Simultaneous Determination of Benzene, Toluene, Ethyl Benzene, xylene (BTEX) and Methyl *Tertiary*-Butyl Ether (MTBE) in Soil by Headspace Gas Chromatography-Mass Spectrometry.

Ho-Sang Shin¹· Hye-Sil-Ahn² and Tae Seung Kim³

¹Department of Environmental Education,

²Department of Environmental Science, Kongju National University, Kongju, Korea

³Soil & Groundwater Division, National Institute of Environmental Research(NIER), Environmental Research Complex, Kyungseo-Dong, Seo-Gu, Incheon, Korea

A headspace gas chromatography-mass spectrometric assay method was developed for the simultaneous determination of benzene, toluene, ethyl benzene, xylene(BTEX) and methyl tertiary-butyl ether(MTBE) in soil contaminated with gasoline. 2g of soil sample were placed in 10 mL vial filled with 5 mL of phosphoric acid solution (pH 2) saturated with NaCl, and the solution was spiked with fluorobenzene as an internal standard and sealed with cap. The vial was placed in a heating block for 50min at 80°C. The detection limits of the assay were 0.1 ng/g for MTBE, benzene, toluene and m-xylene, and, 0.2 ng/g for ethyl benzene, o,p-xylene. A regression line of peak area ratio for target compounds to internal standard on concentration using a least-squares fit demonstrated a linear relationship with correlation coefficient being greater than 0.995. The reproducibility of the assay was very good. For five independent determinations at 10ng/g, the relative standard deviations were less than 10%. The developed method may be valuable to be used to the national monitoring project of BTEX and MTBE in soil.

Table 1. GC-MS conditions for analysis of MTBE and BTEX

Parameter	Condition							
Column Carrier Oven Temp. Split Ratio Injector Temp. Transfer Temp.	HP-5MS(30m×0.2mmI.D.× 0.25 μ m F.T) He at 0.9mL/min 35 °C (1 min) → 5 °C/min → 70 °C (3 min) 1:10 250 °C 280 °C							
	Group	Start Time(min)	Selected Ions, m	/z				
Selected Ion Group	1 2 3 4	1.4 2.1 3.5 6.5	41, 57, 73 78, 96 91, 92 91, 106					

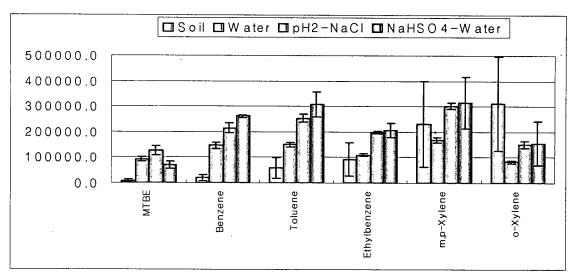


Figure 1. Extraction yield according to several conservation solutions

Table 2. QA & QC results for the analysis of MTBE and BTEX in soil

Compounds	Linear regression	Correlation	MDL	Spiked	average± SD	
Compounds	Linear regression	coefficient	(ng/g)	Conc.(ng/g)	(RSD%)	
MTBE	Y = 0.0129x + 0.0286	0.9954	0.1	10	$9.1 \pm 1.3 (13.7)$	
Benzene	Y = 0.0323x + 0.0367	0.9962	0.1	10	$10.9 \pm 0.8 (7.3)$	
Toluene	Y = 0.0512x + 0.1587	0.9958	0.1	10	8.6 ± 0.7 (8.6)	
Ethylbenzene	Y = 0.0552x + 0.082	0.9985	0.2	10	9.3 ± 0.6 (6.4)	
m,p-Xylene	Y = 0.0800x + 0.1988	0.9963	0.1	10	$7.3 \pm 0.7 (9.4)$	
o-Xylene	Y = 0.0465x + 0.0533	0.9983	0.2	10	8.8 ± 0.7 (7.6)	