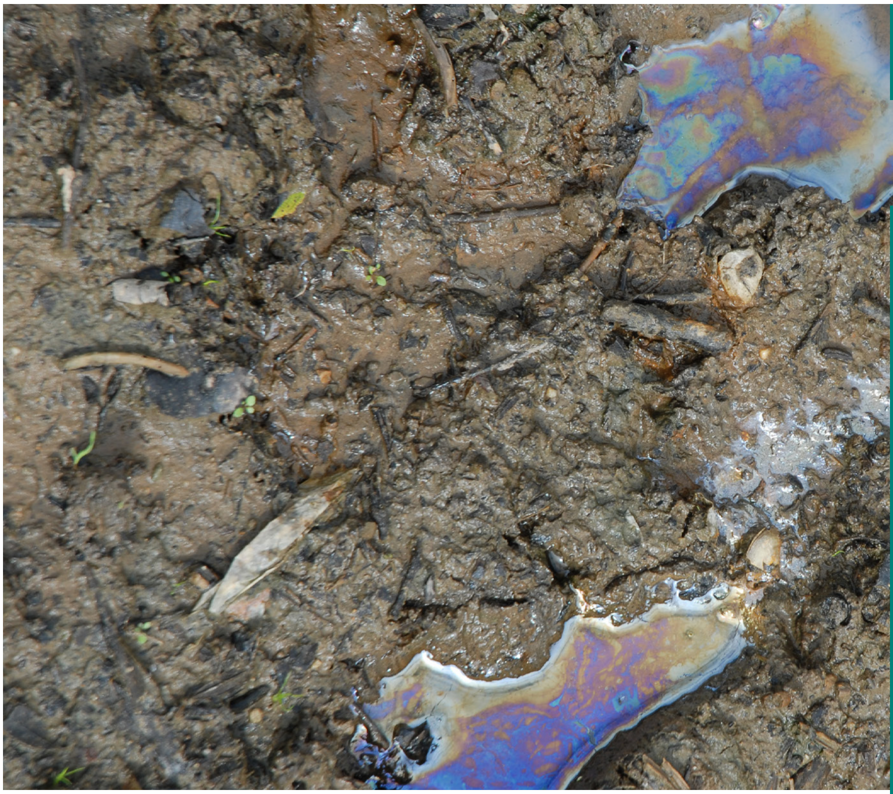

APPLICATION NOTE

Gas Chromatography

**Authors:**

**Kira. Yang**

**Yong. Xu**

PerkinElmer, Inc.
Shanghai, PRC

# Determination of Volatile Organic Compounds (VOCs) in Soil by HS-GC/FID

## Introduction

Volatile organic compounds (VOCs) are a series of compounds with high vapor pressure and boiling points from 50 to 250 degrees centigrade. These characteristics lead to the tendency for large numbers of molecules to evaporate, or volatilize, from their solid state into the air. VOCs are present in the environment from a number of sources, both anthropogenic and naturally occurring. In soil, VOCs are mainly derived from discharged industrial and domestic sewage, oil spills, and chemical solvent leakages. VOCs in atmospheric particulates can also enter the soil through dry and wet deposition. Monitoring of VOC contamination in soils by both qualitative and quantitative analyses is important to ensure that the potential negative health impacts of VOC exposure are mitigated.

US EPA Method 5021 is a general-purpose method for the preparation of soil samples using a headspace procedure for the determination of VOCs by gas chromatography/mass spectrometry (GC/MS), as described in Method 8260. The China Environmental Protection standard HJ 741-2015 has also been established for determination of VOCs in soil and sediment by HS-GC/FID, and is applicable to thirty seven VOCs.

In this study, thirty seven VOCs are investigated in a soil matrix, using a PerkinElmer Clarus® GC/FID and TurboMatrix™ HS-40. Detailed instrument method parameters are presented with precision, linearity and reporting limit results.

## Experimental

### Instrumentation

A PerkinElmer Clarus GC/FID and TurboMatrix HS-40 were utilized to perform these experiments, with detailed conditions presented in Table 1. A PerkinElmer Elite-624 column (60 m x 0.25 mm x 1.4 µm) was used to separate the eluting compounds. The headspace transfer line was installed into the GC split injector port. A patented zero dilution liner was used in the programmable split/splitless capillary injector (PSSI). A pulse sample introduction technique was utilized to provide high sensitivity and repeatability.

### Sample Preparation

10 µg/mL of calibration standard (37 VOCs mixture) was obtained from an independent laboratory. Analytical grade NaCl was purchased from Sinopharm Chemical Reagent Co. Ltd., and the pure water utilized in the experiment was produced by Mini-Q.

A standard sample was prepared by adding a volume of the calibration standard, 2.0 g of quartz sand and 10.0 mL of saturated sodium chloride solution to the 22 mL glass headspace vials with PTFE-faced septa. The vials were sealed immediately with the PTFE side of the septum facing toward the sample.

Method precision was investigated by spiking six vials with 0.50 µg of standard sample in quartz sand. Reporting limits were determined by analyzing the standard at an amount of 0.10 µg.

### Calibration

The calibration curve was prepared by adding 10, 20, 50, 100 and 200 µL of calibration standard to the headspace vials. The amount of VOCs in each vial was 0.10, 0.20, 0.50, 1.00 and 2.00 µg, respectively.

## Results and Discussion

The chromatogram of the calibration standard is shown in Figure 1. The calibration curves were plotted as the peak area versus the amount of the analyte. The coefficient of determination ($r^2$) was over 0.994, showing the reliability of the analysis in the range of 0.10 - 2.00 µg (Table 2). Table 2 summarizes the results for retention time (RT), linearity, repeatability and signal-to-noise ratio (S/N) at the reporting limit. Area repeatability, measured by the relative standard deviation (RSD), was found to be 2.77 - 6.75%.

*Table 1.* Analytical parameters.

| **HS Parameters** | |
|---|---|
| Needle Temperature | 85 °C |
| Transfer Line Temperature | 110 °C |
| Oven Temperature | 100 °C |
| Thermostatting Time | 20 min |
| Pressurization Time | 1 min |
| Injection Time | 0.1 min |
| Column Pressure | 38 psi |
| Vial Pressure | 45 psi |
| Operation Mode | Constant |
| **GC Parameters** | |
| GC Column | Elite-624 (60 m x 0.25 mm x 1.4 um) |
| Headspace Connector | Split Mode Connection |
| Inlet Temp | 220 °C |
| GC Injector | PSSI with a zero dilution liner |
| Carrier Gas | Nitrogen |
| Carrier Gas Flow Program 1 | 2.0 ml/min for 1.0 min. (Rate 999) |
| Carrier Gas Flow Program 2 | 1.0 min for remainder of run (hold 999) 1.0 ml/min |
| Split Flow | 1 ml/min |
| Initial Oven Temp | 40 °C |
| Oven Hold | 5 min |
| Ramp | 8 °C/min |
| 2nd Oven Temp | 100 °C |
| Oven Hold | 5 min |
| Ramp | 6 °C/min |
| 3rd Oven Temp | 200 °C |
| Oven Hold | 10 min |
| FID Temperature | 220 °C |
| $H^2$ | 40 mL/min |
| Air | 400 mL/min |
| Attenuation | -6 |

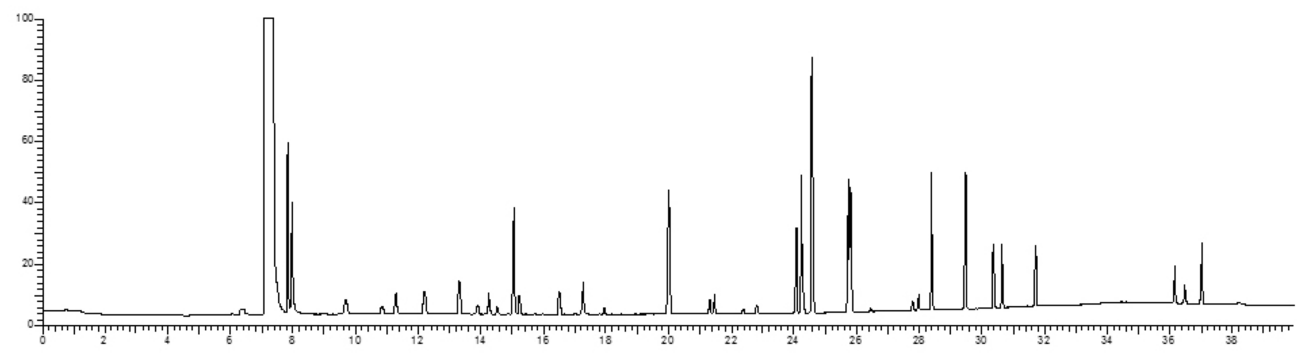

*Figure 1.* The chromatogram of a calibration standard (2.00 µg).

*Table 2.* Results for retention time, repeatability and reporting limit.

| **Compound Name** | **CAS No** | **RT** | **Repeatability** | **Reporting Limit** | **Linearity** | | |
|---|---|---|---|---|---|---|---|
| | | (min) | (RSD %) | (S/N at 0.1 µg) | Slope | Intercept | $r^2$ |
| Chloroethylene | 75-01-4 | 3.81 | 6.75 | 3.36 | 9501 | -538 | 0.9983 |
| 1,1-Dichloroethylene | 75-35-4 | 6.35 | 4.70 | 7.37 | 17804 | -356 | 0.9997 |
| Dichloromethane | 75-09-2 | 7.51 | 4.30 | 5.18 | 7291 | 417 | 0.9995 |
| E-1,2-Dichloroethylene | 156-60-5 | 7.96 | 5.51 | 7.46 | 18765 | -410 | 0.9992 |
| 1,1-Dichloroethane | 75-34-3 | 8.92 | 5.50 | 7.2 | 17703 | -273 | 0.9989 |
| Z-1,2-Dichloroethylene | 156-59-2 | 10.09 | 5.30 | 8.67 | 20622 | 6538 | 0.9993 |
| Chloroform | 67-66-3 | 10.7 | 6.21 | 7.08 | 6424 | -138 | 0.9988 |
| 1,1,1-Trichlorethane | 71-55-6 | 10.98 | 4.07 | 15.55 | 15105 | -47 | 0.9984 |
| Carbon tetrachloride | 56-23-5 | 11.22 | 4.08 | 5.79 | 5485 | -99 | 0.9982 |
| Benzene | 71-43-2 | 11.71 | 4.45 | 71.72 | 70291 | -1660 | 0.9990 |
| 1,2-Dichloroethane | 107-06-2 | 11.9 | 5.06 | 13.93 | 12889 | -378 | 0.9991 |
| Trichloroethylene | 79-01-6 | 12.97 | 6.51 | 16.76 | 15686 | -286 | 0.9998 |
| 1,2-Dichloropropane | 78-87-5 | 13.57 | 5.05 | 6.68 | 23831 | -733 | 0.9989 |
| Bromodichloromethane | 75-27-4 | 14.16 | 4.40 | 1.80 | 4501 | 73 | 0.9996 |
| Toluene | 108-88-3 | 16.02 | 4.97 | 29.77 | 71819 | 995 | 0.9991 |
| 1,1,2-Trichloroethane | 79-00-5 | 17.48 | 2.77 | 8.22 | 8648 | -180 | 0.9989 |
| Tetrachloroethylene | 127-18-4 | 17.58 | 4.50 | 10.03 | 12114 | -173 | 0.9986 |
| Dibromochloromethane | 124-48-1 | 18.68 | 4.27 | 8.30* | 3057 | -158 | 0.9995** |
| 1,2-Dibromoethane | 106-93-4 | 19.11 | 3.18 | 1.92 | 5331 | -195 | 0.9985 |
| Chlorobenzene | 108-90-7 | 20.61 | 3.30 | 39.32 | 45523 | -665 | 0.9991 |
| Ethylbenzene | 100-41-4 | 20.86 | 4.90 | 61.09 | 83158 | -1645 | 0.9986 |
| 1,1,1,2-Tetrachloroethane | 630-20-6 | 20.86 | 4.90 | 61.09 | 83158 | -1645 | 0.9986 |
| m, p-Xylene | 108-38-3/<br>106-42-3 | 21.26 | 5.78 | 115.1 | 71236 | -2876 | 0.9985 |
| o-Xylene | 95-47-6 | 22.60 | 5.18 | 57.95 | 70050 | -1686 | 0.9986 |
| Styrene | 100-42-5 | 22.71 | 2.73 | 58.24 | 64212 | -708 | 0.9989 |
| Bromoform | 75-25-2 | 23.40 | 5.04 | 5.64* | 1822 | -9 | 0.9994** |
| 1,1,2,2-Tetrachlorethane | 79-34-5 | 25.07 | 4.39 | 5.43 | 5279 | -17 | 0.9942 |
| 1,2,3-Trichloropropane | 96-18-4 | 25.24 | 5.26 | 7.70 | 8586 | -349 | 0.9975 |
| 1,3,5-Trimethylbenzene | 108-67-8 | 25.76 | 5.51 | 43.21 | 70260 | -2081 | 0.9988 |
| 1,2,4-Trimethylbenzene | 95-63-6 | 26.99 | 4.76 | 33.73 | 68152 | -1784 | 0.9991 |
| 1,3-Dichlorobenzene | 541-73-1 | 27.96 | 2.69 | 29.97 | 31762 | -420 | 0.9991 |
| 1,4-Dichlorobenzene | 106-46-7 | 28.27 | 3.78 | 30.84 | 31268 | -174 | 0.9989 |
| 1,2-Dichlorobenzene | 95-50-1 | 29.47 | 4.32 | 15.74 | 31347 | -477 | 0.9991 |
| 1,2,4-Trichlorobenzene | 120-82-1 | 34.44 | 5.35 | 13.79 | 21533 | -284 | 0.9992 |
| Hexachlorobutadiene | 87-68-3 | 34.81 | 4.74 | 7.46 | 12185 | -160 | 0.9994 |
| Naphthalene | 91-20-3 | 35.28 | 3.33 | 21.7 | 37201 | -1193 | 0.9987 |

* Reporting limit was determined by analyzing the standard at an amount of 0.50 µg.

** The calibration curves were plotted in the range of 0.20 to 2.00 µg.

## Summary

In this study, the analysis of thirty-seven VOCs in soil is performed efficiently and effectively by the PerkinElmer TurboMatrix HS 40 - Clarus GC/FID system. It is a simple, flexible, cost effective, accurate and precise technique for the detection of VOCs in soil, enabling accurate detecting of environmental samples.

**PerkinElmer, Inc.**
940 Winter Street
Waltham, MA 02451 USA
P: (800) 762-4000 or
(+1) 203-925-4602
**www.perkinelmer.com**

**For a complete listing of our global offices, visit www.perkinelmer.com/ContactUs**