

MTBE Residue in Environmental Water and Soil Analysis by Agilent 7820 GC-FID

Application Note

Authors

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Abstract

A method was developed for the detection of MTBE residue in environmental water and soil using an Agilent 7820 GC with FID. The method showed linear calibration with a detection limit range of 4.0–80.0 ng/mL, and an $R^2 = 0.9996$.

t-Butyl methyl ether (MTBE) has been used in U.S. gasoline at low levels since 1979, replacing lead as an octane enhancer to help prevent engine knock. Underground storage tank leaks and spills are the major sources of MTBE pollution. Car accidents, boats, and storm water runoff also release gasoline into the environment. MTBE moves quickly through soil, dissolves easily in water, and takes longer to break down than some other chemicals. A growing number of studies have detected MTBE in ground water. In some instances, these are contaminated sources of drinking water. Low levels of MTBE can make drinking water supplies undrinkable owing to its offensive taste and pungent odor. Drinking water regulations indicate that there is little likelihood that MTBE in drinking water will cause adverse health effects at concentrations between 20 and 40 ppb or below.

An Agilent 7820 GC with an Agilent 7697A Headspace Sampler were used to measure the MBTE in water and soil in this application note.



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Experiment

Reagents and chemicals

t-Butyl methyl ether (from Fisher Scientific, pure);
Na₂SO₄(from Sinopharm chemical reagent Co., Ltd, AC grade)

Equipment and materials

This method was developed on the Agilent 7820 GC and 7697A Headspace Autosampler (p/n G4556). Carrier gas was controlled by a 7820 Electronic Pneumatic Controller (EPC) and the system included a split/splitless inlet and flame ion detection (FID). The 7697A transfer line was installed through the inlet septum. The GC liner was direct 2-mm id (p/n 5181-8818). The 7697A used 20-mL vials (p/n 5190-2288); 20-mm crimper (p/n 5040-4669); Headspace Al crimp cap (PTFE/Si sep, 20-mm, p/n 5183-4477).

Table 1. Agilent 7697A Headspace Sampler and GC/MS Instrument Conditions

Agilent 7697A Headspace Sampler conditions	
Oven temperature	80 °C
Loop temperature	85 °C
Transfer line temperature	100 °C
Vial equilibration time	30 minutes
Injection time	0.5 minutes
GC cycle	32 minutes
Vial	20 mL, default fill mode
Fill pressure	He, 15 psi
Extraction mode	single extraction
GC conditions	
Inlet	150 °C, 8:1split
Carrier gas	He, flow mode: 1.5 mL/min
Injection volume	1 mL from 1 mL headspace loop
Column	Agilent DB-624 (30 m × 0.25 mm, 1.4 μm)
Oven temperature gradient	13 minutes hold at 35 °C, then 15 °C/min to 185 °C, hold for 1 minute
FID	250 °C ; H ₂ : 30 mL/min; make up + constant flow: He, 25 mL/min; air: 400 mL/min
FID signal	20 Hz

Sample preparation

1. Dilute the soil to ten times volume with pure water.
2. Seal the samples.
3. Stir for 2 hours.
4. Filter the supernatant.
5. Remove 10 mL of supernatant.
6. Add to a 20-mL headspace vial containing 3 g of sodium sulfate.
7. Cap the sample vial and heat to 80 °C.
8. Transfer the sample to the GC.

Results and Discussion

Calibration

Dilute MTBE with pure water to make a 10 ug/mL MTBE solution. Inject 2, 5, 10, 20, 30, 40 μL, of 10 μg/mL MTBE solution into six separate 20-mL headspace vials containing 5 mL of pure water. The calibration curve was constructed with these six calibration standards and the correlation coefficients (R²) were 0.9996. The detection limit was 1.7 ng/mL. The calibration curve is shown in Figure 1. The MTBE chromatogram is shown in Figure 2.

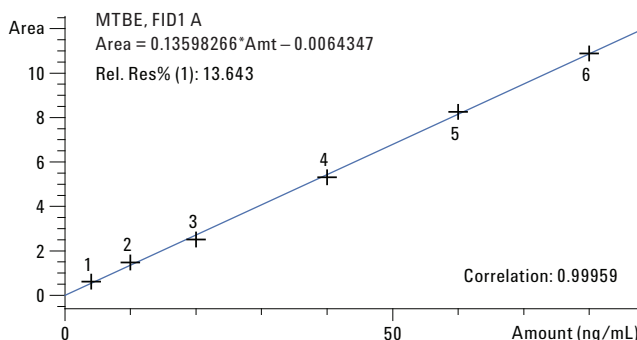


Figure 1. MTBE calibration curve.

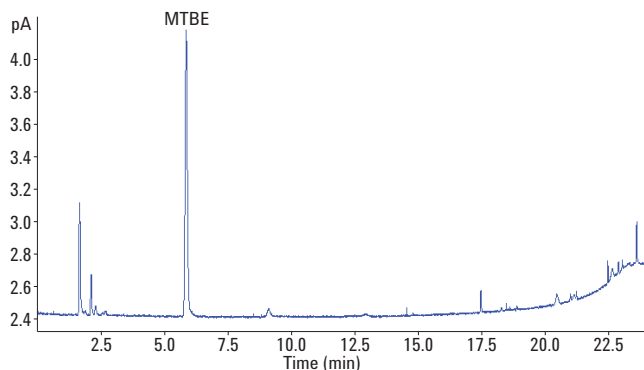


Figure 2. 40 µL, 10 µg/mL calibration solution chromatogram, MTBE retention time is 5.87 minutes.

Repeatability

Method repeatability was tested using five separate 20-µL injections of 10 µg/mL MTBE solution with the headspace sampler. The retention time and area RSD% of five injections are listed in Table 2. Figure 3 shows the overlay chromatogram.

Table 2. RSD% of MTBE

Name (n = 5)	RSD (%)	
	RT	Area
MTBE	0.02	5.48

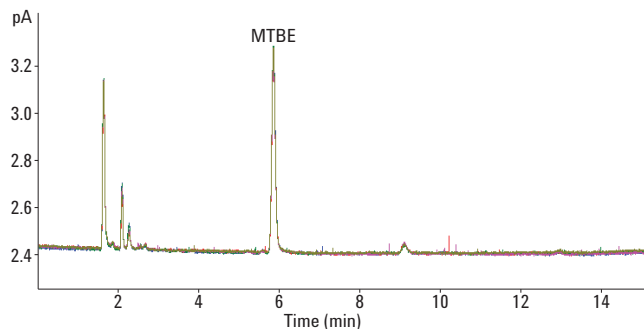


Figure 3. Overlay of five chromatograms.

Method validation

Soil

The new method was validated using actual soil and water samples. A 10-mL pure soil sample supernatant was analyzed according to the method, showing no detectable MTBE in the sample. An 80 ppb amount of MTBE was then added to the soil sample and reanalyzed. The results of these tests are shown in Figure 4 as a chromatographic overlay. As the figure shows, there was no MTBE detected in the pure sample. However, MTBE was detected in the injected sample. MTBE recovery was 95.65%.

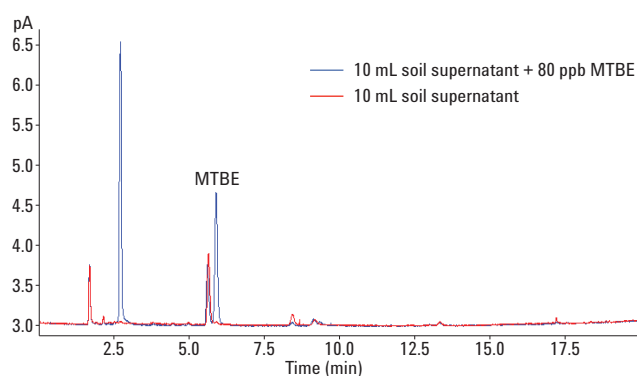


Figure 4. Contrast chromatograms of soil sample (red) and standard added sample (blue).

Water

A 10-mL water sample was analyzed for MTBE under experimental method conditions. Similarly to the soil, there was no MTBE detected in the pure sample. A 40 ppb amount of MTBE was injected into the sample and analyzed under the same conditions. The results are shown in Figure 5 as a chromatographic overlay. The figure shows MTBE detected in the second sample, and the recovery was 90.20%.

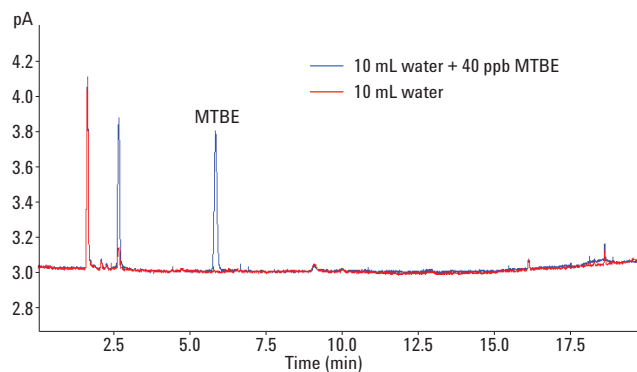


Figure 5. Contrast chromatograms of water sample (red) and standard added sample (blue).

Conclusions

A practical method was developed on Agilent 7820 GC and validated using real soil and water samples. The method has good linearity and repeatability for MTBE analysis.

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