WATER QUALITY GUIDELINES
FOR METHYL TERTIARY-
BUTYL ETHER (MTBE)

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EXECUTIVE SUMMARY

This report provides methyl tertiary-butyl ether (MTBE) water quality guidelines for the province of British Columbia. Human drinking water guidelines were developed based on a summary of guidelines from other jurisdictions. Freshwater aquatic life, marine aquatic life, irrigation, and livestock watering guidelines were developed using toxicological effects data and protocols from the Canadian Council for Ministers of the Environment.

MTBE is a gasoline additive that is volatile in the atmosphere and mobile in groundwater. The primary source of MTBE in the environment is from leaking underground storage tanks containing reformulated gasoline. Automobile fuel blended with an oxygenate additive such as MTBE is known as reformulated gasoline. Laboratory microcosms, field-scale tests, and empirical site data have shown that MTBE biodegrades slowly in both aerobic and anaerobic environments.

Proposed MTBE water quality guidelines are summarized in Table 2 in this report. The human drinking water guideline (0.02 mg/L) was based on taste and odour. Recreational water quality guidelines from other jurisdictions were not found and, thus, this guideline was set at the human drinking water value. Freshwater aquatic life, marine aquatic life, and livestock watering guidelines were 3.4 mg/L, 0.44 mg/L, and 11 mg/L, respectively. No studies were available on the toxicity of MTBE to terrestrial plants and, therefore, an irrigation guideline could not be developed.

Collection of water samples for MTBE analysis should follow protocols used for volatile organic compounds. GC/PID can be used as a screening method. However, this method is prone to false positives and interference problems. Should a detection occur using GC/PID, confirmatory analysis by GC/MS would be required. GC/MS is the recommended methodology for MTBE analysis.
1. INTRODUCTION

This report presents water quality guidelines developed for methyl tertiary-butyl ether (MTBE) for the province of British Columbia. The work was completed by Komex International Ltd., under contract #WMB01-032 (the Contract), to the British Columbia Ministry of Environment, Lands & Parks, Water Management Branch.

1.1 Background

The 1990 Clean Air Act Amendment in the United States required fuel oxygenates to be added to gasoline in some metropolitan areas to reduce atmospheric concentrations of carbon monoxide, carbon dioxide, and ozone. MTBE is the most commonly used fuel oxygenate because of its low cost, ease of production and transfer, and blending characteristics (e.g., Mormile et al., 1994). MTBE has become an important groundwater contaminant because of its mobility (Garrett et al., 1986; Environment Canada, 1993; Davidson, 1995), recalcitrant nature (Moller and Arvin, 1990; Yeh and Novak, 1991; Mormile et al., 1994), and potential toxicity (USEPA, 1993; ATSDR, 1994; HEI, 1996).

The use of MTBE has become controversial, due primarily to issues with contaminated groundwater supplies. California banned the use of MTBE, effective 31 December 2002. MTBE has been used in British Columbia since 1995.

1.2 Scope of Work

The scope of work for this project, detailed in the Contract, was to:

- identify, review, describe, and assess all available literature related to MTBE in raw waters (i.e., causes of release, health effects, guidelines, and other potentially useful information);
- review analytical methods for the analysis of MTBE in water and propose a methodology that achieves the detection limit, accuracy, and precision required for compliance with the recommended guidelines;
- recommend sampling protocols and other monitoring considerations that may be pertinent;
- compile and recommend guidelines from other jurisdictions to use in the assessment of MTBE in drinking water and recreational waters;
- develop scientifically-based water quality guidelines to protect water quality in BC, for uses including freshwater and marine aquatic life, irrigation, and livestock watering;
- provide guidance on research needs and data gaps; and,
- prepare a technical report using a similar format to other criteria/guidelines in BC.
1.3 Protocols

Environmental quality guidelines for MTBE were developed using the following protocols developed by the Canadian Council of Ministers of the Environment (CCME):


For ease of reference in this document, the phrase “the Protocol” refers to whichever of the above documents is applicable. Note that the two water Protocols listed above (Aquatic Life, and Agricultural Water Uses) were originally published as CCME (1991), and CCME (1993), respectively, and were reproduced with minor changes in CCME (1999).

The above Protocols define two types of guidelines. Full guidelines reflect a dataset which is of high quality and sufficiently complete to allow a high degree of confidence in the resulting guideline. Interim guidelines are derived from a dataset which was not sufficient to allow derivation of a Full guideline, but conformed to a less strict set of requirements. For the purposes of this document, a third type of guideline was introduced. “Preliminary” guidelines are derived from a dataset that did not meet all the requirements for Interim guidelines. Preliminary guidelines were derived where it was felt that a guideline derived from a limited dataset was more useful than no guideline at all. Once sufficient toxicological data become available, it is anticipated that preliminary guidelines will be updated to Interim or Full status.

1.4 Toxicity Data Search

An extensive literature search was conducted to identify toxicity data for MTBE to mammals, marine and freshwater aquatic life, terrestrial invertebrates, plants, and other organisms. Databases and other information sources searched included Biological Abstracts, TOXLINE, AGRICOLA, ECOTOX, MEDLINE, Uncover Web, SETAC, and Springer Link. Keywords used for the search were “methyl tertiary-butyl ether and MTBE”.

2. METHYL TERTIARY-BUTYL ETHER IN THE ENVIRONMENT

2.1 Production and Uses

There are no natural sources of MTBE. There was no production in Canada before 1992. Production began in 1992 by Alberta Envirosfuels, Inc., Edmonton, Alberta, with an estimated 500,000 tonnes per year (Solsberg, 1991). Most of the MTBE produced was shipped by rail from Edmonton to Kitimat, BC and then by ocean tanker to US.

Canadian importation of MTBE for the purpose of blending with unleaded gasoline from 1986 to 1990 was between 7000 and 25,000 tonnes per year. Over the past six years MTBE has been used at one time or another in all areas of Canada. If and when it was used it was blended to a maximum 15% volume in premium and super premium grades. Because some mid-grade products are a blend of 50% premium and 50% regular they too could have contained MTBE if and when MTBE was blended. Based on preliminary information from the fourth quarter 1999, Federal "Benzene in Gasoline Report" the average concentrations of MTBE in Canada is 0.35 vol.%. approximately one-tenth that in the USA.

The last CPPI member company to use MTBE in gasoline in Western Canada ceased doing so, including all gasoline sold or exchanged to others, as of October 2000. No companies have plans to again use MTBE in gasoline in Western Canada.

2.2 Chemical and Physical Properties

MTBE (CAS Registry Number 1634-04-4) is an aliphatic ether. Representative chemical and physical properties of MTBE are summarized in Table 1. Synonyms include:

- 2-methoxy-2-methylpropane;
- 2-methyl-2-methoxypropane;
- Methyl 1, 1-dimethylethyl ether; and,
- tert-butyl methyl ether.

MTBE is volatile (vapour pressure is 33 kPa at 25°C) and has a high aqueous solubility (pure phase ~40,000 mg/L). MTBE has a specific gravity of 0.74 (20/4 °C). The organic carbon partition coefficient (log Koc) for MTBE is approximately 0.94 (Table 1). This indicates little adsorption of MTBE to aquifer material, which is consistent with field observations. Thus, little to no attenuation of MTBE is expected in a saturated medium with typical organic carbon contents. The octanol/water partition coefficients (log Kow) for MTBE is approximately 1.6 (Table 4). This indicates that MTBE is not likely to accumulate in human or animal fat tissue.
2.3 Organoleptic Thresholds

MTBE is reported to have a distinctive, terpene-like odour in air (Gilbert and Calabrese, 1992). A number of different studies have reported taste and order thresholds for MTBE (Table 1).

The American Petroleum Institute taste and odour thresholds of approximately 39 ppb and 53 ppb, respectively (API, 1993). Young et al. (1996) reported taste and odour thresholds of 40 ppb and 15 ppb, respectively. The Orange County Water District in California performed a study on threshold odor concentrations of MTBE. That study, reported in Shen et al. (1997), found geometric means of 13.5 to 43.5 ppb, indicating that half of the panelists detected MTBE at those levels. Shen et al. (1997) also demonstrated that MTBE odour may be detected at levels as low as 2.5 ppb. However, as a lower concentration was not tested, it is possible that the odour threshold could be less than 2.5 ppb for some individuals.

2.4 Sources of MTBE in the Environment

A major source of MTBE in the environment is from leaking underground storage tanks containing reformulated gasoline (Squillace et al., 1996). Reformulated gasoline commonly contains between ~10% to 15% MTBE (volume/volume). MTBE is used by some gasoline retailers in Canada and has been used by some gasoline retailers in British Columbia since 1995. Another localized source of MTBE in the environment is atmospheric release from refining/blending facilities (NPRI, 2000).

The United States Geological Survey initiated a national survey in 1991 of groundwater quality under the National Water Quality Assessment (NAWQA) program (Squillace et al., 1996). This survey, which focused on the occurrences and possible sources of MTBE in groundwater, showed that 27% of the shallow wells sampled in urban areas had MTBE concentrations >0.0002 mg/L and that 1.3% had concentrations that exceeded 0.020 mg/L.

In a recent survey of groundwater wells in Washington state, MTBE was identified in 30 of 62 sites (48 percent). Fifteen of the sites had MTBE concentrations greater than the USEPA's national drinking water advisory level of 0.020 mg/L.

In November 2000, BC MELP analyzed MTBE in water samples collected at 59 groundwater wells. MTBE was detected in one sample.

Environment Canada’s Aquatic Sciences Section participated in a survey with the US Geological Survey in 1997 of the shallow Abbotsford-Sumas Aquifer, which is located in southwestern BC, near the US border. The survey tested for some 200 chemicals and found trace amounts of MTBE.
in a section of the aquifer; specifically, in 2 of 9 wells tested over a 10 km section of the aquifer. The levels were below the quantification limits of the instruments used (USGS, 1999).

2.5 Biodegradation

Laboratory microcosms, field-scale tests, and empirical site data have shown that MTBE biodegrades slowly in both aerobic and anaerobic environments (Yeh and Novak, 1995; Sulflita and Mormile, 1993; Mormile et al., 1994). The poor biodegradation of MTBE has been attributed to the stable and unreactive ether bond, the inability of MTBE to be transported into cells, and/or the lack of existing enzyme activities that can attack the ether bond (Salinitro et al., 1994). The main breakdown products of MTBE biodegradation include formaldehyde and tertiary-butyl alcohol (e.g., Clary, 1997 and references therein).

With respect to development of water quality guidelines for freshwater and marine aquatic life, MTBE can be considered a “persistent variable” because it has a biodegradation half-life of greater than 8 weeks. Howard et al. (1991) report a biodegradation half-life ranging from four weeks to six months for the surface water environment.

3. WATER GUIDELINES FOR METHYL TERTIARY-BUTYL ETHER

The water quality guidelines for MTBE developed below are summarized in Table 2. Human drinking water guidelines were developed based on a summary of guidelines from other jurisdictions. Freshwater aquatic life, marine aquatic life, irrigation and livestock watering guidelines were developed using applicable CCME protocols and toxicity data. Details of the Protocol data requirements in terms of data quality and quantity are discussed below.

3.1 Water Guidelines From Other Jurisdictions

3.1.1 Drinking Water

3.1.1.1 Review of Available Information

A review of information from regulatory agencies worldwide was completed to determine what guidelines were being used by other jurisdictions for acceptable levels of MTBE in drinking water. Water quality guidelines have not been developed by the Canadian federal government or provincial regulators. The only drinking water quality guideline found for Canada was in Ontario,
which reports a value of 0.70 mg/L in "Guideline for use at Contaminated Sites". This guideline does not consider aesthetics due to low odor and taste thresholds for MTBE.

Drinking water guidelines for federal and state jurisdictions in the U.S. are summarized in Table 3. Three main endpoints have been used by various jurisdictions in guideline development: 1) taste and odour; 2) health effects, considering MTBE as a carcinogen; and 3) health effects, considering MTBE as a non-carcinogen. These approaches are discussed further below.

The United States Environmental Protection Agency (USEPA, 1997) has issued a drinking water advisory for MTBE. This document recommends a range of 0.02 mg/L to 0.04 mg/L based on taste and odour thresholds. Thirteen American states have a guideline that is either based, or assumed to be based, on the taste and odour threshold. Most of these states use a value within, or close to, the range given by the USEPA (1997) (Table 3). California has issued a secondary maximum contaminant level of 0.005 mg/L, to protect the taste, odour and/or appearance of drinking water (Ca DHS, 2001).

Six states have calculated health-based guidelines considering MTBE a carcinogen (Table 3). New York and California calculate the most conservative guidelines (0.010 mg/L, and 0.013 mg/L, respectively). Maine, Massachusetts, New Hampshire, and New Jersey calculate guidelines that range from 0.035 mg/L to 0.070 mg/L.

Six states have calculated health-based guidelines by considering MTBE a non-carcinogen. The guidelines calculated by these states range from 0.035 mg/L (Arizona) to 0.52 mg/L (Louisiana) (Table 3). Other jurisdictions either have not made the basis of their guideline available, or have not adopted a guideline at the present time (Table 3).

3.1.1.2 Recommended Guideline

The health-based guidelines discussed above used a variety of methodologies to derive a drinking water guideline from a number of toxicological studies. Only two states, New York and California calculated guidelines (0.010 mg/L, and 0.013 mg/L, respectively) that were below the lower end of the taste and odour threshold (0.020 mg/L) published by USEPA (1997).

The health-based drinking water guideline was recalculated using receptor parameters from the Canadian Council for Ministers of the Environment, the California Environmental Protection Agency cancer slope factor (0.0018 (mg/kg-d)^{-1}), and assuming a lifetime exposure weighted for five human life stages and a maximum acceptable excess cancer risk of 1 in 1,000,000. The recalculated health-based MTBE human drinking water guideline is 0.021 mg/L.
Based on procedures and parameters normally used in British Columbia, the most sensitive endpoint for drinking water is the taste and odour threshold. It is therefore recommended that the lower end of the range for taste and odour threshold published by USEPA (1997) be adopted, and that the drinking water guideline be set at 0.020 mg/L. This guideline is identical to the health-based drinking water guideline protective of the public for a maximum acceptable excess cancer risk of 1 in 1,000,000.

3.1.2 Recreational Uses

No guidelines for acceptable levels of MTBE in water for recreational uses were found from any jurisdictions. It is proposed that the recreational water guideline be equivalent to the drinking water guideline (i.e., 0.020 mg/L), based on the taste and odour threshold.

Consideration of multiple routes of exposure (i.e., ingestion, inhalation, and dermal absorption) was not given in proposing a recreational water guideline equivalent to the drinking water guideline. However, the exposure frequency in a recreational setting is considerably lower than a domestic setting. For this reason, the drinking water guideline (i.e., 0.020 mg/L) will be fully protective of recreational exposure.

3.2 Freshwater Aquatic Life Guideline

3.2.1 Toxicological Behaviour and Effects

Freshwater aquatic data are summarized in Table 4. Four main groups of freshwater aquatic biota are identified (vertebrates, invertebrates, plants, and other biota). The data for each are discussed in the following Sections.

3.2.1.1 Vertebrates

A total of 23 data points were available (Table 4) from 13 studies for 6 species of freshwater aquatic vertebrates: rainbow trout (Oncorhynchus mykiss), fathead minnow (Pimephales promelas), Japanese medaka (Oryzias latipes), common frog (Rana temporaria), carp (Leuciscus idus melanotus), and bluegill (Lepomis macrochirus). Test durations varied from 1 to 7 days, and thus would be considered acute studies, since the test duration is not a significant fraction of the lifespan of the organism. Endpoints were mostly survival, with the exception of two tests that considered growth/development endpoints. Reported LC50 values ranged from 672 mg/L (fathead minnow) to 2,500 mg/L (frog). The LOEC for fathead minnow growth was 388 mg/L.
3.2.1.2 Invertebrates

Seven studies considered the toxicity of MTBE to 4 species of aquatic invertebrates (Table 4): two species of water fleas (*Daphnia magna* and *Ceriodaphnia dubia*), and a rotifer (*Brachionus calyciflorus*). The majority of the data were from acute lethality tests, with durations ranging from 24 hours to 96 hours. Reported LC50 values for these tests ranged from 340 mg/L (*C. dubia* 48-hour survival) to 1,118 mg/L (*D. magna* 24-hour survival). Chronic studies considered reproduction and survival in *C. dubia*. The lowest LOEC reported for a chronic study was 342 mg/L (*C. dubia* 5-day reproduction).

3.2.1.3 Plants

Two studies (Table 4) considered the toxicity of MTBE to the green alga *Selenastrum capricornutum*. Rousch and Sommerfeld (1998) report a LOEC of 4,789 mg/L for a growth endpoint at 5 days in this species. Ben Kinney *et al.* (1994; as cited in U.C. Davis, 1998), reported an EC50 of 184 mg/L for an unknown endpoint. However, the original study was not available to determine the endpoint and the study’s relevance.

3.2.1.4 Other Biota

Other aquatic biota include all aquatic organisms not included in the animal or plant kingdoms. This covers organisms from the kingdoms Monera, Protista and Fungi. Rousch and Sommerfeld (1998) report a LOEC of 2,868 mg/L for a growth endpoint at 3 days in the diatom *Navicula pelliculosa*, and a LOEC of 2,489 mg/L for a growth endpoint at 5 days in the cyanobacteria *Synechococcus leopoliensis* (Table 4). Kado *et al.* (1998) report a 48 hour growth LOEC of 7.4 mg/L for *Salmonella typhimurium*. However, *S. typhimurium* is not a typical freshwater species, and thus, was not included in guideline development.

3.2.2 Data Analysis

Freshwater aquatic life guidelines were developed using the Protocol (“A Protocol for the Derivation of Water Quality Guidelines for the Protection of Aquatic Life; CCME, 1999). This Section summarizes the requirements of the Protocol and discusses the available dataset in terms of these requirements. The toxicological dataset was summarized in Table 4, and discussed above.

The Protocol defines: (1) the requirements for a toxicological study to be acceptable for guideline derivation (data quality); (2) the minimum required dataset for Full and Interim guideline development (data quantity); and (3) the process for deriving guidelines. The following
paragraphs provide a summary of the requirements of the Protocol, and assess the toxicological dataset.

3.2.2.1 Data Quality

The data quality requirement in the Protocol may be summarized as follows. For a toxicological study to be considered “Secondary Data”, all relevant environmental variables (e.g., temperature, pH, hardness, dissolved oxygen, etc.) should be measured and reported, and the survival of controls must be reported. In addition, for data to be considered “Primary Data”, tests must employ currently acceptable practices, concentrations must be measured at the beginning and end of a test, and, in general, dynamic (i.e., flow-through) tests are required. Data that do not conform to the requirements for Primary or Secondary Data are “Unacceptable Data”.

The toxicological dataset is summarized in Table 4 and classified as Primary, Secondary, and Unacceptable. It should be noted that studies classified as “Unacceptable Data” may, in fact, represent acceptable (i.e., Primary or Secondary) data, but insufficient information was available to confirm this. According to the Protocol only Primary or Secondary Data can be used in the derivation of Interim guidelines.

3.2.2.2 Data Quantity

The minimum requirements for an Interim freshwater aquatic life guideline is as follows. At least two studies on freshwater fish species, and at least two studies on freshwater invertebrate species are required. The tests may be acute or chronic. One of the fish must be a cold water species, and two different classes of invertebrates must be represented, one of which includes a planktonic species resident in North America (e.g., daphnid).

The Protocol requirements for the Interim guidelines were met by the Primary and Secondary Data in Table 4. The acute tests on rainbow trout and fathead minnow fulfill the requirement for tests of two freshwater fish species, with the rainbow trout fulfilling the requirement for a cold water species. Acceptable test results were available for three species of invertebrate: *Daphnia magna* and *Ceriodaphnia dubia*, representing the class Branchiopoda of the phylum Arthropoda and *Brachionus caliciflorus*, representing a class of the phylum Rotifera. Accordingly, the requirement for data from two species of invertebrates from different classes was met.

3.2.3 Guideline Derivation

The Protocol defines procedures for deriving guidelines from chronic or acute data. Guidelines were calculated from both acute and chronic data, and the lower value was adopted as the freshwater aquatic life guideline. A guideline can be calculated from chronic data, by using the
lowest LOEC from the most sensitive endpoint of the most sensitive lifestage of the most sensitive species, multiplied by a safety factor of 0.1 to give the freshwater aquatic life guideline. The lowest chronic LOEC for Primary or Secondary Data in this dataset is 342 mg/L for the 5 day reproduction endpoint for *Ceriodaphnia dubia*. This yields a guideline value of 34 mg/L.

A guideline can also be calculated from acute data, by using the lowest LC50 or EC50 value, and multiplying by an “application factor” of 0.01 for persistent variables. MTBE is assumed to have a biodegradation half-life of greater than 8 weeks, and thus is considered a persistent variable as discussed in Section 2.3. The lowest LC50 in the acute dataset is 340 mg/L for 48 hour *C. dubia* survival (Hockett, 1997b). Multiplying this value by an application factor of 0.01 gives a guideline of 3.4 mg/L. This value is lower than the guideline calculated from the chronic dataset, and thus the Interim freshwater aquatic guideline for MTBE is 3.4 mg/L (Table 2).

### 3.3 Marine Aquatic Life Guideline

#### 3.3.1 Toxicological Behaviour and Effects

Marine aquatic data are summarized in Table 5. Four main groups of marine aquatic biota are identified (vertebrates, invertebrates, plants, and other biota). The data for each are discussed in the following Sections.

**3.3.1.1 Vertebrates**

A total of 4 data points were available (Table 5) from 4 studies for 3 species of marine aquatic vertebrates: sheepshead minnow (*Cyprinodon variegatus*), bleak (*Alburnus alburnus*), and inland silverside (*Menidia beryllina*). Test durations varied from 1 to 4 days, and thus were considered acute, based on the test duration not being a significant fraction of the lifespan of the organism. All tests considered the survival endpoint. Reported LC50 values ranged from 574 mg/L (inland silverside) to >2,500 mg/L (sheepshead minnow).

**3.3.1.2 Invertebrates**

Six studies considered the toxicity of MTBE to 4 species of marine aquatic invertebrate (Table 5): two specific opossum shrimp (*Neomysis mercedis* and *Mysidopsis bahia*), a copepod (*Nitocra spinipes*), and an amphipod (*Chaetogammarus marinus*). All the data were acute lethality tests, with test durations being 96 hours except for one test where this information was not available. Reported LC50 values for these tests ranged from 44 mg/L (*M. bahia* 96 hour survival) to >10,000 mg/L (*N. spinipes*, 96 hour survival).
3.3.1.3 Plants

No toxicity data for marine aquatic plants were available.

3.3.1.4 Other Biota

Other aquatic biota include all aquatic organisms not included in the animal or plant kingdoms. This covers organisms from the kingdoms Monera, Protista, and Fungi. The only toxicity data for other biota were for marine bacterium Photobacterium phosphoreum (Microtox™). Gupta and Lin (1995) reported a 15 minute EC50 value of 31 mg/L.

3.3.2 Data Analysis

Marine aquatic life guidelines were developed using the Protocol (“A Protocol for the Derivation of Water Quality Guidelines for the Protection of Aquatic Life; CCME, 1999). This Section summarizes the requirements of the Protocol and discuss the available dataset in terms of these requirements. The toxicological dataset was summarized in Table 5, and discussed above.

The Protocol defines: (1) the requirements for a toxicological study to be acceptable for guideline derivation (data quality requirement); (2) the minimum required dataset for Full and Interim guideline development (data quantity requirement); and (3) the process for deriving guidelines. The following paragraphs provide a summary of the requirements of the Protocol, and assess the toxicological dataset.

3.3.2.1 Data Quality

The data quality requirement in the Protocol may be summarized as follows. For a toxicological study to be considered “Secondary Data”, all relevant environmental variables (e.g., temperature, pH, hardness, dissolved oxygen, etc.) should be measured and reported, and the survival of controls must be reported. In addition, for data to be considered “Primary Data”, tests must employ currently acceptable practices, concentrations must be measured at the beginning and end of a test, and, in general, dynamic (i.e., flow-through) tests are required. Data that do not conform to the requirements for Primary or Secondary Data are “Unacceptable Data”.

The toxicological dataset was summarized in Table 5 and data were classified as Primary, Secondary, and Unacceptable. It should be noted that studies classified as “Unacceptable Data” may, in fact, represent acceptable (i.e., Primary or Secondary) data, but insufficient information was available to confirm this. According to the Protocol only Primary or Secondary Data can be used in the guideline derivation process.
3.3.2.2 Data Quantity

The Protocol requirement for the quantity of data needed for Interim marine aquatic life guidelines may be summarized as follows. At least two studies on marine fish species, and at least two studies on marine invertebrate species are required. The tests may be acute or chronic. One of the fish must be a temperate species, and two different classes of invertebrates must be represented, one of which is a temperate species.

The Protocol requirements were not met by the Primary and Secondary Data in Table 5, as only one fish species is represented, and the two invertebrate species are in the same class. Accordingly, no CCME guideline can be calculated for marine aquatic life for MTBE. However, it may be useful to have a preliminary marine aquatic guideline until further data can be collected, or further information for existing data becomes available. Accordingly a preliminary guideline was calculated, based on the Primary, Secondary, and Unacceptable data in Table 5. Note that the Microtox™ data were not included in the guideline derivation process, as they may not represent a typical toxicological response in marine aquatic life.

If the Primary, Secondary, and Unacceptable data from Table 5 are used, then the Protocol data quantity requirements can be met. The fish data requirement was met by three species of fish (i.e., sheepshead minnow, bleak, and inland silverside). The Protocol requires that at least one fish is a temperate species. The sheepshead minnow is native to eastern U.S. coastal waters as far north as Massachusetts, and so would be considered temperate. The invertebrate data requirement was met by four species of invertebrates from two classes (Maxillopoda and Malacostraca) of the phylum Arthropoda. The Protocol requires at least one temperate marine invertebrate species. The mysid Neomysis mercedis is native to Californian coastal waters, and so would be considered temperate.

3.3.3 Guideline Derivation

The data quantity requirements of the Protocol could be met only by including the Unacceptable data in Table 5. The Protocol methodology was used with this extended dataset to calculate a “Preliminary” marine aquatic life guideline. The Protocol defines procedures for deriving guidelines from chronic or acute data. Only acute data were available in the marine aquatic dataset.

According to the Protocol, a guideline can be calculated from acute data by using the lowest LC50 or EC50 value, and multiplying by an “application factor” of 0.01 for persistent variables. The lowest LC50 in the acute dataset is 44 mg/L for Mysidopsis bahia survival (Boeri et al., 1994). Multiplying this value by an application factor of 0.01 gives a Preliminary guideline of 0.44 mg/L (Table 2).
3.4 Irrigation Guideline

No studies were available on the toxicity of MTBE to terrestrial plants. Accordingly, no irrigation guideline for MTBE could be developed.

3.5 Livestock Watering Guideline

3.5.1 Toxicological Behaviour and Effects

Available data on the toxicity of MTBE to mammalian species is summarized in Table 6. No data on the toxicity of MTBE to livestock species was available. The mammalian toxicity data available for MTBE uses laboratory animals (rats, mice, and rabbits) as test species.

3.5.2 Data Analysis

Livestock watering guidelines were developed using the Protocol (“Protocols for Deriving Water Quality Guidelines for the Protection of Agricultural Water Uses, CCME, 1999). This Section summarizes the requirements of the Protocol and discusses the available dataset in terms of these requirements. The toxicological dataset was summarized in Table 6.

Protocol requirements for Interim guidelines include toxicological data for at least two studies on two or more mammalian species raised in Canada, including at least one livestock species, and for at least one study on one or more avian livestock species raised in Canada. The dataset summarized in Table 6 does not include any data on livestock or avian species. Accordingly, the data quantity requirements of the Protocol are not met, and no CCME livestock watering guideline can be derived.

It may, however, be useful to have a Preliminary livestock watering guideline until sufficient livestock toxicological data become available. Accordingly, the most applicable study from Table 6 was used and a guideline was derived based on the methodology in the Protocol.

A livestock watering guideline addresses the issue of chronic ingestion of a contaminant. Accordingly the Robinson et al. (1990) study was considered to be the most applicable toxicological study as it used an oral route of exposure (gavage), and was the longest duration (90 days) of such studies. This study has been used by several jurisdictions in the United States as the basis of their human health drinking water criteria (CT DEP, 2000; MA DEP, 1995; NH DHHS, 2000).
3.5.3 Guideline Derivation

A Preliminary livestock watering guideline was developed using toxicological data from Robinson et al. (1990) and methodology from the Protocol. The Robinson et al. (1990) study was subchronic because of its 90-day duration.

The first step in the guideline derivation process outlined in the Protocol is the Tolerable Daily Intake (TDI) calculation (CCME, 1999):

\[
TDI \ (mg \ / \ kg - bw/day) = \left( \frac{LOAEL \cdot NOAEL}{UF} \right)^{0.5}
\]

Where:
- TDI = tolerable daily intake (1.73 mg/kg-bw/day);
- LOAEL = lowest adverse effect level (300 mg/kg-bw/day, Table 6);
- NOAEL = no adverse effect level (100 mg/kg-bw/day, Table 6); and,
- UF = uncertainty factor (100; see discussion below).

The 100-fold uncertainty factor was based on a 10-fold factor to extrapolate from subchronic data to chronic, and a 10-fold factor to extrapolate from rodent data to livestock. Based on the above equation, the TDI for MTBE applicable to livestock is 1.73 mg/kg-bw/day.

The next step in the guideline derivation process was to calculate the reference concentration (RC), which represents the Livestock Watering Guideline. The reference concentration is calculated using the body weight and water ingestion rate of a particular species. Dairy cattle were selected to represent livestock, based on a high water consumption relative to their body weight. The equation used was:

\[
RC (mg/L) = \left( \frac{TDI \times BW}{WIR} \right)
\]

Where:
- RC = reference concentration (10.91 mg/L; dairy cattle);
- TDI = tolerable daily intake (1.73 mg/kg-bw/day; calculated above);
- BW = body weight (862 kg for dairy cattle; CCME, 1999); and,
- WIR = daily water intake rate (137 L/day for dairy cattle; CCME, 1999).

The calculated RC for dairy cattle was rounded off to 11 mg/L (Table 2). This value represents a concentration below which livestock health is not expected to be impacted.
The Livestock Watering Guideline determined using published rodent data and algorithms in the Protocol is greater than the taste and odour threshold. While it is known that dairy cattle will drink water with taste and/or odour, it is not known if cattle will tolerate the terpene-like odour of MTBE.

4. DATA GAPS

Data gaps in the guideline development process were identified and are presented in the sections below.

4.1 Freshwater Aquatic Life

The available dataset was sufficient to derive an Interim freshwater aquatic life guideline. In order to derive a Full freshwater aquatic life guideline, the following additional Primary data would be required:

- two chronic studies on two freshwater fish species, one of which was a cold water species (e.g., trout);
- one chronic study on an invertebrate species not from the Class Bronchiopoda (e.g., Daphnia); and,
- one study on a freshwater vascular plant or algal species.

4.2 Marine Aquatic Life

The available dataset was insufficient to derive an Interim marine aquatic life guideline. A Preliminary guideline was derived, based on Primary, Secondary, and Unacceptable data. In order to derive an Interim marine aquatic life guideline, the following additional Primary or Secondary data would be required:

- one acute or chronic study on a marine fish species other than the sheepshead minnow; and,
- one acute or chronic test of an invertebrate of a different class from an opossum shrimp.

4.3 Irrigation

No data were available concerning the toxicity of MTBE to plants. The following Primary or Secondary data would be required to derive an Interim irrigation guideline:

- studies on two species of cereals, tame hays, or pasture crops grown in Canada; and,
• studies on two species of plants grown in Canada including two of the following groups: Leguminosae, Compositae, Cruciferacea, Cucurbitaceae, Liliaceae, Solanaceae, Umbelliferae, and Chenopodiaceae.

4.4 Livestock Watering

The available dataset was insufficient to derive an Interim livestock watering guideline. A Preliminary guideline was derived, based on the most applicable data from the available mammalian laboratory animal studies. In order to derive an Interim livestock watering guideline, the following additional Primary or Secondary data would be required:

• acute or chronic studies on two mammalian species raised in Canada, including at least one livestock species; and,
• one acute or chronic study of an avian livestock species raised in Canada.

4.5 Environmental Monitoring Data

There currently exist limited ambient data for MTBE in British Columbia. Monitoring locations should include high-risk areas such as drinking water reservoirs, municipal drinking water supplies, marinas, and decommissioned or operating gas stations know to have underground storage tanks containing reformulated fuel.

MTBE has been detected in stormwater in other areas. A report by ENCON for Environment Canada on Sources and Releases of Toxic Substances in Wastewaters noted that there was no information on MTBE on stormwater releases to Georgia Basin.
5. ANALYTICAL METHODOLOGY

Analysis of water samples for MTBE requires following appropriate procedures that include sample collection, sample preparation, and analytical methods. USEPA methods have not been developed specifically for the analysis of MTBE. The USEPA and other researchers have published data, however, indicating that MTBE can be measured in environmental samples using one of three SW-846 methods. USEPA methods 8015, 8021, and 8260 have been expanded to include MTBE based on the similarity of chemical and physical characteristics and the common occurrence of MTBE with volatile organic hydrocarbons.

5.1 Sample Collection

Collection of water samples for MTBE analysis are based on protocols established for volatile organic compounds such as benzene. Water collection can be conducted using a dedicated bailer, an inertial pump (e.g., Waterra system), or a peristaltic pump. Samples are collected in clean, laboratory-provided 40 mL screw-cap septum vials. The number of vials required by laboratories for analysis varies between two and three 40 mL vials. Following collection, samples are stored at approximately 4º C (e.g., in an ice-filled insulated cooler) and shipped to the laboratory under standard chain-of-custody documentation.

5.2 Sample Preparation

Sample analysis using USEPA methods are conducted in conjunction with specific preparation procedures. Sample preparation methods applicable for MTBE are summarized in Table 7 (after Rhodes and Verstuyft, 1999). For these methods and a water matrix, chemical sample extraction is not required. Purge-and-trap or headspace methods are appropriate, based on the volatile characteristics of MTBE and volatile organic hydrocarbons. Standard sample preparation methods have been developed for analyte separation using gas chromatography (GC).

Purge-and-trap involves the purging of a water sample with an inert gas such as helium. Volatile compounds are purged from the dissolved phase into the gas phase and are collected in a cryogenic trap attached to a GC. The trap is subsequently heated and the sample injected into the GC column.

Headspace analysis involves removal of a specified volume from a 40 mL vial through the septum. The sample vial is then heated and allowed to equilibrate. A specified volume of the headspace gas is withdrawn using a gas-tight syringe and directly injected into the GC column. This method is based on the equilibrium concentration in the gas and water phase according to Henry’s Law.
5.3 Analytical Methods

USEPA methods 8015, 8021, and 8260 used for the quantification of aromatic and volatile petroleum hydrocarbons and chlorinated hydrocarbons have been expanded to include MTBE. These methods rely on purge-and-trap or headspace gas chromatography but they differ in the selection of detector. Detection methods for MTBE analysis include: 1) flame ionization detector (FID); 2) photoionization detector (PID); and 3) mass spectrometry (MS). USEPA methods are summarized in Table 8. However, none of these methods has been validated by USEPA for MTBE analysis and very little data have been published on the accuracy and precision of these methods (Uhler and Stout, 2000).

Rhodes and Verstuyft (1999) reviewed USEPA methods 8015, 8021, and 8260 for their suitability for MTBE analysis. The three methods were considered to be comparable in terms of sensitivity, accuracy, and precision. Significant differences were determined with respect to their selectivity. Selectivity describes the level of confidence associated with the identification of a compound using a specific detector. A non-selective detector responds universally to many compounds and is prone to false positive results in the presence of interferences.

The FID (USEPA 8015) is considered non-selective. Compound identification using GC/FID is achieved based on retention time comparison between a standard and a sample. Based on the high probability for interferences and co-elution of non-target compounds with MTBE, there is a high probability of false positive results or an overestimation of the MTBE concentration. Thus, the FID has a poor selectivity (Table 8).

The PID (EPA 8021) is selective for aromatic compounds but also responds to other compounds, including MTBE, depending on the ionization potential of the PID lamp. As for the FID method, compound identification using GC/PID is achieved based on retention time comparison between a standard and a sample. While the PID method greatly reduces interferences, the presence of dissolved gasoline compounds (e.g., saturated hydrocarbons and olefins) can lead to false positive results and/or an overestimation of the MTBE concentration. Thus, the PID has a good selectivity (Table 8).

The MS method (USEPA 8260) identifies compounds based on their mass spectra. Mass spectra are unique patterns determined by molecular size and charge that allow a positive compound identification. Therefore, the MS has an excellent selectivity (Table 8) and, as a result, is not prone to false positive results and/or an overestimation of the MTBE concentration.

The USEPA has not published validation data for the performance of this method. Validation data in Table 9 were published by the US Geological Survey (cited in Rhodes and Verstuyft, 1999).
using a purge-and-trap GC/MS methodology. The reported detection limit was 0.00006 mg/L, which is three orders of magnitude lower than the proposed human drinking water guideline. The accuracy and precision of the purge-and-trap GC/MS methodology is excellent for environmental sampling.

### 5.4 Recommended Analytical Methodology

The recommended methods for MTBE analysis require the use of GC/MS technology (USEPA, 1997). Methods include USEPA 8260, 8240, 624, and 524.2. The analysis should be performed by a laboratory certified to perform these methods.

The GC/PID method could be used for screening purposes. Confirmatory analysis by GC/MS would be required should a detection occur using GC/PID.
6. CLOSURE

The information presented in this report was produced exclusively for the purposes stated in the Scope of Work. Komex International Ltd. provided these guidelines for the British Columbia Ministry of Environment, Lands & Parks, solely for the purpose noted above, and does not accept any responsibility for the use of the guidelines for any purpose other than intended or to any third party.

Komex International Ltd. has exercised reasonable skill, care, and diligence to assess the information acquired during the preparation of this report. The methodologies used deriving the guidelines are based on current federal regulatory protocols and current understanding of biological systems, mechanisms of exposure, and toxicological properties of chemicals.

Questions concerning the derivation or use of the guidelines in this report should be directed to Dr. James H. Sevigny.
7. REFERENCES

7.1 General


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7.2 Guidelines – Other Jurisdictions

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7.3 Effects on Freshwater Aquatic Life


### 7.4 Effects on Marine Life


7.6 Effects on Terrestrial Plants


7.7 Mammalian Toxicology


### 7.8 Avian Toxicity

No references identified in our search.

### 7.9 Analytical


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<th>Property</th>
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<td>----</td>
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<td>Density (20 °C)</td>
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<td>Aqueous solubility (20 °C)</td>
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<td>Vapour pressure (25 °C)</td>
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<td>kPa</td>
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<td>Solubility in gasoline</td>
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<td>Henry's law constant (25 °C)</td>
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<td>Conversion factor (in air)</td>
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<td>Verschueren, 1983</td>
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<td>Flash point</td>
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<td>Odour in air</td>
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<td>Odour threshold</td>
<td>53</td>
<td>ppb</td>
<td>API, 1993</td>
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<td></td>
<td>15</td>
<td>ppb</td>
<td>Young et al., 1996</td>
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<tr>
<td></td>
<td>13.5 to 43.5</td>
<td>ppb</td>
<td>Shen et al., 1997</td>
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<td>ppb</td>
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<td></td>
<td>40</td>
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Table 2. Proposed Water Quality Guidelines for MTBE

<table>
<thead>
<tr>
<th>Water Use</th>
<th>Human Drinking Water (mg/L)</th>
<th>Recreational (mg/L)</th>
<th>Freshwater Aquatic Life (mg/L)</th>
<th>Marine Aquatic Life (mg/L)</th>
<th>Irrigation (mg/L)</th>
<th>Livestock Watering (mg/L)</th>
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<td>Preliminary(2)</td>
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</table>

1. These guidelines are based on a review of values used in other jurisdictions that have developed guidelines.
2. Insufficient data was available to satisfy CCME protocol requirements for an Interim guideline. Guideline is designated “Preliminary”.

J:\52330000\TABLES\Final Word Tables.doc
### Table 3. MTBE Drinking Water Guidelines from Other Jurisdictions

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<thead>
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<th>State</th>
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**NOTES:**

a. indicates information unavailable  
b. indicates presumed to be based on USEPA (1997) Drinking Water Advisory
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![Table 4. Toxicity of MTBE to Freshwater Aquatic Life](J:\52330000\TABLES\Final Excel Tables.xls) - Table 4 Page 1 of 104/17/2001 - 2:17 PM

NOTES:  
1. Data are classified as Primary, Secondary, or Unacceptable, based on criteria in CCME (1996). Unacceptable data may represent studies which are, in fact, acceptable, but for which insufficient information was available.  
2. Salmonella typhimurium data may not be representative of the toxicity of MTBE to freshwater aquatic life and are not included in the guideline derivation process.  
3. The matrix of temperature and pH is limited to those commonly occurring in nature.  
4. All NOECs and LOECs are statistically derived.  
5. Toxicity data may not be representative of the toxicity of MTBE to freshwater aquatic life and are not included in the guideline derivation process.
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NOTES:
1. Data are classified as Primary, Secondary, or Unacceptable, based on criteria in CCME (1996). Unacceptable data may represent studies which are, in fact, acceptable, but for which insufficient information was available.
2. Microtox data may not be representative of the toxicity of MTBE to marine life, and are not included in the guideline derivation process.
3. nr = not reported
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<td>10 males and 10 females per dose group;</td>
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<td>Hypersensitivity, muscular weakness, hypothermia, inactivation and death; the symptoms were reversible in sub lethal doses. Inflammation of the stomach and/or small intestine in animals that died</td>
<td>2 to 5 fold increase in hepatic cell labelling index in the absence of mutagenic marker). 2 fold increase in hepatic 7-ethoxy-resorufin-o-deethylase activity (EROD activity) marker. 2 fold increase in hepatic 7-ethoxyresinolin-o-deethylase activity (EROD activity) marker.</td>
<td>2 fold increase in hepatic cell labelling index in the absence of mutagenic marker). 2 fold increase in hepatic 7-ethoxy-resorufin-o-deethylase activity (EROD activity) marker. 2 fold increase in hepatic cell labelling index in the absence of mutagenic marker). 2 fold increase in hepatic 7-ethoxy-resorufin-o-deethylase activity (EROD activity) marker. 2 fold increase in hepatic cell labelling index in the absence of mutagenic marker). 2 fold increase in hepatic cell labelling index in the absence of mutagenic marker). 2 fold increase in hepatic cell labelling index in the absence of mutagenic marker). 2 fold increase in hepatic cell labelling index in the absence of mutagenic marker). 2 fold increase in hepatic cell labelling index in the absence of mutagenic marker). 2 fold increase in hepatic cell labelling index in the absence of mutagenic marker). 2 fold increase in hepatic cell labelling index in the absence of mutagenic marker). 2 fold increase in hepatic cell labelling index in the absence of mutagenic marker). 2 fold increase in hepatic cell labelling index in the absence of mutagenic marker). 2 fold increase in hepatic cell labelling index in the absence of mutagenic marker). 2 fold increase in hepatic cell labelling index in the absence of mutagenic marker). 2 fold increase in hepatic cell labelling index in the absence of mutagenic marker). 2 fold increase in hepatic cell labelling index in the absence of mutagenic marker). 2 fold increase in hepatic cell labelling index in the absence of mutagenic marker). 2 fold increase in hepatic cell labelling index in the absence of mutagenic marker). 2 fold increase in hepatic cell labelling index in the absence of mutagenic marker). 2 fold increase in hepatic cell labelling index in the absence of mutagenic marker). 2 fold increase in hepatic cell labelling index in the absence of mutagenic marker). 2 fold increase in hepatic cell labelling index in the absence of mutagenic</td>
<td>7</td>
<td>Industrial Bio-Test Laboratories, 1989</td>
<td>Y</td>
<td>non-pub report</td>
<td></td>
<td></td>
</tr>
<tr>
<td>acute</td>
<td>Rat (strain ?)</td>
<td>288 mg/kg</td>
<td>LD50</td>
<td>Carboplatin depression, ataxia, laboured respiration and death</td>
<td>2000, 3500, 4500, 6000, 10200 mg/kg bw</td>
<td>7</td>
<td>ARCO, 1987</td>
<td>Y</td>
<td>Y</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>chronic</td>
<td>Sprague-Dawley Rat</td>
<td>1430 / 36000 &gt;10/90 mg/kg-day</td>
<td>LOEL for general toxicity/NOAEL for reproductive effects</td>
<td>no adverse effects at 1430, decreased body weight, body weight gain, and food consumption in parents, hypersensitivity, lack of sterile reflex and bleohemorrhap, reduced pup body weight and body weight gain post natale at 10700 and up.</td>
<td>99% pure 0, 1430, 10700, 28600 mg/kg</td>
<td>7</td>
<td>Heesper-Bredenkamp, 1991; Beven et al., 1997</td>
<td>Y</td>
<td>non-pub report</td>
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<td>chronic</td>
<td>Sprague-Dawley Rat</td>
<td>6440 / 12140 mg/kg</td>
<td>NOAEC/LOAEC for reproductive effects</td>
<td>slightly decreased pup viability in F1b at 12140</td>
<td>5 males and 30 females group</td>
<td>5 males and 30 females group</td>
<td>5, 1070; 1445; 12140 mg/kg</td>
<td>7</td>
<td>Biles et al., 1987</td>
<td>Y</td>
<td>non-pub report</td>
<td></td>
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<tr>
<td>chronic</td>
<td>Fisher-344 Rat</td>
<td>1440 / 10700 &gt; 1440 mg/kg-day</td>
<td>NOAEC for females/LOEL for males (neoplasia)</td>
<td>increased mortality &amp; decreased mean survival time in males at 1440. bleohemorrhap, hypersensitivity, ataxia, lack of sterile reflex, swollen pericelar tissue, salivation, decreased body weight. A body weight gain in both sexes at 28600. Increased body weight and body weight gain in males at 1430 and 10700. Decreased concomitance in males at 28600. Increased relative liver &amp; kidney weight in both sexes at 10700. Increased absolute liver &amp; kidney weight in females at 10700 &amp; 28600. increased relative adrenal weight in males at 28600. Increased incidence &amp; severity of chronic progressive nephropathy in males at 1430 &amp; 1440, and females at 10700 &amp; 28600. nephropathy included increased in severity of glomerulosclerosis, tubular proteinosis, interstitial nephritis &amp; interstitial fibrosis in both sexes at 28600 &amp; 28600. nephropathy associated with secondary lesions (fibrous osteodystrophy, pancreas hyperplasia, &amp; mineralization in numerous tissues). increased incidence of renal tubular cell adenomas &amp; carcinomas, &amp; dose related increase in Leydig cell adenomas in males at 10700 &amp; 28600. renal tubular cell carcinomas only at 10700. expt 1 30 weeks, 20/6 weeks; group 1 30/sex/group, inhalation chambers; group control received filtered air</td>
<td>356, 1430, 10700; 28600 mg/kg</td>
<td>7</td>
<td>Cullen et al., 1992; Bird et al., 1997</td>
<td>Y</td>
<td>non-pub report</td>
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Table 6. Toxicity of MTBE to Mammalian Species

<table>
<thead>
<tr>
<th>Type</th>
<th>Species</th>
<th>Concentration or Dose</th>
<th>Effect</th>
<th>Observations</th>
<th>Test design</th>
<th>Text description</th>
<th>Formulation</th>
<th>Test Concentrations</th>
<th>Control Survival</th>
<th>Reference</th>
<th>Available</th>
<th>Note</th>
</tr>
</thead>
<tbody>
<tr>
<td>acute</td>
<td>CD-1 Mice</td>
<td>1440 mg/m³</td>
<td>NOEL for toxicity.</td>
<td>increased mortality, increased decrease in mean survival time in males at 28600. ataxia, biphosphatemia, anorexia, dehydration, and histochemical analysis of bone marrow in males at 28600. decreased body weight and body weight gain in all sexes at 28600. increase in absolute liver weight and kidney weight in males at 14300 and up. increase in kidney weight in females at 28600. increase in serum corticosterone in male mice at 28600. increased depression and ataxia in both sexes at 28600. among dead males high frequency of urinary bladder dilatation/augmentation. increased incidence of liver masses in both sexes at 28600. increase in hepatic fibrosis and atrophy in males at 28600. decreased mineralization in brain of both sexes at 28600. dose-related increased incidence of cystic renal endometrial hyperplasia females. increase in incidence of hepatocellular adenomas in females at 28600.</td>
<td>18 weeks; 5/12 sex/week; 6 females/group per inhalation chamber</td>
<td>(not sig. at 21 d). 48% decrease in uterine weight at 3d, 65% decrease at 21 d. 65% decrease in relative ovarian weight, at 21 d, 2.1 fold increase in estrogen metabolism. mild centrilocular to midzonal hepatocyte swelling at 3d, no change at 21 d. 1.8 fold increase in EROD at 3d, 3.2 fold at 21d. no histological hepatotoxicity. no change in serum ALAT.</td>
<td>0, 2860, 14300, 28600 mg/m³</td>
<td>3 or 21 days</td>
<td>Dodd and Kintigh, 1989; From WHO, 1998</td>
<td>?</td>
<td>??</td>
<td></td>
</tr>
<tr>
<td>subchronic</td>
<td>Sprague-Dawley Rat</td>
<td>3600 / 3600 mg/m³</td>
<td>NOEL = LOEC for hematological (males) and respiratory (females) effects</td>
<td>increased hematocrit; weight in males at 28600. decrease in relative and absolute lung weights in females at 28600.</td>
<td>3 weeks; 5/12 sex/week; 6 females/group per inhalation chamber</td>
<td>(not sig. at 21d). 40% decrease in uterine weight at 3d, 65% decrease at 21 d. 65% decrease in relative ovarian weight, at 21 d, 2.1 fold increase in estrogen metabolism. mild centrilocular to midzonal hepatocyte swelling at 3d, no change at 21 d. 1.8 fold increase in EROD at 3d, 3.2 fold at 21d. no histological hepatotoxicity. no change in serum ALAT.</td>
<td>0, 2860, 14300, 28600 mg/m³</td>
<td>3 or 21 days</td>
<td>Dodd and Kintigh, 1989; From WHO, 1998</td>
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<tr>
<td>subchronic</td>
<td>Fisher-344 Rat</td>
<td>2860 mg/m³</td>
<td>NOEL = LOEC for hematological (males), hepatic (males), and renal (male) effects.</td>
<td>increased hematocrit; weight in males at 28600. decrease in relative and absolute lung weights in females at 28600.</td>
<td>3 weeks; 5/12 sex/week; 6 females/group per inhalation chamber</td>
<td>(not sig. at 21d). 40% decrease in uterine weight at 3d, 65% decrease at 21 d. 65% decrease in relative ovarian weight, at 21 d, 2.1 fold increase in estrogen metabolism. mild centrilocular to midzonal hepatocyte swelling at 3d, no change at 21 d. 1.8 fold increase in EROD at 3d, 3.2 fold at 21d. no histological hepatotoxicity. no change in serum ALAT.</td>
<td>0, 2860, 14300, 28600 mg/m³</td>
<td>3 or 21 days</td>
<td>Dodd and Kintigh, 1989; From WHO, 1998</td>
<td>?</td>
<td>??</td>
<td></td>
</tr>
<tr>
<td>acute</td>
<td>B6C3F1 Mouse</td>
<td>7200 mg/m³</td>
<td>NOEL = LOEC for hematological (males) and respiratory (females) effects</td>
<td>increased hematocrit; weight in males at 28600. decrease in relative and absolute lung weights in females at 28600.</td>
<td>3 weeks; 5/12 sex/week; 6 females/group per inhalation chamber</td>
<td>(not sig. at 21d). 40% decrease in uterine weight at 3d, 65% decrease at 21 d. 65% decrease in relative ovarian weight, at 21 d, 2.1 fold increase in estrogen metabolism. mild centrilocular to midzonal hepatocyte swelling at 3d, no change at 21 d. 1.8 fold increase in EROD at 3d, 3.2 fold at 21d. no histological hepatotoxicity. no change in serum ALAT.</td>
<td>0, 2860, 14300, 28600 mg/m³</td>
<td>3 or 21 days</td>
<td>Dodd and Kintigh, 1989; From WHO, 1998</td>
<td>?</td>
<td>??</td>
<td></td>
</tr>
<tr>
<td>acute</td>
<td>New Zealand White Rabbit</td>
<td>13750 mg/m³</td>
<td>NOEL = LOEC for hematological (males) and respiratory (females) effects.</td>
<td>increased hematocrit; weight in males at 28600. decrease in relative and absolute lung weights in females at 28600.</td>
<td>3 weeks; 5/12 sex/week; 6 females/group per inhalation chamber</td>
<td>(not sig. at 21d). 40% decrease in uterine weight at 3d, 65% decrease at 21 d. 65% decrease in relative ovarian weight, at 21 d, 2.1 fold increase in estrogen metabolism. mild centrilocular to midzonal hepatocyte swelling at 3d, no change at 21 d. 1.8 fold increase in EROD at 3d, 3.2 fold at 21d. no histological hepatotoxicity. no change in serum ALAT.</td>
<td>0, 2860, 14300, 28600 mg/m³</td>
<td>3 or 21 days</td>
<td>Dodd and Kintigh, 1989; From WHO, 1998</td>
<td>?</td>
<td>??</td>
<td></td>
</tr>
<tr>
<td>acute</td>
<td>New Zealand White Rabbit</td>
<td>13750 mg/m³</td>
<td>NOEL = LOEC for hematological (males) and respiratory (females) effects.</td>
<td>increased hematocrit; weight in males at 28600. decrease in relative and absolute lung weights in females at 28600.</td>
<td>3 weeks; 5/12 sex/week; 6 females/group per inhalation chamber</td>
<td>(not sig. at 21d). 40% decrease in uterine weight at 3d, 65% decrease at 21 d. 65% decrease in relative ovarian weight, at 21 d, 2.1 fold increase in estrogen metabolism. mild centrilocular to midzonal hepatocyte swelling at 3d, no change at 21 d. 1.8 fold increase in EROD at 3d, 3.2 fold at 21d. no histological hepatotoxicity. no change in serum ALAT.</td>
<td>0, 2860, 14300, 28600 mg/m³</td>
<td>3 or 21 days</td>
<td>Dodd and Kintigh, 1989; From WHO, 1998</td>
<td>?</td>
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40/17/2001 - 3:40 PM
J:\52330000\TABLES\Final Excel Tables.xls - Table 6
Page 2 of 3
<table>
<thead>
<tr>
<th>Type</th>
<th>Species</th>
<th>Concentration or Dose</th>
<th>Effect</th>
<th>Observations</th>
<th>Test design</th>
<th>Formulation</th>
<th>Test Concentrations</th>
<th>Control Survival</th>
<th>Reference</th>
<th>Available?</th>
<th>Notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>acute</td>
<td>Rat (strain?)</td>
<td>3400 mg/m³</td>
<td>LOEL for renal effects in males</td>
<td>glomerulotoxic increase and protein (drenal) accumulation in rats at 14300 and 10760, alpha-2u-globulin immunonoactivity in protein droplets, mild dose dependent increase in alpha 2u with exposure related increase in cell proliferation, no differences found in females.</td>
<td>10 days; 3x/week</td>
<td>7</td>
<td>2</td>
<td>3400, 10760</td>
<td>7</td>
<td>Prescott-Mathews et al., 1997</td>
<td>y?</td>
</tr>
<tr>
<td>acute</td>
<td>Sprague-Dawley Rat</td>
<td>9000 mg/m³</td>
<td>NOEL for developmental toxicity</td>
<td>decreased food consumption in 900 and up, no significant developmental toxicity</td>
<td>gestation days 15</td>
<td>25 males/dose</td>
<td>7</td>
<td>2</td>
<td>9000, 28600, 9000 mg/m³</td>
<td>7</td>
<td>Conaway et al., 1985</td>
</tr>
<tr>
<td>acute</td>
<td>CD-1 Mice</td>
<td>9000 mg/m³</td>
<td>NOEL for developmental toxicity</td>
<td>slightly decreased food and water consumption (dose related, but not sig), no significant developmental toxicity</td>
<td>gestation days 15</td>
<td>25 mice/dose</td>
<td>7</td>
<td>2</td>
<td>9000, 28600, 9000 mg/m³</td>
<td>7</td>
<td>Conaway et al., 1985</td>
</tr>
<tr>
<td>acute</td>
<td>CD-1 Mice</td>
<td>28600 (0, 14300 mg/m³)</td>
<td>NOEL: LOEL for developmental and maternal toxicity</td>
<td>clinical signs of toxicity and reduced fetal body weight at 14300 and 28600; reduced body weight, body weight gain, and food consumption, increased number of non-viable implantations, reduced number of viable implantations and % male fetuses, increased incidence of cleft palate at 28600.</td>
<td>gestation days 15</td>
<td>30 mice/dose</td>
<td>7</td>
<td>2</td>
<td>28600, 14300, 10760 mg/m³</td>
<td>7</td>
<td>Yip and Neuper-Bradley, 1989</td>
</tr>
<tr>
<td>acute</td>
<td>Fisher-344 Rat</td>
<td>2860 mg/m³</td>
<td>MUNE: LUEA: neurotoxicity</td>
<td>no mortality or cerebellar toxicity at any concentration, at 1x increase incidence and severity of ataxia and dach-walk gait in both sexes for 4000 and 40000ppm: 4000ppm males: ataxia, decreased muscle tone, decreased rectal temperature, increased exhalation performance time, increased hind limb spay, FOBT alterations, and increased incidence of total respiration. 4000 and 40000ppm females: increased incidence of ataxia, perection, ataxia, dach-walk gait, decreased rectal temperature, and decreased hind limb grip strength. 8000ppm females, increased incidence of total respiration and latency to rotate on inclined screen, increased motor activity at 80000, decreased in 40000ppm males, no difference in female activity, no alterations at 6 or 24 h after exposure.</td>
<td>6 hours</td>
<td>0</td>
<td>? ? ?</td>
<td>4 ?</td>
<td>2</td>
<td>2860, 14300, 28600 mg/m³ (0, 800, 4000, 4000 ppm)</td>
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<tr>
<td>acute</td>
<td>White Mouse (strain?)</td>
<td>51,000 mg/m³</td>
<td>LD50</td>
<td>median concentration for anesthesia (AC50) = 106 mg/L (1.2 mmoi/L.)</td>
<td>5 minutes</td>
<td>whole body exposure; 20 mice/exposure group</td>
<td>7</td>
<td>7</td>
<td>Marshe and Leake, 1950</td>
<td>y?</td>
<td>From WHD, 1998</td>
</tr>
<tr>
<td>acute</td>
<td>Weiss Mouse</td>
<td>20,000 mg/kg (200-2000 mg/kg)</td>
<td>LC50</td>
<td>LC50 for anesthesia 200 mg/kg, air (150-300 mg/kg); convulsive hyperventilation, hyperactivity, ataxia, loss of righting reflex, toxic and opercular convulsive seizures</td>
<td>7 minutes</td>
<td>whole body exposure; observation for 48 hours following exposure; 4 males/exposure group</td>
<td>7</td>
<td>7</td>
<td>Industrial Bio-Test Laboratories, 1969</td>
<td>y?</td>
<td>non-pub report</td>
</tr>
<tr>
<td>acute</td>
<td>Weiss Mouse</td>
<td>0.6 ml - 8 ml/kg (185/5v/v)</td>
<td>LC50</td>
<td>death occurred within 1 hour</td>
<td>10-15 minutes</td>
<td>whole body exposure: 20% v/v in air in 3, 4, 5, 6, 9, 12 minutes 6, 12, 17, 20, 22, 26% v/v in air for 10 minutes</td>
<td>7</td>
<td>7</td>
<td>Shangpinnelli, 1980</td>
<td>y?</td>
<td>non-pub report</td>
</tr>
<tr>
<td>acute</td>
<td>Rat (strain?)</td>
<td>142,000 mg/m³</td>
<td>LC50</td>
<td>eye irritation, incoordination, ataxia, wipe, loss of righting reflex, and death</td>
<td>0</td>
<td>1</td>
<td>7</td>
<td>7</td>
<td>APCC, 1981</td>
<td>y?</td>
<td>non-pub report</td>
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<tr>
<td>chronic</td>
<td>New Zealand White Rabbit</td>
<td>70,200 mg/kg/day</td>
<td>LD50</td>
<td>no deaths, no gross pathological alterations other than necrosis at application site</td>
<td>28 days</td>
<td>2 males and 2 females/dose level</td>
<td>7</td>
<td>7</td>
<td>Industrial Bio-Test Laboratories, 1969</td>
<td>y?</td>
<td>non-pub report</td>
</tr>
<tr>
<td>acute</td>
<td>Rabbit (strain?)</td>
<td>10,000 mg/kg/day</td>
<td>LD50</td>
<td>nephritis, edema, scaling, furring and blanching dermatitis in animals that died</td>
<td>0</td>
<td>1</td>
<td>7</td>
<td>7</td>
<td>APCC, 1987</td>
<td>y?</td>
<td>non-pub report</td>
</tr>
<tr>
<td>acute</td>
<td>Sprague-Dawley Rat</td>
<td>0.2 mg/kg bw</td>
<td>LD50</td>
<td>intraperitoneal injection caused 100% mortality (pulmonary injury); intracerebral injection caused 90% mortality and peripheral vein injection injection 17% mortality, the pulmonary injury included congestion, hemorrhage, and interstitial edema.</td>
<td>0</td>
<td>7</td>
<td>2</td>
<td>7</td>
<td>Akimoto et al., 1992</td>
<td>y?</td>
<td>From WHD, 1998</td>
</tr>
<tr>
<td>acute</td>
<td>Weiss Mouse</td>
<td>0.6 ml/kg bw</td>
<td>LD50</td>
<td></td>
<td>0</td>
<td>7</td>
<td>2</td>
<td>7</td>
<td>Shangpinnelli, 1980</td>
<td>y?</td>
<td>non-pub report</td>
</tr>
<tr>
<td>acute</td>
<td>Mouse (strain?)</td>
<td>200 (194-215)</td>
<td>LD50</td>
<td></td>
<td>0</td>
<td>7</td>
<td>2</td>
<td>7</td>
<td>Martin and et al., 1930</td>
<td>y?</td>
<td>From WHD, 1998</td>
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<tr>
<td>acute</td>
<td>Wistar Rat</td>
<td>3.56 mg/kg bw</td>
<td>LD50</td>
<td>in lethal dosage: nervous depression, sometimes followed by short clinical convulsions, autonomic activity (hyperventilation, arrhythmia, delirium) and respiratory disorders; death occurred within 30 minutes. surviving animals: no toxic symptoms or signs of nervous depression lasting more than 15 to 20 min.</td>
<td>4</td>
<td>7</td>
<td>2</td>
<td>7</td>
<td>Shangpinnelli, 1980</td>
<td>y?</td>
<td>non-pub report</td>
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<tr>
<td>acute</td>
<td>Wistar Rat</td>
<td>3.7 mg/kg bw</td>
<td>LD50</td>
<td></td>
<td>0</td>
<td>7</td>
<td>2</td>
<td>7</td>
<td>Shangpinnelli, 1980</td>
<td>y?</td>
<td>non-pub report</td>
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<tr>
<td>acute</td>
<td>Weiss Mouse</td>
<td>2.8 mg/kg bw (3.9-3.57 mg/kg)</td>
<td>LD50</td>
<td></td>
<td>0</td>
<td>7</td>
<td>2</td>
<td>7</td>
<td>Shangpinnelli, 1980</td>
<td>y?</td>
<td>non-pub report</td>
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### Table 7. Sample Preparation Methods

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<th>USEPA SW-846 Method</th>
<th>Technology</th>
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<td>purge-and-trap</td>
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*Summarized from Rhodes and Verstuyft (1999).*
Table 8. Summary of Methods Applicable to MTBE Analysis

<table>
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<tr>
<th>EPA SW-846 Method</th>
<th>Detector</th>
<th>Target Analytes</th>
<th>Comparable EPA Methods</th>
<th>Selectivity</th>
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<tbody>
<tr>
<td>8015</td>
<td>FID</td>
<td>non-halogenated organics</td>
<td>None</td>
<td>Poor</td>
</tr>
<tr>
<td>8021</td>
<td>PID</td>
<td>aromatic and halogenated volatiles</td>
<td>8020, 602, 502.3</td>
<td>Good</td>
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<tr>
<td>8260</td>
<td>MS</td>
<td>volatile organic compounds</td>
<td>8240, 624, 524.2</td>
<td>Excellent</td>
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</tbody>
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### Table 9. Performance of Purge-and-Trap GC/MS Analysis for MTBE in Water

<table>
<thead>
<tr>
<th>Compound</th>
<th>Detection Limit (mg/L)</th>
<th>Accuracy (% Recovery)</th>
<th>Precision (%)</th>
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<tbody>
<tr>
<td>MTBE</td>
<td>0.00006</td>
<td>97</td>
<td>2.5</td>
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Rhodes and Verstuyft (1999):