

Technical Report

Analysis of Volatile Organic Compounds in the Environment Using the Restore Function of TD-GC/MS

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Abstract:

Measuring the concentration of volatile organic compounds (VOCs) in the air serves as a means of determining air pollution and is used to monitor pollution in a wide variety of environments, such as within manufacturing plants, urban areas, and indoor environments. VOC measurements are commonly performed using a thermal desorption-gas chromatograph mass spectrometer (TD-GC/MS). To reduce the risk of analysis failures due to the time-consuming process of sampling the atmosphere, multiple tubes are typically used for backup sampling or samples are re-analyzed using a TD restore function. However, previous TD restore functionality did not control the sample line temperature adequately, which made it difficult to simultaneously restore compounds with a wide range of low and high boiling points. To solve such problems with TD functionality, the authors developed a TD system (TD-30R) that can adjust the sample line temperature as appropriate for each analytical process step. This report describes the results from studying the appropriate sample line temperature for each section (trap tube cooling/desorption temperatures, sample tube desorption temperature and flowrate, and joint, valve, and transfer line temperatures) in terms of optimizing analytical conditions for simultaneously measuring VOCs with a wide range of low and high boiling points using TD restore functionality. The report also describes the results from using the optimized conditions to simultaneously analyze, with high sensitivity and a high restore rate, a wide range of low and high boiling-point VOCs in the air inside an actual indoor environment.

Keywords: GC/MS, TD, restore, VOCs, TO-17, and air pollution

1. Introduction

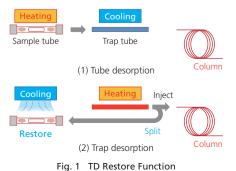
Measuring the concentration of volatile organic compounds (VOCs) in the air serves as a means of determining air pollution and is used to monitor pollution in a wide variety of environments. Method TO-17, specified by the United States Environmental Protection Agency (U.S. EPA), is known as the most common method for analyzing VOCs. Method TO-17 actively samples the air (adsorbs VOC components from the air by pumping the air through an adsorption tube) and then analyzes the VOCs in the tube using a thermal desorption-gas chromatograph mass spectrometer (TD-GC/MS). However, because the method requires a long time for sampling the atmosphere, multiple tubes are typically used to collect back samples or TD restore functionality is used to reduce the risk of failing to analyze the VOCs.

The TD restore function thermally desorbs the sample gases trapped inside the sample tube and then restores the split sample gases in the tube before injection into the GC/MS system. Specifically, as shown in Fig. 1, volatile components thermally desorbed from the sample tube are trapped in a trap tube (Fig. 1 (1)). Then the trap tube is heated to desorb the components and inject them into the column. The components in the sample gases that were split off before injection into the column are then trapped again in the sample tube (Fig. 1 (2)). Consequently, the restore function ensures the sample gas can be easily re-measured even if a problem occurs during analysis.

However, it is difficult to simultaneously restore compounds with a wide range of low and high boiling points with the TD restore function because it is difficult to restore low boiling-point compounds while the sample tube is still hot immediately after desorption during sample gas restoring. Therefore, the sample tube will typically be replaced before

split sample gases enter the sample tube, but this increases the risk of gas leakage. If leakage occurs, it can cause both the analysis and restore process to fail. Using the original sample tube for restore provides an effective way to avoid such failures. That means that the sample tube must be cooled quickly before restoring low boiling-point compounds and also that the sample line temperature setting must be as low as possible. On the other hand, if the sample line temperature is too low, it could increase the likelihood of carryover for high boiling-point compounds.

To address these concerns, the TD-30R moves the sample tube away from the heat source while blow-cooling the sample during restoring, enabling faster cooling than previously possible. In addition, taking into account the TD-30R sample line conditions (cooling and desorption temperature of the trap tube, desorption temperature and flow rate in the sample tube, temperature of the joint valve transfer line), we investigated the possibility of analyzing compounds with a wide range of boiling points with high sensitivity and a high restore rate, and report on the results here.



2. Method

2-1. Measurement Sample Gas Preparation

Components from one to four liters of air from the atmosphere (the sample gas) were trapped by an adsorbent in a sample tube. Carboxen® was used as the adsorbent. It was analyzed within 30 days (stored at 4 °C max.) of trapping the sample gas components. Toluene-d8 was added as an internal standard substance before analysis. Sample gas components trapped in the trap tube were dry-purged with carrier gas before injection into the GC/MS system.

2-2. Analytical Conditions

A GCMS-QP2020 single quadrupole mass spectrometer was used as the GC/MS system. The thermal desorption (TD) method with a TD-30R pretreatment unit was used for sample gas injection. Detailed analytical conditions are indicated in Table 1. Only the final optimized TD-30R analytical conditions are indicated.

Table 1	GC-MS Analytical Conditions	
Tuble I	de mis / mary fieur contantions	

System Configuration		GC			
GC/MS	: GCMS-QP™2020	Carrier Gas	: Helium		
Pretreatment Unit	: TD-30R	Control Mode	: Pressure		
Software	: GCMSsolution™ Ver.4.31	Pressure	: 69 kPa		
	TD-30 Control Software	Injection Mode	: Split 1: 10		
Column	: SH-Rtx [™] -624 (60 m x 0.32 mm l.D., df = 1.8 µm, P/N: 221-75864-60) (SHIMADZU)	Column Oven Temperature	(1.69 mL/min column flowrate) : 35 °C (5 min) − (5 °C/min) – 230 °C (5 min)		
TD-30R		MS			
Tube Desorption Temper	ature : 250 °C (10 min)	Ion Source Temperature	: 230 °C		
Tube Desorption Flowrat	e : 70 mL/min	Interface Temperature	: 200 °C		
Trap Cooling Temperatur	re : -25 °C	Measurement Mode	: Scan/SIM Simultaneous Measuremen		
Trap Desorption Tempera	ature : 250 °C (2 min)	Scan Mass Range	: <i>m/z</i> 20-600		
Joint Temperature	: 75 °C	Scan Event Time	: 0.2 sec.		
Valve Temperature	: 185 °C	Scan Speed	: 3333 u/sec.		
Transfer Line Temperatur	re : 220 °C	SIM Monitor Ion	: Compliant with TO-17 regulation		

3. Analysis Results

3-1. Study of Trap Tube Cooling Temperature

Due to the higher volatility of low boiling-point compounds, they are more likely than high boiling-point compounds to remain untrapped if the trap tube is not sufficiently cooled, resulting in poor analytical sensitivity. Therefore, the relationship between the trap tube cooling temperature and the sensitivity for analyzing low boiling-point compounds was considered at three trap tube cooling temperatures: 0, -20, and

-25 °C. Chromatograms for the compounds with particularly low boiling points detected in the given sample ((a) chloroethene and (b) ethyl chloride) are shown as examples in Fig. 2. (a) chloroethene has a very low boiling point of -13 °C, but it can be detected with high sensitivity by decreasing the trap tube cooling temperature to -25 °C.

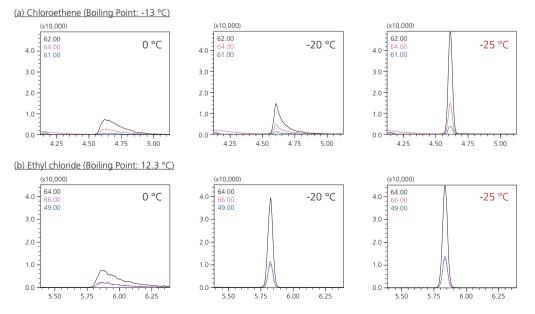


Fig. 2 Sensitivity Comparison for Low Boiling-Point Compounds Using Different Trap Tube Cooling Temperatures

3-2. Study of Trap Tube Desorption Temperature

Considering cost and ease of maintenance, it is preferable to set the trap tube desorption temperature as low as possible. However, if the desorption temperature is too low, desorption of high boiling-point compounds will be more difficult, which can prevent their detection. Therefore, the relationship between the trap tube desorption temperature and the sensitivity for analyzing high boiling-point compounds was considered at two trap tube temperatures, 250 °C and

3-3. Study of Sample Tube Desorption Temperature

For thermal desorption, the sample tube and adsorbent can be reused as many times as desired by reconditioning the sample tube. However, if the sample tube thermal desorption process is insufficient, sample gas components can carry over from the previous measurement and affect the next measurement results. Consequently, we considered the sample tube desorption temperature and flow-rate. Chromatograms for two compounds ((a) 1,2,4-trichlorobenzene and (b) hexachloro-1,3-butadiene) that carried over from among all the compounds detected in the given sample gas are shown in Fig. 4. The following two desorption temperature and flowrate conditions

325 °C. Chromatograms for the compounds with particularly high boiling points detected in the given sample ((a) 1,2,4-trichlorobenzene and (b) o-dichlorobenzene) are shown in Fig. 3. Compound (a) 1,2,4-trichlorobenzene has a 213 °C boiling point, but can be detected with high sensitivity using a trap tube desorption temperature of 250 °C.

were considered. Before optimization, the sample tube desorption temperature was 250 °C (5 min) and the tube desorption flowrate was 30 mL/min. After optimization, the sample tube desorption temperature was 250 °C (10 min) and the tube desorption flowrate was 70 mL/min. Given the conditions before optimization, the carryover value for (a) 1,2,4-trichlorobenzene was 5.4 %. In contrast, by specifying a longer thermal desorption time and a higher flowrate during desorption, as specified in the conditions after optimization, sufficient sample gas desorption occurred and the carryover value decreased to 0.7 %.

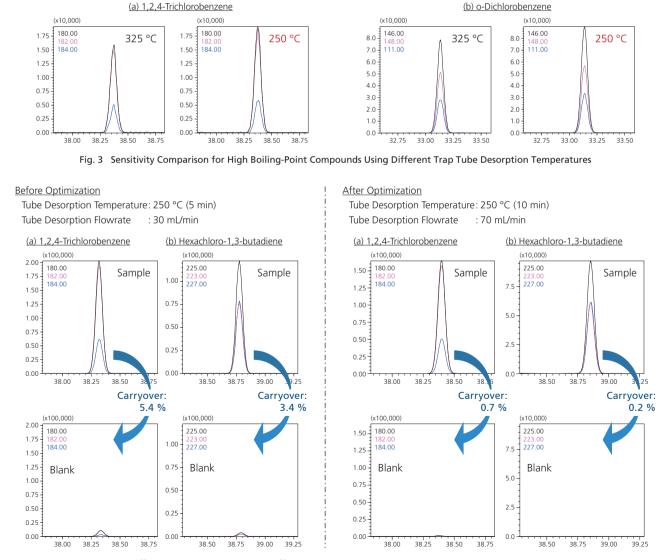
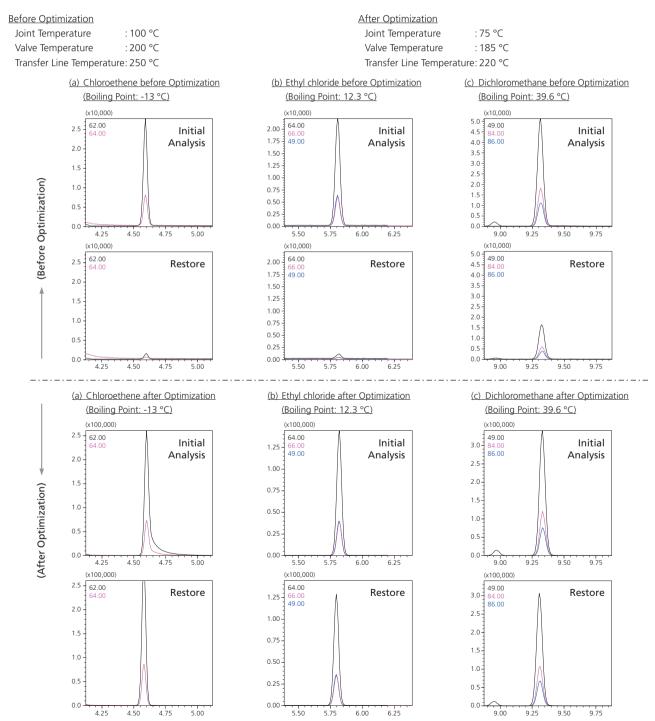


Fig. 4 Differences in Carryover Due to Different Desorption Temperatures and Flowrates in the Sample Tube

3-4. Study of Joint Temperature and Other Conditions

A low joint, valve, or transfer line temperature can cause cold spots in flow lines, whereas a high temperature can make it difficult to restore low boiling-point compounds in the sample tube. Therefore, it is important to specify joint, valve, and transfer line temperatures appropriately. The following presents an example of optimizing conditions that would enable restoring low boiling-point compounds. Before optimization, the joint temperature was 100 °C, the valve temperature was 200 °C, and the transfer line temperature was 250 °C. After optimization, the joint temperature was 75 °C, the valve temperature was 185 °C, and the transfer line temperature was 220 °C. As a

comparison of restore rates for low boiling-point compounds before and after optimization, chromatograms for (a) chloroethene (boiling point: -13 °C), (b) ethyl chloride (boiling point: 12.3 °C), and (c) dichloromethane (boiling point: 39.6 °C) are shown in Fig. 5. Almost no low boiling-point components were detected during restoring before optimization, but lowering the temperature conditions for joints and other areas resulted in trapping the components in the sample tube and detecting them with adequate sensitivity after optimization.



3-5. Reproducibility and Restore Rate of Restore Function

Optimal analytical conditions were determined from the studies described above. Then the reproducibility and restore rates for the restore function were determined for those conditions. First, a standard sample gas (mixture containing 1 µg each of 40 substances analyzed by the TO-17 method) was added to six sample tubes to determine whether or not adequate measurement reproducibility was achieved based on initial analysis and restore results. Those measurement results are shown in Table 2. Mean values from measuring the six sample tubes were used as the standard sample quantitation values. Quantitation values were calculated from the calibration curve prepared in accordance with TO-17 based on the peak area ratio for each concentration obtained from

adding 1 to 50 µg of the standard sample. Table 3 shows the linearity of the calibration curve. Table 2 shows that the relative standard deviation (%RSD) is within 10 % for all substances and the %RSD value was about the same for both the initial analysis and restoring for most substances, which indicates good analytical reproducibility even when using the restore function. The difference between the %RSD value for the initial analysis and restore was large only for ethyl chloride (I.D. No. 5 in Table 2). That large difference was presumably caused by the larger difference in recovery rates between ethyl chloride and toluene-d8, used as an internal standard substance during restoring, than for other substances.

Table 2	Reproducibility for	1 µg Star	ndard Sample (Gas
	Reproducionity for	i pg stai	iduru sumpre v	Ju.

Table 3 Calibration Curve Linearity (1 to 50 µg Standard Sample Gas Added)

Linearity

Restore

0.9993

0.9997

0.9987

0.9957

		Mean Calibration	Curve Value	%RSD (r	1=6)				
D.	Component	Initial Analysis Restore		Initial Analysis	Restore	I.D.	Component	Initial Anal	
1	Freon 114	1.04	1.03	3.55	4.04	1	Freon 114	0.9991	
2	Chloroethene	1.01	1.00	4.81	4.57	2	Chloroethene	0.9999	
3	1,3-Butadiene	1.03	1.03	4.90	3.95	3	1,3-Butadiene	0.9995	
4	Methyl bromide	0.94	0.99	3.56	4.88	4	Methyl bromide	0.9962	
5	Ethyl chloride	1.04	1.09	3.69	9.18	5	Ethyl chloride	0.9986	
6	Freon 11	1.03	1.04	3.41	3.80	6	Freon 11	0.9994	
7	1,1-Dichloroethene	1.03	1.03	3.39	3.71	7	1,1-Dichloroethene	0.9991	
3	Freon 113	1.02	1.03	3.03	3.75	8	Freon 113	0.9992	
9	3-Chloro-1-propene	0.98	0.99	4.34	3.94	9	3-Chloro-1-propene	0.9999	
0	Methylene chloride	1.04	1.02	3.32	3.84	10	Methylene chloride	0.9990	
1	Acrylonitrile	0.96	0.93	5.03	5.10	11	Acrylonitrile	0.9991	
2	1,1-Dichloroethane	1.03	1.02	3.10	3.77	12	1,1-Dichloroethane	0.9995	
3	cis-Di-1,2-Dichloroethylene	1.02	1.01	2.88	3.78	13	cis-Di-1,2-Dichloroethylene	0.9995	
4	Chloroform	1.04	1.07	4.64	6.45	14	Chloroform	0.9997	
5	1, 1, 1-Trichloroethane	1.03	1.04	2.85	3.50	15	1,1,1-Trichloroethane	0.999	
6	Carbon tetrachloride	1.03	1.03	3.00	4.16	16	Carbon tetrachloride	0.999	
7	Benzene	1.05	1.04	2.32	2.68	17	Benzene	0.9999	
8	1,2-Dichloroethane	1.02	1.01	2.76	2.37	18	1,2-Dichloroethane	0.999	
9	Trichloroethylene	0.86	0.75	3.45	6.26	19	Trichloroethylene	0.9998	
0	1,2-Dichloropropane	1.02	1.02	2.09	2.39	20	1,2-Dichloropropane	0.9996	
1	1,3-Dichloropropene	0.95	0.90	9.60	9.87	21	1,3-Dichloropropene	0.9999	
2	Toluene	0.96	0.89	1.80	2.80	22	Toluene	0.9999	
3	1,1,2-Trichloroethane	1.02	1.02	1.57	1.15	23	1,1,2-Trichloroethane	0.9999	
4	Tetrachloroethylene	0.90	0.84	2.19	2.96	24	Tetrachloroethylene	0.999	
5	1,2-Dibromoethane	1.01	1.01	1.66	0.54	25	1,2-Dibromoethane	0.9998	
6	Chlorobenzene	1.01	0.99	1.73	2.09	26	Chlorobenzene	1.0000	
7	Ethylbenzene	1.03	1.03	1.48	2.20	27	Ethylbenzene	0.9999	
8	m-Xylene	1.01	1.02	1.20	1.86	28	m-Xylene	0.9998	
9	p-Xylene	1.01	1.02	1.20	1.86	29	p-Xylene	0.9998	
0	o-Xylene	1.02	1.02	1.34	1.60	30	o-Xylene	0.9998	
1	Styrene	1.01	1.01	1.04	0.51	31	Styrene	0.9998	
2	1,1,2,2-Tetrachloroethane	0.95	0.92	1.71	1.90	32	1,1,2,2-Tetrachloroethane	0.9994	
3	1,3,5-Trimethylbenzene	1.01	1.01	0.80	1.20	33	1,3,5-Trimethylbenzene	0.999	
4	1,2,4-Trimethylbenzene	1.02	1.03	1.02	1.37	34	1,2,4-Trimethylbenzene	0.9994	
5	4-Ethyltoluene	0.97	1.03	3.41	3.74	35	4-Ethyltoluene	0.9995	
6	m-Dichlorobenzene	1.00	1.01	1.50	0.51	36	m-Dichlorobenzene	0.9995	
7	p-Dichlorobenzene	0.99	1.01	1.52	0.63	37	p-Dichlorobenzene	0.9995	
8	o-Dichlorobenzene	1.00	1.01	2.07	0.40	38	o-Dichlorobenzene	0.9992	
9	1,2,4-Trichlorobenzene	0.85	0.82	7.55	6.30	39	1,2,4-Trichlorobenzene	0.9993	
10	Hexachloro-1,3-butadiene	1.03	1.00	4.97	4.17	40	Hexachloro-1,3-butadiene	0.9983	

We dry bronnae			
Ethyl chloride	0.9986	0.9996	
Freon 11	0.9994	0.9995	
1,1-Dichloroethene	0.9991	0.9991	
Freon 113	0.9992	0.9993	
3-Chloro-1-propene	0.9999	1.0000	
Methylene chloride	0.9990	0.9995	
Acrylonitrile	0.9991	0.9981	
1,1-Dichloroethane	0.9995	0.9995	
cis-Di-1,2-Dichloroethylene	0.9995	0.9995	
Chloroform	0.9997	0.9999	
1,1,1-Trichloroethane	0.9997	0.9998	
Carbon tetrachloride	0.9997	0.9998	
Benzene	0.9999	0.9999	
1,2-Dichloroethane	0.9995	0.9995	
Trichloroethylene	0.9998	0.9999	
1,2-Dichloropropane	0.9996	0.9996	
1,3-Dichloropropene	0.9999	0.9997	
Toluene	0.9999	0.9998	
1,1,2-Trichloroethane	0.9999	0.9999	
Tetrachloroethylene	0.9995	0.9996	
1,2-Dibromoethane	0.9998	0.9999	
Chlorobenzene	1.0000	0.9999	
Ethylbenzene	0.9999	0.9999	
m-Xylene	0.9998	0.9998	
p-Xylene	0.9998	0.9998	
o-Xylene	0.9998	0.9997	
Styrene	0.9998	0.9997	
1,1,2,2-Tetrachloroethane	0.9994	0.9993	
1,3,5-Trimethylbenzene	0.9995	0.9994	
1,2,4-Trimethylbenzene	0.9994	0.9992	
4-Ethyltoluene	0.9995	0.9992	
m-Dichlorobenzene	0.9995	0.9993	
p-Dichlorobenzene	0.9995	0.9993	
o-Dichlorobenzene	0.9992	0.9990	
1,2,4-Trichlorobenzene	0.9993	0.9988	
Hexachloro-1,3-butadiene	0.9983	0.9983	

Quantitation results for an actual sample gas are shown in Table 4. For the actual sample gas, air was sampled from two different locations within a room. Results marked "N.D." in the table indicate that none of the substance was detected in the actual sample gas using the given analytical equipment, whereas "<LOQ" indicates that the measured value was less than the lower limit of quantitation. In this case, the lower limit of quantitation is ten times the standard deviation value σ calculated from measurement values in Table 2. Table 4 shows that for each component detected in the actual sample gas at a concentration higher than the lower limit of quantitation, the quantitated values from the initial analysis are about the same as from restoring, which indicates that quantitation values have good reproducibility. Furthermore, the measurement values for each component detected in the measurement sample gas at a concentration higher than the lower limit of quantitation tend to be larger when restoring than from the initial analysis. That tendency was presumably due to the lower recovery rates of toluene-d8, used as an internal standard substance during restoring, than for other substances.

The above results show that the TD-30R restore function can be used to analyze samples that contain VOCs with a wide range of low to high boiling points with accuracy that is comparable to the initial analysis. The restore function not only can be used to hedge against risks when analyzing precious samples, but also enables high-sensitivity analysis with high restore rates.

	Component	Actual Sample Gas 1			Actual Sample Gas 2			
I.D.		Initial Analysis	Restore	Ratio (Restore/Initial)	Initial Analysis	Restore	Ratio (Restore/Initial)	
1	Freon 114	<loq< td=""><td><loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<></td></loq<>	<loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<>	-	<loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<>	<loq< td=""><td>-</td></loq<>	-	
2	Chloroethene	N.D.	N.D.	-	N.D.	N.D.	-	
3	1,3-Butadiene	<loq< td=""><td><loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<></td></loq<>	<loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<>	-	<loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<>	<loq< td=""><td>-</td></loq<>	-	
4	Methyl bromide	N.D.	N.D.	-	N.D.	N.D.	-	
5	Ethyl chloride	<loq< td=""><td><loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<></td></loq<>	<loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<>	-	<loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<>	<loq< td=""><td>-</td></loq<>	-	
6	Freon 11	<loq< td=""><td><loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<></td></loq<>	<loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<>	-	<loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<>	<loq< td=""><td>-</td></loq<>	-	
7	1,1-Dichloroethene	N.D.	N.D.	-	<loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<>	<loq< td=""><td>-</td></loq<>	-	
8	Freon 113	<loq< td=""><td><loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<></td></loq<>	<loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<>	-	<loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<>	<loq< td=""><td>-</td></loq<>	-	
9	3-Chloro-1-propene	N.D.	N.D.	-	<loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<>	<loq< td=""><td>-</td></loq<>	-	
10	Methylene chloride	0.69	0.77	1.12	1.23	1.37	1.11	
11	Acrylonitrile	<loq< td=""><td><loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<></td></loq<>	<loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<>	-	<loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<>	<loq< td=""><td>-</td></loq<>	-	
12	1,1-Dichloroethane	N.D.	N.D.	-	<loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<>	<loq< td=""><td>-</td></loq<>	-	
13	cis-Di-1,2-Dichloroethylene	N.D.	N.D.	-	N.D.	N.D.	-	
14	Chloroform	<loq< td=""><td><loq< td=""><td>-</td><td>13.5</td><td>13.7</td><td>1.01</td></loq<></td></loq<>	<loq< td=""><td>-</td><td>13.5</td><td>13.7</td><td>1.01</td></loq<>	-	13.5	13.7	1.01	
15	1,1,1-Trichloroethane	<loq< td=""><td><loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<></td></loq<>	<loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<>	-	<loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<>	<loq< td=""><td>-</td></loq<>	-	
16	Carbon tetrachloride	<loq< td=""><td><loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<></td></loq<>	<loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<>	-	<loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<>	<loq< td=""><td>-</td></loq<>	-	
17	Benzene	0.32	0.34	1.06	0.53	0.55	1.04	
18	1,2-Dichloroethane	<loq< td=""><td><loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<></td></loq<>	<loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<>	-	<loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<>	<loq< td=""><td>-</td></loq<>	-	
19	Trichloroethylene	0.4	<loq< td=""><td>-</td><td>0.39</td><td><loq< td=""><td>-</td></loq<></td></loq<>	-	0.39	<loq< td=""><td>-</td></loq<>	-	
20	1,2-Dichloropropane	<loq< td=""><td><loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<></td></loq<>	<loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<>	-	<loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<>	<loq< td=""><td>-</td></loq<>	-	
21	1,3-Dichloropropene	N.D.	N.D.	-	N.D.	N.D.	-	
22	Toluene	3.08	3.26	1.06	3.95	4.06	1.03	
23	1,1,2-Trichloroethane	<loq< td=""><td><loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<></td></loq<>	<loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<>	-	<loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<>	<loq< td=""><td>-</td></loq<>	-	
24	Tetrachloroethylene	0.2	<loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<>	-	<loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<>	<loq< td=""><td>-</td></loq<>	-	
25	1,2-Dibromoethane	N.D.	N.D.	-	<loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<>	<loq< td=""><td>-</td></loq<>	-	
26	Chlorobenzene	<loq< td=""><td><loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<></td></loq<>	<loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<>	-	<loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<>	<loq< td=""><td>-</td></loq<>	-	
27	Ethylbenzene	0.42	0.43	1.02	0.18	<loq< td=""><td>-</td></loq<>	-	
28	m-Xylene	0.20	0.21	1.05	0.3	0.3	1.00	
29	p-Xylene	0.20	0.21	1.05	0.3	0.3	1.00	
30	o-Xylene	0.14	0.15	-	<loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<>	<loq< td=""><td>-</td></loq<>	-	
31	Styrene	0.12	0.13	1.08	<loq< td=""><td>0.08</td><td>-</td></loq<>	0.08	-	
32	1,1,2,2-Tetrachloroethane	N.D.	N.D.	-	<loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<>	<loq< td=""><td>-</td></loq<>	-	
33	1,3,5-Trimethylbenzene	<loq< td=""><td><loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<></td></loq<>	<loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<>	-	<loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<>	<loq< td=""><td>-</td></loq<>	-	
34	1,2,4-Trimethylbenzene	<loq< td=""><td><loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<></td></loq<>	<loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<>	-	<loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<>	<loq< td=""><td>-</td></loq<>	-	
35	4-Ethyltoluene	2.41	2.81	1.17	1.82	2.04	1.12	
36	m-Dichlorobenzene	<loq< td=""><td><loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<></td></loq<>	<loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<>	-	<loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<>	<loq< td=""><td>-</td></loq<>	-	
37	p-Dichlorobenzene	<loq< td=""><td>0.09</td><td>-</td><td><loq< td=""><td>0.09</td><td>-</td></loq<></td></loq<>	0.09	-	<loq< td=""><td>0.09</td><td>-</td></loq<>	0.09	-	
38	o-Dichlorobenzene	<loq< td=""><td><loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<></td></loq<>	<loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<>	-	<loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<>	<loq< td=""><td>-</td></loq<>	-	
39	1,2,4-Trichlorobenzene	<loq< td=""><td><loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<></td></loq<>	<loq< td=""><td>-</td><td><loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<></td></loq<>	-	<loq< td=""><td><loq< td=""><td>-</td></loq<></td></loq<>	<loq< td=""><td>-</td></loq<>	-	
40	Hexachloro-1,3-butadiene	<loq< td=""><td><loq< td=""><td>_</td><td><loq< td=""><td><loq< td=""><td>_</td></loq<></td></loq<></td></loq<></td></loq<>	<loq< td=""><td>_</td><td><loq< td=""><td><loq< td=""><td>_</td></loq<></td></loq<></td></loq<>	_	<loq< td=""><td><loq< td=""><td>_</td></loq<></td></loq<>	<loq< td=""><td>_</td></loq<>	_	

Table 4 Quantitation Results for Actual Sample Gas

4. Conclusions

The authors developed a TD system (TD-30R) capable of simultaneous analysis of compounds with a wide range of boiling points and with high sensitivity and high restore rates. As a result of using the optimized analytical conditions determined by experimentally considering the trap tube cooling and desorption temperature, the sample tube desorption temperature and flowrate, and the joint, valve, and transfer line temperatures, VOCs with a wide range of boiling points were analyzed using restore with accuracy levels equivalent to initial analysis.

Consequently, using the TD-30R unit not only can help avoid the risk of wasting precious VOC samples, but also enables simultaneous analysis of compounds with a wide range of boiling points, while also achieving high sensitivity and high restore rates.

TD-30R Thermal Desorption System

Revolutionary Thermal Desorption System Provides Excellent Processing Ability and Reliability

The TD-30 was developed as the optimal solution for gas and materials analysis. Its outstanding processing ability and excellent expandability provide strong support for all types of analysis, from work in research departments to quality control.

Outstanding Processing Ability and Basic Functionality

- Extensive sample capacity capable of accommodating 120 samples
- > Efficient analysis with the overlap function and interrupt function
- ▶ High-sensitivity analysis of high boiling point components using a sample line with no cold points

Excellent Expandability Enables a Variety of Analyses

- Hedging risks with the restore function
- ▶ Highly accurate quantitative analysis using a function that automatically adds an internal standard substance
- Highly reliable sample management using a barcode reader

Simple Operations and Ease of Maintenance

- Easy-to-maintain, user-friendly design
- Simple, easy-to-operate software

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TD-30R

Specifications and Installation Conditions

Tube Size	Outer diameter: 1/4" (6.35 mm); Length: 3.5" (89 mm)
Tube Desorption Temperature	Settings: 0 °C to 430 °C (1 °C increments), Control: Room temperature +15 °C to 430 °C (Accuracy ±1 °C)
Tube Desorption Flow Rate	Settings: 20 mL/min to 200 mL/min (1 mL/min increments; Accuracy ±2 mL/min)
Tube Desorption Time	Settings: 0 min to 240 min (0.01 min increments)
Trap Size	Outer diameter: 1/8" (3.2 mm); Inner diameter: 2 mm; Length: 102 mm SilcoNert® 2000 stainless steel tube rendered inert
Trap Adsorbent	TenaxTA [™] 60–80 mesh (60 mg) is standard. Carbopack [™] (50 mg) + Carbosieve® (10 mg) are optionally available. Carboxen® 1000 (70 mg) is optionally available.
Trap Desorption Temperature	Settings: 0 °C to 350 °C (1 °C increments); Control: 0 °C to 350 °C (Accuracy ±1°C)
Trap Cooling Temperature	Settings: -40 °C to 80 °C (1 °C increments) Control: Room temperature -50 °C to 80 °C (Valve temperature <250 °C); Room temperature -45 °C to 80 °C (Valve temperature > 250 °C); (Accuracy ±1 °C)
Split Ratio	1:5 to 1:200
Sample Path	SilcoNert® 2000
Switching Valve	6-port, 2-position, high temperature valve, motorized
Joint Temperature	Settings: 0 °C to 300 °C (1 °C increments); Control: Room temperature +15 °C to 300 °C (Accuracy ±1 °C)
Valve Temperature	Settings: 0 °C to 300 °C (1 °C increments); Control: Room temperature +15 °C to 300 °C (Accuracy ±1 °C)
Transfer Line Temperature	Settings: 0 °C to 350 °C (1 °C increments); Control: Room temperature +15 °C to 350 °C (Accuracy ±1 °C)
Internal Standard Added (TD-30R)	Fixed volume added: 0.5 mL; Variable volume added: 4 mL to 2000 mL
Dry Purge (TD-30R)	Temperature settings: -40 °C to 140 °C (1 °C increments) Control: Room temperature -50 °C to 140 °C (Valve temperature <250 °C); Room temperature -45 °C to 140 °C (Valve temperature >250 °C); (Accuracy ±1 °C) Flow rate: 20 mL/min to 200 mL/min (1 mL/min increments); Time: 0 min to 30 min (0.01 min increments)
Carrier Gas	High-purity helium or nitrogen, controlled by the advanced flow controller (AFC) built into the GC
Purge Gas	High-purity helium or nitrogen, controlled by the mass flow controller (MFC) built into the TD
PC Interface	USB
Control Software	TD-30 Control Software
Control Software Operating Environment	Microsoft® Windows® 7/10 (64/32 bit)
Environment for Guaranteed Performance	Temperature 18 °C to 28 °C; Relative humidity 20 % to 70 %
Power Supply	100 V AC / 120 V AC / 220 V AC / 240 V AC, 50/60 Hz, 1200 VA max.
Size	TD-30R: W720 × D690 × H470 mm, TD-30: W580 × D550 × H470 mm
Weight	TD-30R: 49 kg, TD-30: 48 kg

First Edition: January, 2019



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