SHIMADZU APPLICATION NEWS

LA180-E007

● GAS CHROMATOGRAPHY

No.G208

Easy Analysis of Residual MTBE in Environmental Water and Soil

MTBE (t-butyl methyl ether) is a compound that was added mainly to high-octane gasoline to boost the octane of the gasoline. In the US, MTBE caused pollution of soil and groundwater and, two or three years later, the decision was made to discontinue the addition of MTBE to gasoline. In Japan, an Environmental Agency investigation detected trace amounts of MTBE in well water. Because of the concern that leaking of gasoline containing MTBE, which has a stimulating odor and is suspected to be carcinogenic, would pollute the groundwater and the soil, major oil distribution companies decided to halt sales of high-octane gasoline containing MTBE in the summer of 2001.

Not much has been done to measure and gain knowledge of the MTBE residue concentrations in environmental water and soil. Comprehensive studies about the affect of residual MTBE will be started in the near future. Headspace GC/MS and purge-and-trap GC/MS methods are commonly used for the analysis of volatile hydrocarbon compounds. GC-FID can be used instead of GC/MS for screening purposes, or when the sample concentration is relatively high.

This Application News introduces the use of headspace GC-FID for easy analysis of residual MTBE in environmental water and soil.

In headspace methods, when solid samples are directly enclosed in to the head space vial and analyzed, quantification may become poor in some cases. Therefore, in this analysis example, the soil was diluted to ten times in volume with blank water, sealed, and stirred with a magnetic stirrer for four hours. Then, 10mL of supernatant was taken and added to a headspace vial containing 3g sodium sulfate, enclosed, and analyzed. This test liquid adjustment method is in accordance with the low molecular halogenated hydrocarbon analysis methods stipulated in the Environmental Agency Bulletin No. 46 of August 23, 1991 (revisions: 1993 Environmental Agency Bulletin No. 19, 1994 Environmental Agency Bulletin No. 25; and 1995 Environmental Agency Bulletin No. 19. (Fig. 1)

The solution with added gasoline was diluted with isopropanol, added to a headspace vial containing 3g sodium sulfate and 10mL blank water, enclosed, and analyzed. Figs. 2 and 3 show the chromatograms of solutions corresponding to 0.01 and 0.07ppm (V/V) MTBE. Fig. 4 shows the linearity of gasoline-added water for 0.01 to 0.07ppm (V/V) MTBE. The figure shows good linearity. The minimum detection limit is about 10ppb concentration in water solutions (about 100ppb in soil). Besides MTBE, toluenes and xylenes could be simultaneously analyzed with the same analytical conditions.

Table 1 shows the repeatability for gasoline-added water corresponding to 0.7ppm (V/V) MTBE. Good repeatability was obtained for both retention time and area value.

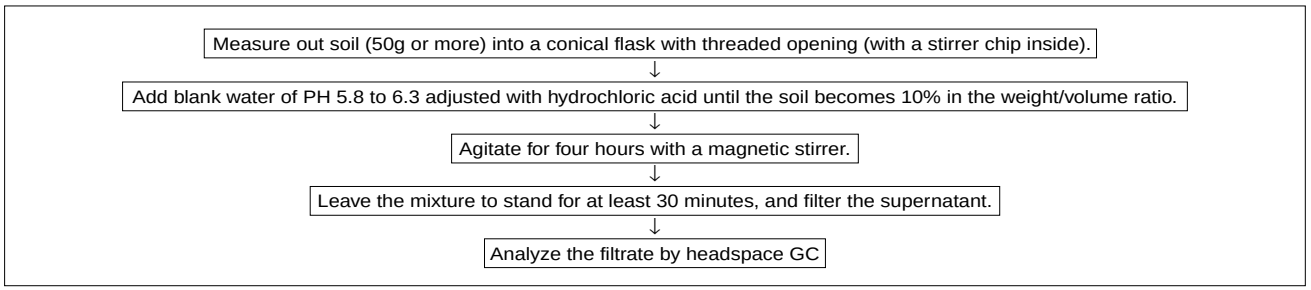

Fig.1 Pretreatment for analysis of residual MTBE in soil (in accordance with Environmental Agency bulletin No. 46 on Aug. 23, 1991, revision No. 19 in 1993, No. 25 in 1994 and No. 19 in 1995)

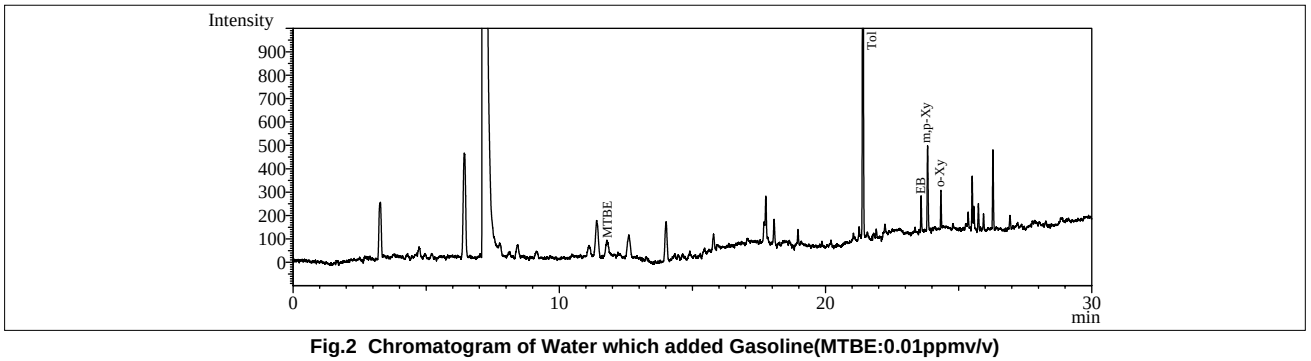

Fig.2 Chromatogram of Water which added Gasoline(MTBE:0.01ppmv/v)

Gasoline with MTBE was added and mixed into soil to correspond to several hundred ppb, and the sample liquid was prepared in the method shown in Fig. 1. Fig.5 shows the chromatogram obtained by analyzing the sample liquid. The MTBE peak is very small. This may be attributed to evaporation during the mixing process.

This analysis method, in which target components in the soil are transferred to blank water, can be applied to the analysis of MTBE in environmental water.
Because the selectivity of GC-FID low, GC/MS is recommended for confirmation testing. Headspace GC-FID is very effective for screening residual MTBE.

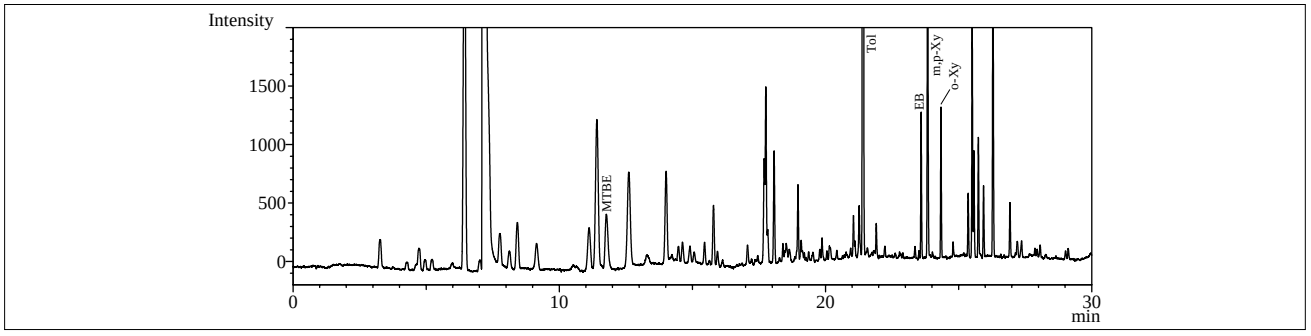

Fig. 3 Chromatogram of water which added gasoline (MTBE: 0.07 ppm v/v)

Table 1 Repeatability of retention time and peak area in water which added gasoline (MTBE: 0.07 ppm v/v)

n	Retention time (min.)	Peak area (mV·sec.)
1	11.739	26912
2	11.740	26757
3	11.742	25463
4	11.741	25942
5	11.737	26334
Average	11.740	26282
S.D.	0.0019	593.98
C.V.%	0.0159	2.26

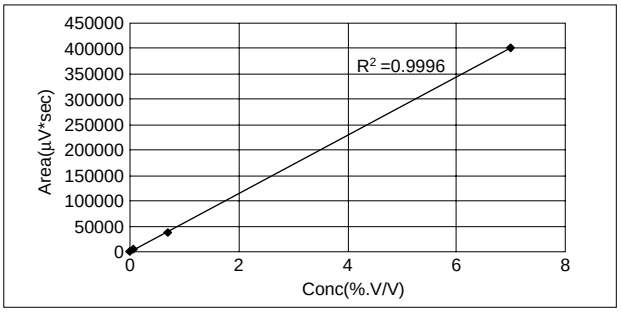

Fig.4 Calibration curve of MTBE in water which added gasoline (MTBE: 0.01 to 0.07 ppm v/v)

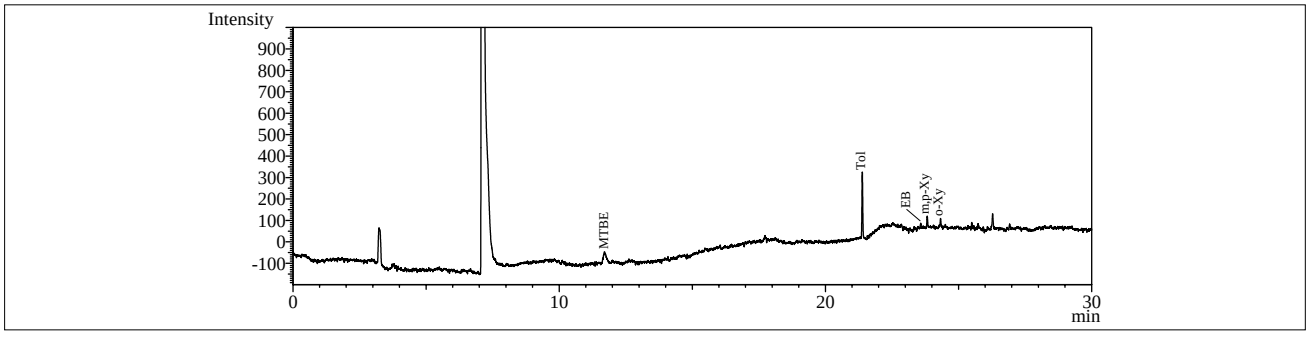

Fig. 5 Chromatogram of water extracted from soil which added gasoline

Table 2 Analytical conditions

Equipment	: GC-2010+Turbomatrix HS40,
Column	: DB-VRX 0.25mm×60m, df=1.4μm
Col.Temp.	: 35˚C (13min)→13˚C/min→185˚C (13min)→15˚C/min→255˚C (10min)
Carrier Gas	: He, 230kPa (2.2mL/min), Inj.Temp.:150˚C, Det:FID, Det.Temp.:250˚C,
Split	: 1:8
Thermostatting Temp.:80˚C, Thermosttating Time:30min, Injection Time:0.1min	

Reference

JIS Handbook Environmental Measurement 1998, edited by Japanese Standards Association (1998)

Environmental Agency Bulletin No. 46 (1991), No. 19 (1993), No, 25 (1994), No. 19 (1995)

SHIMADZU

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