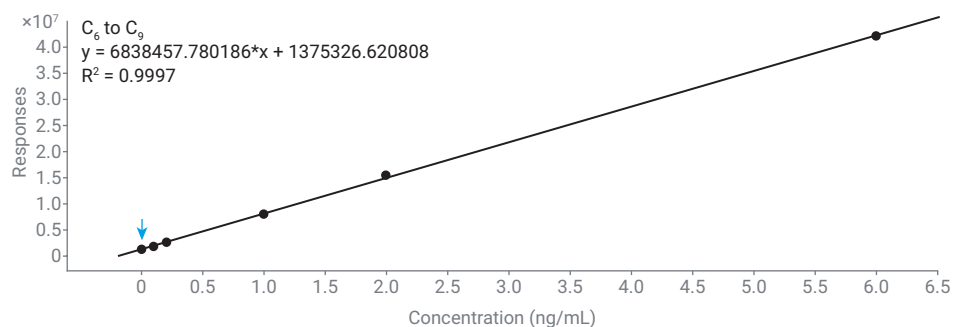


Figure 6. A six-level linear calibration of petroleum hydrocarbons from 0 to 6 µg/mL.

Table 4. Analysis results for petroleum hydrocarbons.

Name	CF R ²	RSD (n = 7)		MDL (mg/kg)	Mean Recovery % (n = 3)	
		0.1 µg/mL	1 µg/mL		sample 1	sample 2
C ₆ to C ₉	0.9997	3.7	0.8	0.02	91.8	89.4

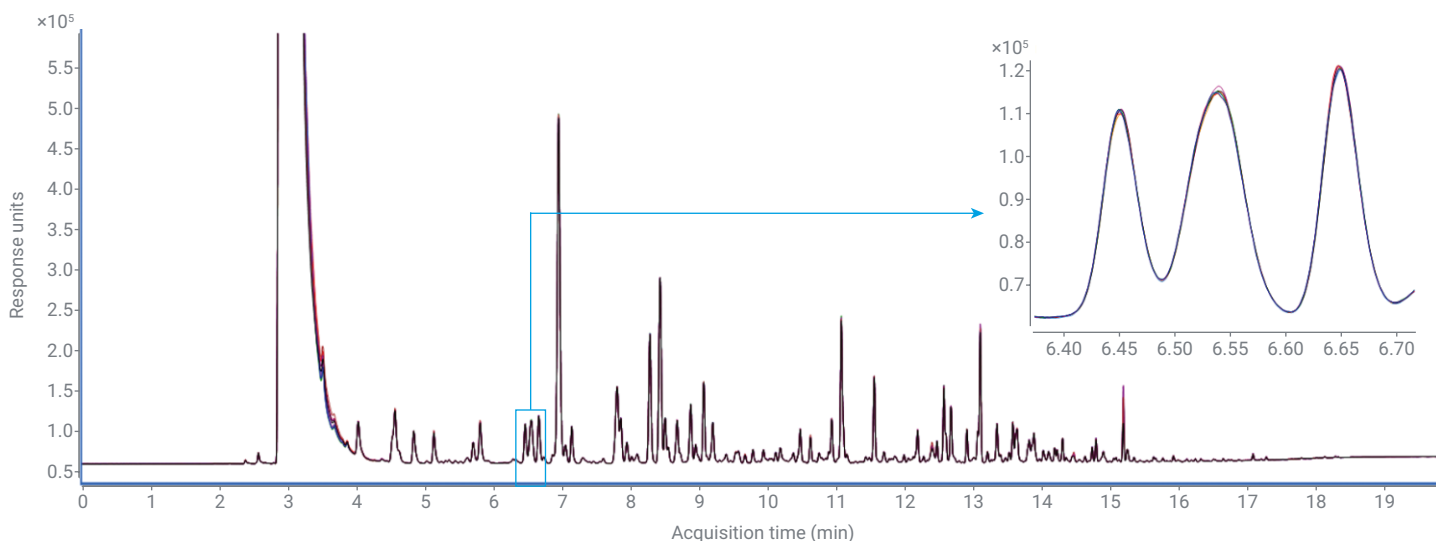


Figure 7. An overlay of seven chromatograms obtained from duplicate analysis at a concentration of 1 µg/mL.

Real sample analysis

Using this unified GC configuration to analyze VOCs gives accurate quantitative results because it is based on specific quantification ions selected for each VOC compound on the MSD channel. But the petroleum hydrocarbon analysis is quantitated by FID signal. If there are VOCs in the soil samples and they elute within the C_6 to C_9 elution window, their response on the FID will be counted in the integrated area of C_6 to C_9 group peak. It will therefore interfere with the quantitation of targeted petroleum hydrocarbons. As a result, the analysis of real samples of petroleum hydrocarbons

could be divided into two situations. The first situation is samples obtained from suspected petroleum hydrocarbon contamination sites, containing only the saturated hydrocarbons. The second is samples with unknown contaminants that may contain both petroleum hydrocarbon and VOCs.

The HJ 605 oven program can be applied to the C_6 to C_9 analysis of the first type of soil samples. Special attention should be given to unknown soil samples that may be subject to VOC interference. Figure 8 shows some VOCs in the HJ 605 compound list that eluted within the C_6 to C_9 RT window. According to

the definition of the HJ 1020 method, the peaks between C_6 (included) and C_{10} (not included) were named C_6 to C_9 . If using the HJ 605 oven program, the HJ 605 VOCs from methylene chloride to 2-chlorotoluene will therefore be identified as C_6 to C_9 , together with other petroleum hydrocarbons. When using the oven program recommended by the HJ 1020 method, the retention ability of compounds with different polarities was different on an Agilent DB-624 Ultra Inert column, and 4-chlorotoluene and 1,3,5-trimethylbenzene eluted earlier than C_{10} .

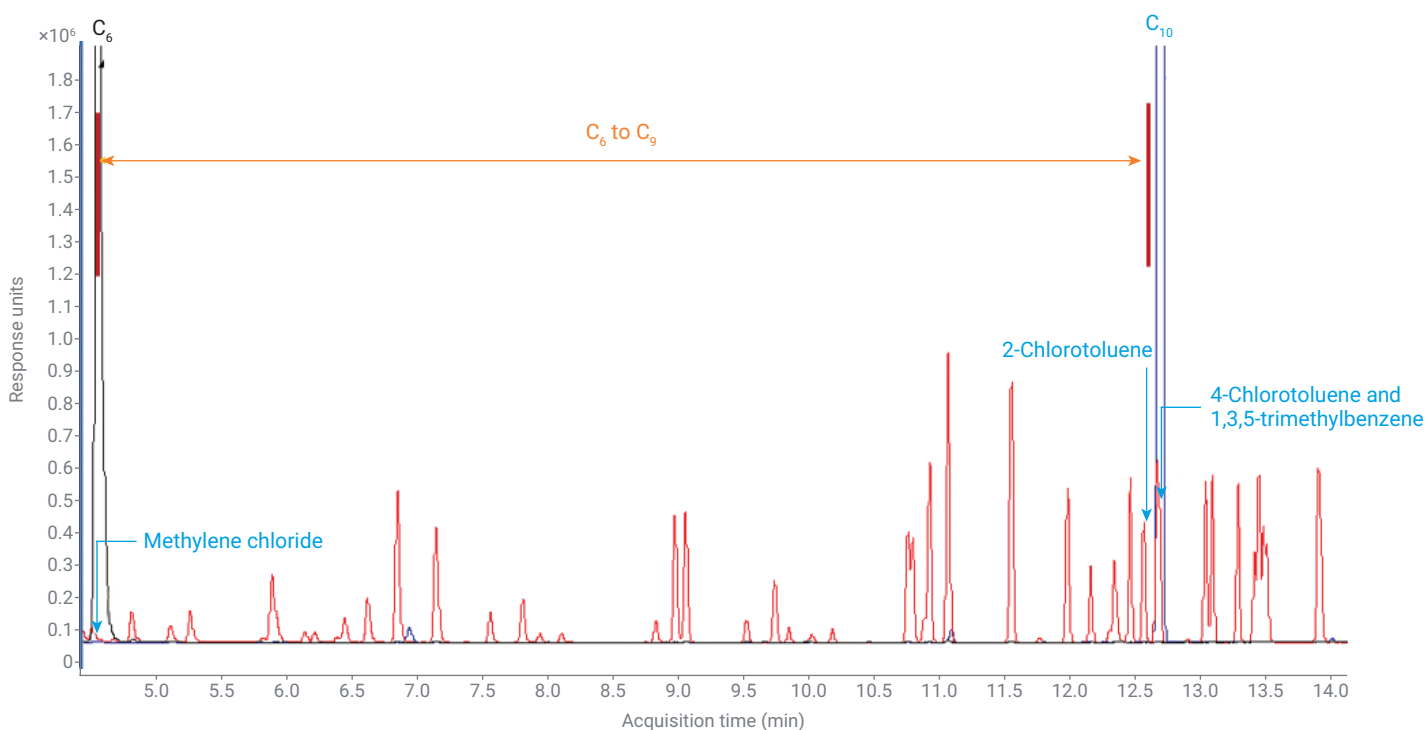


Figure 8. An overlay of FID chromatograms obtained from the system. Black line represents a single standard solution of 2-methylpentane (C_6); the blue line represents a single standard solution of *n*-decane (C_{10}); the red line represents a calibration solution of 65 VOCs. The peaks between C_6 (included) and C_{10} (not included) were named C_6 to C_9 using the HJ 605 oven program.

Figure 9 shows that compounds targeted in the HJ 605 method from methylene chloride to 1,3,5-trimethylbenzene eluted within the C₆ to C₉ RT window using HJ 1020 oven program. Compared to the separation obtained under the HJ 605 oven temperature, two more compounds, 4-chlorotoluene and 1,3,5-trimethylbenzene, were identified as

C₆ to C₉ hydrocarbons using the HJ 1020 oven temperature. We would therefore suggest using the HJ 605 oven program for petroleum hydrocarbon analysis when there is no coexistence of VOCs or VOCs with relatively high boiling points, such as 1,3,5-trimethylbenzene and 4-chlorotoluene. For reliable quantitative results, the HJ 1020 oven program can

be used during petroleum hydrocarbon analysis with real samples containing VOCs with relatively high boiling points. An additional benefit with this unified system is the ability to use the MSD signal to report a separate BTEX result. The MSD signal can also help confirm if there is VOC interference in the petroleum hydrocarbon analysis result.

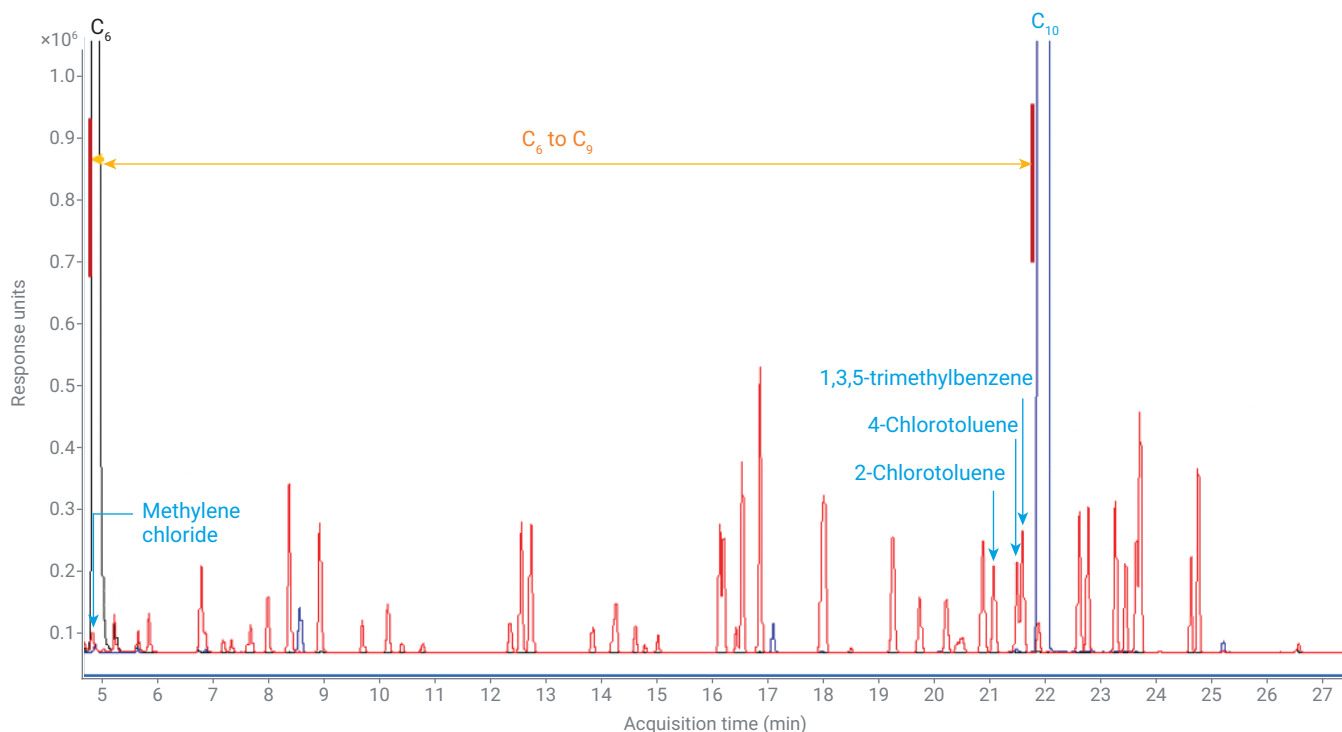


Figure 9. An overlay of FID chromatograms obtained from the system. The black line represents a single standard solution of 2-methylpentane (C₆); blue line represents a single standard solution of *n*-decane (C₁₀); red line represents a calibration solution of VOCs. The peaks between C₆ (included) and C₁₀ (not included) were named C₆ to C₉ using HJ 1020 oven program

Sample 3 was run to evaluate the method under the same experimental conditions. Sample 3 was divided into two parts. One part with the internal standards added was for VOC analysis, the other part, without ISTD, was used for petroleum hydrocarbons analysis. Stir bars in the vials are recommended to ensure thorough mixing and consistent

results for the real samples. Figure 10 shows an overlay of the MSD signal (black trace) and FID signal (red trace) produced from the analysis of sample 3. The two signals could be quantitated according to the calibration curves of VOCs and petroleum hydrocarbons, respectively. All VOC concentrations were calculated and found to be well

below the linear minimum (5 µg/L), except for acetone, 2-hexanone, and 1,2,4-trichlorobenzene. Table 5 shows the calculated VOC results of sample 3. According to the calibration curve of petroleum hydrocarbons (C₆ to C₉), the concentration of C₆ to C₉ was well below 0.1 µg/mL.

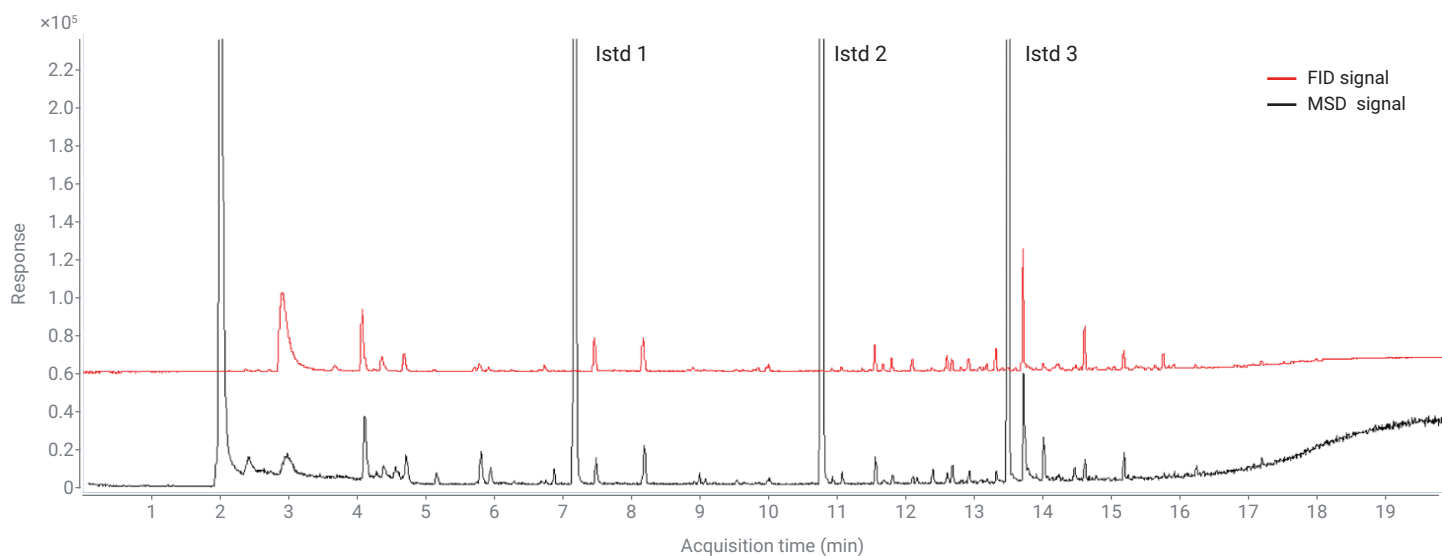


Figure 10. GC/MS-SCAN and FID chromatogram of real sample (sample 3) using the HJ 605 oven program.

Table 5. The results of sample 3.

Compound Name	Calculated Concentration (µg/L)
Acetone	33
2-Hexanone	8
1,2,4-Trichlorobenzene	5

High-throughput real sample analysis

The analyses of VOCs and petroleum hydrocarbons (C_6 to C_9) in real samples were performed by injecting samples separately and then substituting MSD and FID signals into different calibration curves to obtain quantitative results. This configuration can acquire MSD and FID signals simultaneously by sampling real samples in one injection. Figure 11 shows simultaneous acquisition of MSD and FID signals for samples containing both petroleum hydrocarbons and VOCs with relatively low boiling points, using the HJ 605 oven program. Since VOCs are quantified using internal standard methods, three internal standards should be added into the sample. The MSD signal was used for VOC analysis, while the FID signal was used for petroleum hydrocarbons. For the FID signal, the peaks of the internal standards should be subtracted when performing quantitative calculation.

Although the calibration curves of VOCs and petroleum hydrocarbons need to be established by sampling different standard solutions separately, for the real soil sample, one sample injection can work for the analysis of two types of compounds. The data can then be processed separately.

This high-throughput approach is suitable for two situations:

- Samples containing both petroleum hydrocarbons and VOCs with relatively low boiling points (compounds eluted before 4-chlorotoluene on a DB-624 Ultra Inert column), using the HJ 605 oven program (total run time: 20 minutes).
- Samples containing both petroleum hydrocarbons and VOCs using the HJ 1020 oven program (total run time: 35 minutes).

As mentioned in the HJ methods, the total run time for VOCs is 20 minutes and for petroleum hydrocarbons it is 35 minutes. It therefore takes approximately 60 minutes for two separate VOC and petroleum hydrocarbons analyses. As demonstrated in this application note, it takes 20 or 35 minutes to run the high-throughput sample analysis. This new process greatly reduces pretreatment time and improves laboratory productivity. This high-throughput sample analysis is particularly attractive to laboratories trying to maximize sample throughput and minimize turn-around time.

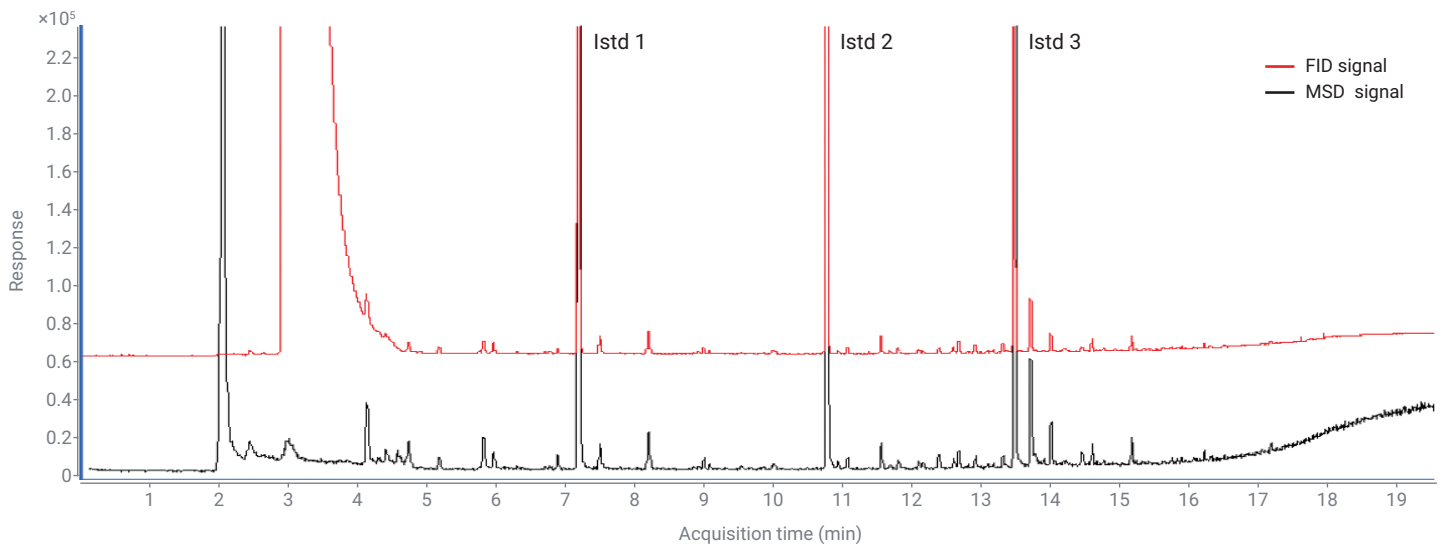


Figure 11. GC/MS-SCAN and FID chromatogram of real sample (sample 3) obtained on the system simultaneously using the HJ 605 oven program.

Conclusion

This application note demonstrates a unified P&T/8890 GC/FID 5977B MSD configuration that provides the flexibility to run either VOCs or petroleum hydrocarbons (C₆ to C₉) on a single instrument. If the HJ standard requirements need to be strictly obeyed, VOCs and petroleum hydrocarbons can be tested separately on the same system, using the method parameters recommended by the HJ 605 and HJ 1020 methods. Fast analysis is also mentioned in this study, and, for samples containing both petroleum hydrocarbons and VOCs with relatively low boiling points, one injection with the HJ 605 oven program can be used for the analysis of the two kinds of compounds simultaneously. The results obtained from this versatile system meet all performance criteria outlined by HJ 605-2011 and HJ 1020-2019 methods. The system is particularly attractive to laboratories seeking low costs and high flexibility.

References

1. Soil and Sediment-Determination of Volatile Organic Compounds-Purge and Trap Gas Chromatography/Mass Spectrometry Method. HJ 605-2011.
2. Soil and Sediment-Determination of Petroleum Hydrocarbons (C₆-C₉)-Purge and Trap Gas Chromatography. HJ 1020-2019.

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