

# Investigation of methyl *tert*-butyl ether levels in river-, ground-, and sewage-waters analyzed using a purge-and-trap interfaced to a gas chromatograph–mass spectrometer

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## Abstract

River water collected from 27 sites in 21 rivers, groundwater from 66 sites in 34 cities, and 2 sewage plants in the Niigata Prefecture, Japan (total 301 samples) were analyzed for methyl *tert*-butyl ether (MTBE) using an improved purge-and-trap-GC–MS method. The amount of MTBE found in water samples from the 27 river sites ranged from 0.003 to 5.3  $\mu\text{g l}^{-1}$ . MTBE was found in all 27 sites during winter but it was found at only 9 sites and 14 sites, respectively, during the summer. MTBE was found most frequently (in 21 sites) at levels ranging from 0.003 to 0.009  $\mu\text{g l}^{-1}$  during February. The highest levels of MTBE were always found in the samples collected from the same site. The amount of MTBE found in sewage water samples ranged from <0.003 to 0.025  $\mu\text{g l}^{-1}$ . No significant differences were observed between the amounts of MTBE recovered from inflow and outflow waters. Relatively high levels (0.02–0.034  $\mu\text{g l}^{-1}$ ) of MTBE were found in January at two sites, which were located on the upper course of the Shinano River. MTBE levels ranged from 0.004 to 0.035  $\mu\text{g l}^{-1}$  and from 0.005 to 0.041  $\mu\text{g l}^{-1}$  at the mouths of the Shinano River and Agano River, respectively. The levels of MTBE in groundwater collected from 66 sites in 34 cities in Niigata Prefecture ranged from <0.003 to 5.9  $\mu\text{g l}^{-1}$ .

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**Keywords:** Groundwater; Gas chromatography–mass spectrometry; Methyl *tert*-butyl ether; Purge-and-trap; River water; Sewage water

## 1. Introduction

Human exposure to methyl *tert*-butyl ether (MTBE) has begun to receive much attention from environmental scientists as a chemical that has been released into the environment constantly and consistently [1,2]. It has been used in large quantities as an anti-knocking agent in gasoline, oxyfuels, and low-boiling-point solvents. Consequently, concerns have been raised about its possible adverse effects on humans [3]. Therefore, the US Environmental Protection Agency (EPA)

has proposed to develop guidelines under the Toxic Substances Control Act to eliminate or limit the use of MTBE as a fuel additive in gasoline [4]. MTBE has also received much attention as a chemical requiring serious investigation from counterpart agencies in Japan [5–8].

Among variability of exposure studies, the time of sampling may play an important role because MTBE is volatile and readily decomposes in UV light. For example, when 40 ml of vapor-phase MTBE was exposed to a regular home fluorescent lamp for 18 h, 2.72  $\mu\text{mol}$  of formaldehyde, 25.5  $\mu\text{mol}$  of butanal, and 12  $\mu\text{mol}$  of 2-butanone were formed [9]. *tert*-Butyl formate, *tert*-butyl alcohol, methyl acetate, and acetone were reported as breakdown products of MTBE oxidized with Fenton's reagent [10]. However, once

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it enters surface water or groundwater, MTBE is extremely difficult to eliminate due to its high water solubility ( $4.8 \text{ g } 100 \text{ g}^{-1}$ ). Moreover, MTBE is strongly persistent toward microbial degradation [11,12]. Therefore, it is important to determine the levels of MTBE in surface water and groundwater to assess the environmental contamination caused by MTBE.

There are many reports on trace analysis of MTBE in environmental water samples such as groundwater samples [13,14], seawater samples [15], and lake water samples [16]. These studies were performed using purge-and-trap gas chromatography–mass spectrometry, which is the method that has been used most commonly for MTBE analysis in environmental samples.

In the present study, MTBE levels of water samples collected from 21 rivers, 66 wells in 34 cities, and 2 sewage plants in Niigata Prefecture, Japan were analyzed to evaluate the seasonal distribution of MTBE levels in the environment using purge-and-trap/gas chromatography–mass spectrometry.

## 2. Experimental

### 2.1. Chemical and reagents

MTBE (analytical grade for pesticide residues) stock solution ( $1000 \mu\text{g l}^{-1}$  in methanol) was purchased from Kanto

Kagaku (Tokyo, Japan). [ $^2\text{H}_{12}$ ] (MTBE- $\text{d}_{12}$ ) was purchased from CDN Isotopes (Quebec, Canada). Water (VOC analytical grade) was bought from Wako (Osaka, Japan). The standard stock solutions of MTBE and MTBE- $\text{d}_{12}$  were prepared in a methanol solution ( $1000 \mu\text{g l}^{-1}$ ).

### 2.2. Sample collections

Water samples from rivers were collected from 27 sites in 21 rivers shown in Fig. 1 in February and July 2002, and July 2003 (a total of 81 samples). Also, river waters were collected from 6 sites in the main stream of the Shinano River (Sites 5–10) once a month on the same day of the month from January and December 2002 (a total of 72 samples). Downstream (Site 6), samples were collected twice a month from January to March 2002 and in November and December 2003 (a total of 10 samples). At the mouths of the Shinano River (Site A) and the Agano River (Site B), samples were collected once a month on the same day of the month from January to December 2003 (total 24 samples).

Water samples were collected from the inflows and outflows of the two sewage plants in Niigata City, Niigata Prefecture during two days in December 2002 and August 2003. A total of 48 water samples from each plant (24 samples each from inflow and from outflow) were collected from 10 a.m.

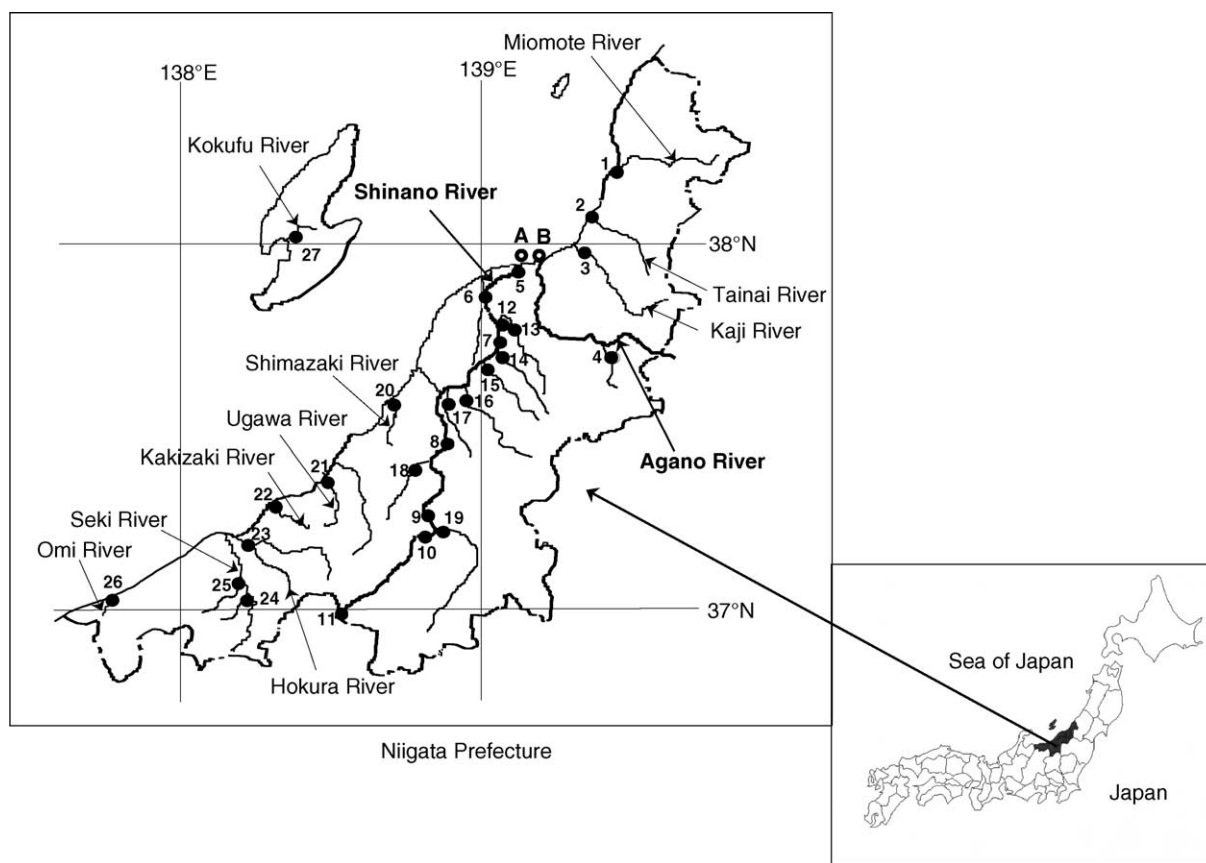


Fig. 1. Sampling sites in Niigata Prefecture, Japan. (●): Sites on rivers (Sites 1–27) (○): Sites at mouth of Shinano River (Sites A) and mouth of Agano River (Site B).

on the first day to 9 a.m. the second day at 1 h intervals using an auto-sampler.

Groundwater samples were collected from April to August 2001 at 66 sites in 34 cities in Niigata Prefecture (66 samples).

Analyses were performed upon arrival of the samples at the laboratory or stored at 4 °C until analyses (not more than 2 days).

### 2.3. Purge-and-trap system

A water sample (44 ml) for analysis was placed in a vial (GL Sciences, Tokyo, Japan). After 4.4 ng of MTBE-d<sub>12</sub> (internal standard) was added to the sample, the vial was sealed.

A Tekmar LSC 4000J concentrator (Tekmar, Ohio, Canada) on-line coupled with the GC–MS system and equipped with a Tenax TA adsorbent trap (GL Sciences, Tokyo, Japan) was used. Helium was used as a purging gas. Cryo-focus temperature was –180 °C.

It is important to find optimum conditions for analysis of volatile chemicals, such as MTBE, to obtain reliable results. Therefore, using an MTBE standard solution (0.06 µg l<sup>-1</sup> in purified water), the optimum conditions of purging time (4, 6, 8, 10, 12, and 14 min), purging temperature (20, 30, 40, and 50 °C), desorbing time (6, 8, 10, 12, and 14 min) and desorbing temperature (160, 180, 200, and 220 °C) for the purge-and-trap process were examined. With optimum conditions obtained from the above experiments, the LOD of MTBE was determined using a standard MTBE solution (0.010 µg l<sup>-1</sup> in purified water). The recovery efficiencies of MTBE from river water, ground-water, and seawater were also determined using solutions containing 0.06 µg l<sup>-1</sup> of MTBE for each.

### 2.4. GC–MS analyses

A Hitachi Model G-7000M GC equipped with a 60 m × 0.25 mm I.D. (*d<sub>f</sub>* = 1.4 µm) DB-VRX fused silica capillary column (J & W Scientific, Folsom, CA, USA) and a Hitachi M-9000 MS (Hitachi, Tokyo, Japan) was used to analyze MTBE. The GC oven temperature was held at 40 °C for 3 min and programmed at 4 °C min<sup>-1</sup> to 90 °C, and then 8 °C min<sup>-1</sup> to 230 °C and held for 7 min. GC–MS interface temperature was 200 °C. Ion source temperature was 180 °C. Emission current was 10 µA and photomultiplier voltage was 330 V. The mass spectrometer was operated in multiple reactant monitoring mode (MRM) to observe *m/z* 73 and 57 for MTBE, and 82 for MTBE-d<sub>12</sub>. The GC retention times of MTBE and MTBE-d<sub>12</sub> were 12.02 and 11.75 min, respectively.

## 3. Results and discussion

There have been several reported methods for trapping MTBE in ground and surface waters. These techniques include the headspace method [17], purge and trap method [13–19], and solid-phase microextraction method [20,21].

MTBE trapped by these methods was subsequently analyzed by GC–MS. Among these methods, the level of MTBE recovered is much higher by the solid phase micro-extraction method than by the headspace method. However, a sample recovered by solid phase micro-extraction method tends to be contaminated by the chemicals degraded from solid phase materials that interfere with GC–MS analysis [22]. Therefore, the purge-and-trap method, which has high recovery and reproducibility, was improved and used in the present study. In the case of GC–MS analysis, surrogate chemicals are used as an internal standard to increase accuracy. MTBE-d<sub>3</sub> is commonly used for MTBE analysis [13,18,20]. However, among the monitoring mass spectral peaks of MTBE-d<sub>3</sub> (*m/z* = 76 and 57), *m/z* 57 is consistent with that of MTBE. Therefore, *m/z* 57 is not applicable as a fragment ion for confirmation. Also, *m/z* 76 from MTBE-d<sub>3</sub> is the same as a base peak of carbon disulfide. Therefore, if an MTBE sample contained carbon disulfide, it would interfere with MTBE quantitation because MTBE and carbon bisulfide are not readily separated by a GC column. All of these problems were solved by using MTBE-d<sub>12</sub> and monitoring peaks *m/z* 82 and 66 in the present study.

### 3.1. Optimization of analytical conditions

The optimum conditions obtained for purge-and-trap process were 40 °C for purging temperature, 10 min for purging time, 220 °C for desorbing temperature, and 10 min for desorbing time.

The limit of detection (LOD) of MTBE in the water samples by GC–MS in the literature ranged from 0.01 to 2.0 µg l<sup>-1</sup> [14–19]. Recently, a lower LOD (0.001 µg l<sup>-1</sup>) than those was reported [13]. However, the value in this report was calculated using a conventional signal-to-noise ratio of 3 method. The LOD, which was determined by the method previously reported [22,23], was 0.0029 µg l<sup>-1</sup> in the present study.

The recovery efficiencies of MTBE at the level of 0.06 µg l<sup>-1</sup> were 99.1 ± 3.4% (*n* = 7) for the river-water, 103 ± 3.4% (*n* = 7) for the ground-water, and 100 ± 2.1% (*n* = 7) for the sea-water in the present study.

### 3.2. MTBE concentrations in the rivers in Niigata Prefecture during summer and winter

Fig. 1 shows the 27 collection sites from the 21 rivers in Niigata Prefecture. Fig. 2 shows MTBE concentrations found in the samples collected during the winter (February 2002) and summer (June 2002 and 2003). The frequencies of MTBE appearance in the sites were 27/27 (100%) in February 2002, 9/27 (32.5%) in June 2002, and 14/27 (51.9%) in June 2003. Namely, MTBE was found in all sites during the winter but it was found at only 9 sites and 14 sites during the summers of 2002 and 2003, respectively. MTBE was found in at more sites during winter than summer. However, the levels of MTBE found during all three collection times (9 sites) were

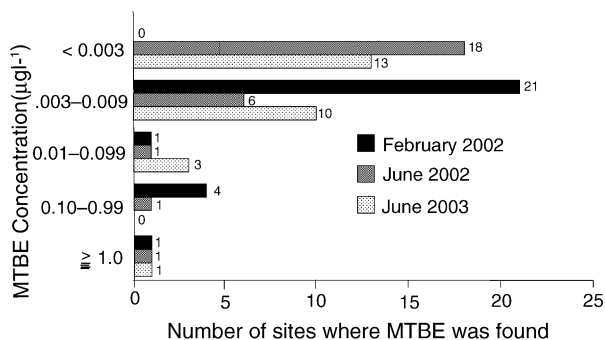


Fig. 2. Number of sites where MTBE was found at various levels in river waters.

not higher during the winter than during the summer. The levels of MTBE found at six sites during the summer were higher than those found during the winter. MTBE appeared most frequently at levels ranging from 0.003 to 0.009  $\mu\text{g l}^{-1}$  during February 2002 (21 sites).

The levels of MTBE in the samples from the 26 sites—except for one site, which contained a high level of MTBE ( $5.3 \mu\text{g l}^{-1}$ )—ranged from 0.003 to  $0.25 \mu\text{g l}^{-1}$ . Levels of MTBE exceeded  $0.10 \mu\text{g l}^{-1}$  at only four sites. The highest levels of MTBE were always found in the samples collected from the same site. These levels are similar to the ones reported in other parts of Japan [21]. These levels are also consistent with the levels reported in the waters from the other parts of the world, including a small urban creek in Germany ( $0.025\text{--}0.10 \mu\text{g l}^{-1}$ ) [20]; rivers in New Jersey, California, and Finland ( $0.3\text{--}0.4 \mu\text{g l}^{-1}$ ) [19,24,25]; and rivers in Germany and Italy ( $0.1\text{--}0.25 \mu\text{g l}^{-1}$ ) [26].

### 3.3. MTBE concentrations in sewage water

In order to investigate the sources of MTBE in the river waters, sewage water samples were collected from two sewage plants in Niigata Prefecture. Table 1 shows the results of MTBE analyses in the water samples from the two sewage plants. The amount of MTBE found in sewage water samples ranged from  $<0.003$  to  $0.025 \mu\text{g l}^{-1}$ . The standard deviations of the MTBE levels found in the samples from inflow and outflow waters (Table 1) suggest that no significant differences were observed between the amounts of MTBE recov-

ered from inflow versus outflow waters ( $p < 0.05$ ). The levels of MTBE found in these water samples were relatively low, suggesting that sewage water from plants does not play an important role in the level of MTBE in river waters in Niigata Prefecture. However, if wastewater collected from a stationary source near a river contained MTBE at a level as high as  $30 \mu\text{g l}^{-1}$ , it may contribute to MTBE contamination in river water [16].

### 3.4. MTBE concentrations in the Shinano River

The Shinano River is the largest river in Japan. It flows 36.6 km from Kobushin Peak at the border of the Nagano, Saitama, and Yamanashi prefectures to the Sea of Japan at the city of Niigata. It is approximately 1/10 the length of the Mississippi River in the USA. The Shinano River basin is one of the largest watersheds in Japan and drains an area of approximately 11900  $\text{km}^2$  in four prefectures. For several centuries, there have been rice fields along the Shinano River. In addition to being used for the generation of electricity, Shinano River water is used for agriculture (80.5%), industry (14.4%), drinking water (3%), and other uses (2.1%). There are many water inlets from filtration plants for the drinking waters. Also, a large amount of wastewater and sewage water flows into the Shinano River from villages, towns, and cities located near the river.

Water samples were collected once a month from six sites (Sites 5–10) along the Shinano River from January to December 2002. MTBE was found in all samples except the ones collected from Site 7 in April–July, and September; and from Sites, 8, 9, and 10 in April–September. The levels of MTBE ranged from 0.003 to  $0.46 \mu\text{g l}^{-1}$  in samples from Sites 5–10. This range of levels is consistent with those found in the other sites except one site, at which the level was higher than  $1.0 \mu\text{g l}^{-1}$  (Fig. 2). The frequencies of MTBE appearance in the sites were 12/12 (100%) in Site 5; 12/12 (100%) in Site 6; 7/12 (58.3%) in Site 7; 6/12 (50%) in Site 8; 6/12 (50%) in Site 9; and 6/12 (50%) in Site 10.

Fig. 3 shows the levels of MTBE in the samples obtained from Sites 5, 7, 8, 9, and 10. Values from the samples from Site 6 were excluded in Fig. 3 because some samples from Site 6, which is located on the down course of the river, contained MTBE at levels higher than  $0.10 \mu\text{g l}^{-1}$ . Relatively

Table 1  
MTBE concentrations in inflows and outflows from two sewage plants

Concentration ( $\mu\text{g l}^{-1}$ )	Plant 1				Plant 2			
	Winter		Summer		Winter		Summer	
	Inflow	Outflow	Inflow	Outflow	Inflow	Outflow	Inflow	Outflow
Highest	0.025	0.015	0.013	0.011	0.013	0.016	0.010	0.007
Lowest	0.008	0.011	0.003	0.004	0.006	0.005	0.003	0.003
Mean <sup>a</sup>	0.013	0.013	0.007	0.008	0.009	0.009	0.006	0.005
SD <sup>b</sup>	0.004	0.002	0.002	0.002	0.003	0.002	0.002	0.001

<sup>a</sup> Mean of 24 samples.

<sup>b</sup> Standard deviation.

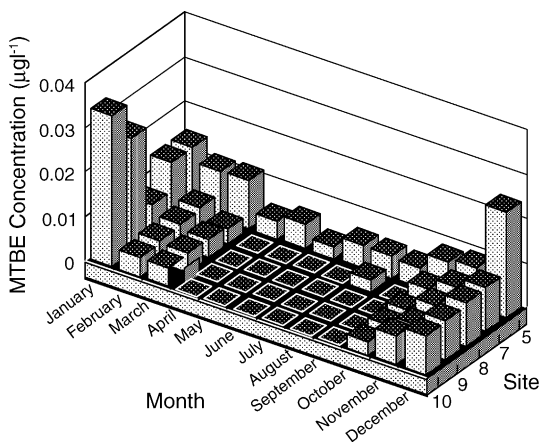


Fig. 3. Monthly variations of MTBE levels at the sites on the Shinano River.

high levels (0.02–0.034  $\mu\text{g l}^{-1}$ ) of MTBE were found in January at Sites 9 and 10, which are located on the upper course of the river. Aside from these two exceptions, generally the samples collected on the down course of the river (Sites 5 and 7) contained higher levels of MTBE than those collected on the upper course of the river (Sites 9 and 10). This trend was consistent with the findings from a similar study done on the Rhine River, Germany [20]. Fig. 3 indicates that MTBE levels were generally higher during winter than summer. The same trend was observed in the seven New Jersey streams, USA [24].

Fig. 4 shows the monthly variations of MTBE levels in the samples collected at Site 6. The levels of MTBE ranged from 0.005 (mid February) to 0.46  $\mu\text{g l}^{-1}$  (April). The average level of MTBE at this site was 0.11  $\mu\text{g l}^{-1}$  and the median value was 0.068  $\mu\text{g l}^{-1}$ . There was no significant difference between the levels of MTBE in the winter and the summer at Site 6. Relatively high levels of MTBE (over 0.10  $\mu\text{g l}^{-1}$ ) were found inconsistently throughout the year (January, March, April, July, and December) at Site 6. The distribution pattern of MTBE at Site 6 was somewhat different from those at the other four sites (5, 7, 8, 9, and 10) in the

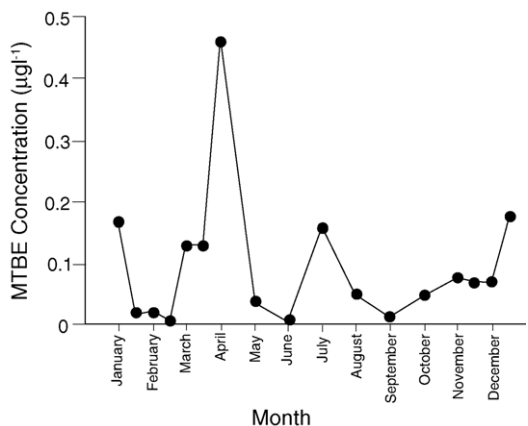


Fig. 4. Monthly variations of MTBE levels at Site 6.

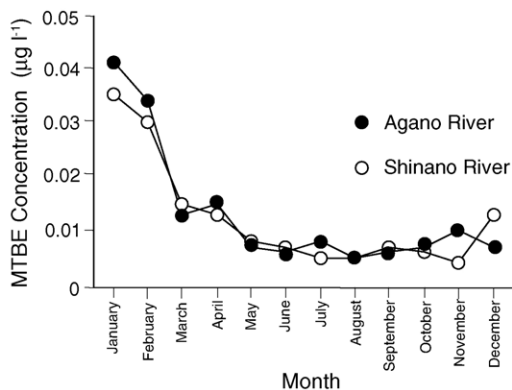


Fig. 5. Monthly variations of MTBE levels at the mouth of the Shinano River and at the mouth of the Agano River.

Shinano River. The factors responsible for the difference are not clear, although there is a sewage plant near Site 6.

Fig. 5 shows the monthly variations of MTBE levels in the samples collected at the mouths of the Shinano River (Site A) and the Agano River (Site B) (refer to Fig. 1). MTBE levels ranged from 0.004 to 0.035  $\mu\text{g l}^{-1}$ , and from 0.005 to 0.041  $\mu\text{g l}^{-1}$ , at the mouths of the Shinano River and Agano River, respectively. The highest levels were obtained in the samples collected in January. Generally, MTBE levels at the mouths of the two rivers were higher in winter than in summer.

A water sample collected from a river where a boat landing contained a relatively high level of MTBE (3.2  $\mu\text{g l}^{-1}$ ) [18]. In the present study, there were no boat landings near the sites where relatively high levels of MTBE were found. However, there are several chemical plants near the rivers, which may contribute to the levels of MTBE in the river waters.

### 3.5. MTBE concentrations in groundwater

Fig. 6 shows the number of sites where MTBE was found at each range of levels. Detectable levels of MTBE were found at 22 sites in 13 cities. Among the 66 sites in the present study, 44 sites did not have detectable levels of MTBE

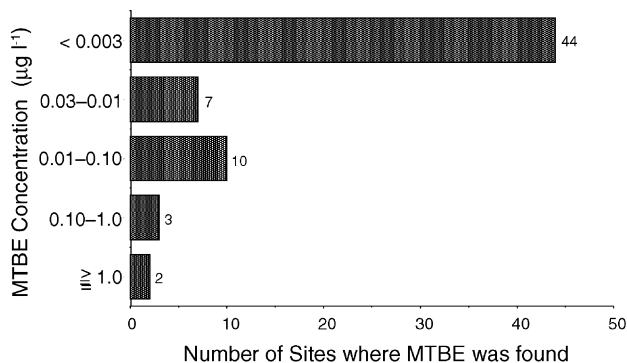


Fig. 6. Number of sites where MTBE was found at various levels in ground waters.

(<0.003). MTBE levels ranged from 0.003 to 5.9  $\mu\text{g l}^{-1}$ . The average value was 0.35  $\mu\text{g l}^{-1}$  and the median value was 0.013  $\mu\text{g l}^{-1}$ . The number of groundwater samples in which MTBE level was less than the detectable level was more than that of river water samples. Generally, the MTBE levels in groundwater were similar to those in river waters.

According to the report on MTBE levels in groundwater (wells) collected near gas stations by the Japan Ministry of Environment in 2000 [27], the levels of MTBE found in 36 sites out of 196 sites ranged from 0.0034 to 0.27  $\mu\text{g l}^{-1}$ . These values are consistent with the ones found in the present study. However, the exact sources of MTBE in the groundwater analyzed in the present study are not clear.

#### 4. Conclusions

A purge-and-trap-GC-MS method for trace MTBE analysis in environmental water samples was successfully improved using MTBE- $d_{12}$  as an internal standard. Optimum conditions for a purge-and-trap process were established. This method was validated using water samples collected from various rivers, ground waters, and sewage waters in Niigata Prefecture, Japan.

There was some seasonal variation in MTBE levels in the rivers in Niigata Prefecture. It was found that sewage water from plants did not play an important role in the level of MTBE in river waters in Niigata Prefecture. The exact sources of MTBE contamination in the rivers are not yet clear. Generally, the MTBE levels in groundwater were similar to those in river waters. The levels of MTBE found in water samples in the present study were consistent with the values reported previously.

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