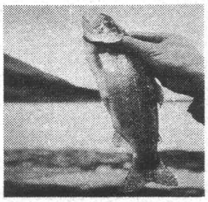
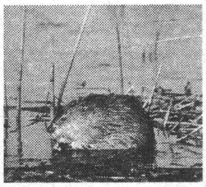
Canada Alberta Northwest Territories

Northern River Basins Study





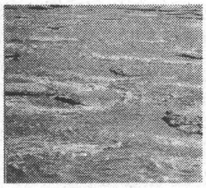



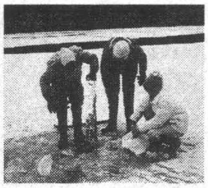


NORTHERN RIVER BASINS STUDY PROJECT REPORT NO. 52

A REVIEW AND ANNOTATED BIBLIOGRAPHY OF WATER AND FISH TAINTING

IN THE PEACE, ATHABASCA AND SLAVE RIVER BASINS







PREFACE:

The Northern River Basins Study was initiated through the "Canada-Alberta-Northwest Territories Agreement Respecting the Peace-Athabasca-Slave River Basin Study, Phase II - Technical Studies" which was signed September 27, 1991. The purpose of the Study is to understand and characterize the cumulative effects of development on the water and aquatic environment of the Study Area by coordinating with existing programs and undertaking appropriate new technical studies.

This publication reports the method and findings of particular work conducted as part of the Northern River Basins Study. As such, the work was governed by a specific terms of reference and is expected to contribute information about the Study Area within the context of the overall study as described by the Study Final Report. This report has been reviewed by the Study Science Advisory Committee in regards to scientific content and has been approved by the Study Board of Directors for public release.

It is explicit in the objectives of the Study to report the results of technical work regularly to the public. This objective is served by distributing project reports to an extensive network of libraries, agencies, organizations and interested individuals and by granting universal permission to reproduce the material.

This report contains referenced data obtained from sources external to the Northern River Basins Study. Individuals interested in using external data must obtain permission to do so from the donor agency.

Prepared for the
Northern River Basins Study
under Project 4412-C1

by

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Published by the
Northern River Basins Study
Edmonton, Alberta
June, 1995

CANADIAN CATALOGUING IN PUBLICATION DATA

Kenefik, Sandra L.

A review and annotated bibliography of water and
fish tainting in the Peace, Athabasca and Slave
River Basins

(Northern River Basins Study project report,
ISSN 1192-3571 ; no. 52)
Includes bibliographical references.
ISBN 0-662-23555-X
Cat. no. R71-49/3-52E

1. Effluent quality -- Alberta -- Athabasca River --
Bibliography.
2. Effluent quality -- Peace River (B.C. and Alta.) --
Bibliography.
3. Effluent quality -- Slave River (Alta. And N.W.T.) -
Bibliography.
4. Wood-pulp industry -- Waste disposal -- Alberta.
I. Hrudey, S.E. (Steve E.)
II. Northern River Basins Study (Canada)
III. Title.
IV. Series.

Z5862.2.W3K56 1995   016.36373'94'0971232   C95-980181-2

NORTHERN RIVER BASINS STUDY
PROJECT REPORT RELEASE FORM

This publication may be cited as:

Kenefik, Sandra L. and Hrudey, Steve E. 1995. ***Northern River Basins Study Project Report No. 52, A Review and Annotated Bibliography of Water and Fish Tainting in the Peace, Athabasca and Slave River Basins.*** **Northern River Basins Study, Edmonton, Alberta.**

Whereas the above publication is the result of a project conducted under the Northern River Basins Study and the terms of reference for that project are deemed to be fulfilled,
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7 June 95
(Date)

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(Dr. P. A. Larkin, Ph.D., Chair)

7 June 95
(Date)

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IT IS HERE APPROVED BY THE BOARD OF DIRECTORS THAT;
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Lucille Partington
(Lucille Partington, Co-chair)

June 8 / 95
(Date)

Robert McLeod
(Robert McLeod, Co-chair)

June 12/95
(Date)

A REVIEW AND ANNOTATED BIBLIOGRAPHY OF WATER AND FISH TAINTING IN THE PEACE, ATHABASCA AND SLAVE RIVER BASINS

STUDY PERSPECTIVE

Complaints about fish and water tainting in the Peace and Athabasca river systems have been documented by government regulators since at least the mid 1970's. Bleached kraft pulp mill effluent and other industrial and municipal effluents have been documented in the scientific literature to have the potential to cause off-flavours in water and fish in the receiving waters. As the first stage in addressing tainting concerns, the Northern River Basins Study supported a project to review the available literature for the incidence and likelihood for off-flavour tainting of water and fish by municipal, agricultural and industrial effluents, with an emphasis on pulp mill effluents. Analytical methods for investigating water and fish taste-odour problems were also reviewed to aid in the selection of a suitable technique for a follow-up project.

Related Study Questions

2) *What is the current state of water quality in the Peace, Athabasca and Slave river basins, including the Peace-Athabasca Delta?*

8) *Recognizing that people drink water and eat fish from these river systems, what is the current concentration of contaminants in water and edible fish tissue and how are these levels changing through time and by location?*

This project review identified a number of potential tainting compounds from various sources, including biological ones. The project report also provides a thorough background of what is presently known about the tainting of water and fish, with an emphasis on the potential sources present in the study area.

The literature and technique review was the initial step towards more detailed odour analysis projects. These projects include: odour tests on river and drinking water along the Athabasca River for the pre-ALPAC and the post-ALPAC start-up, and broad spectrum analysis of water and effluent samples collected from 32 sites along the Athabasca River. This latter project will provide an indication of the relative abundance of some of the tainting compounds in the system.

REPORT SUMMARY

The purpose of this review was to identify incidence of, or the potential for off-flavour tainting of fish and water by components discharged into the Peace, Athabasca, and Slave rivers. The main focus was the tainting effects of effluents released by pulp mills, but there is also a brief discussion of the role of other industrial discharges, municipal waste discharges, and non-point sources of run-off. There is a relatively weak literature base directly related to taste and odour problems attributable to pulp mills in the Northern Alberta river basins. However, discussions of other pulp mill related incidents of taste and odour problems throughout the world have been included.

There are a number of unique analytical methods used in the study of taste and odour problems and it is critical to have a basic understanding of these methods before interpreting reports of tainting incidents. To assist in the development of this understanding, a detailed review of analytical methods used for the investigation of water and fish tainting problems was included. There are a variety of chemical methods used for the isolation (extraction, adsorption and desorption, removal of interferences), fractionation, and instrumental separation and identification of the compounds responsible for taste and odour problems in water and fish. The problems and limitations associated with each of these methods were discussed. It is critical to select the most appropriate method for any specific taste and odour study in order to obtain meaningful results.

The sensory analytical techniques used for the study of taste and odour are even more unique and are subject to variabilities caused by differing human olfactory sensitivities. A detailed review of current sensory methods was completed. Sensory analysis is the only method of providing a qualitative description of the tastes or odours present in a sample. However in order to confirm that specific chemicals are responsible for specific odours in a sample, a combination of sensory and instrumental techniques is required. Chromatographic sniffing (olfactory GC) is the current method of choice when integrating sensory and common instrumental techniques.

Fish tainting as a result of pulp mill discharges is well documented and there have been a number of comprehensive reviews on the subject. Reports of fish tainting indicated that often the tainting could not be linked to any specific compounds and off-flavours were often a result of the combined effects of different industrial discharges into the same water body. The current state of the fish tainting problem in Northern Alberta river basins is relatively undefined. There are 10 pulp / paper mills in the Athabasca and Peace River basins and but other discharges are limited to five non-pulp mill industrial effluents, along with numerous small municipal sewage effluents. The combined tainting effects from multiple, diverse industrial effluents which arise in heavily industrialized regions is not a major concern in these rivers. We have therefore focused on the pulp mill discharges and non-industrial tainting substances (biogenic sources).

Although there is little definition of the specific compounds responsible for tainting problems downstream of pulp mill effluent discharges, water tainting problems in the Northern Alberta river basins have historically been attributed to kraft pulp mill effluents. There is significant discussion of the effects of chlorinated compounds formed as a result of the kraft bleaching process but the literature is not very helpful in elaborating the role of specific odour compounds produced prior to bleaching.

Early work identified a number of process streams prior to bleaching as the most significant contributors to overall mill effluent odour. The importance of these odour sources is dependent upon in-plant spill control and wastewater treatment efficiency but is independent of recent improvements to the bleaching processes. Samples collected downstream from the pulp mill in Hinton have often exceeded odour compliance levels for up to half the length of the Athabasca River (under winter ice) since the mill was constructed. Occasional taste and odour problems at the town of Peace River have also been attributed to upstream pulp mills. Reports of the odour component of water quality studies for the Wapiti-Smoky River system indicate a noticeable increase in odour attributed to kraft mill effluents during low flows (under ice). Biological or natural sources of tastes and odours in water must also be considered as potential causes of off-flavours and the possibility of odour synergism when these natural compounds are combined with anthropogenic tainting compounds is likely.

Thorough (and costly) drinking water treatment processes can be effective in removing most taste and odour compounds, provided the nature of the odour problem is understood and the treatment processes can be optimized for the types of problems that are identified. Removal of many of the relatively volatile, chlorinated organics often thought to be responsible for tainting problems has been demonstrated in pulp mill wastewater treatment system studies. However, removal efficiencies vary and systems must be operated consistently under optimum conditions if tainting incidents are to be avoided. The preferred control method is prevention of the formation of these odorous chlorinated organics. Chlorine dioxide substitution significantly reduces the formation of such compounds, as does the use of hardwood. Where non chlorine bleaching processes such as chemithermomechanical pulping (CTMP) are used there have been no documented taste and odour incidents.

Anecdotal, historical information obtained in discussions and correspondence with various regional fishery biologists in the Fish and Wildlife division of Alberta Environment repeatedly indicated that, in general, people do not eat the fish caught in the northern Alberta river basins. Similar avoidance by the First Nation's peoples was confirmed in communications with the NRBS Traditional Knowledge group leader. It is apparent that the concern surrounding tainting of water and fish by anthropogenic sources such as pulp mill discharges and accidental oil sands wastewater spills is not only based on documented problems but is also founded on expectations of tainting associated with the existence of industrial discharges.

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LIST OF ABBREVIATIONS

AOX	Adsorbable organic halogens
BKME	Bleached kraft mill effluent
BOD	Biochemical oxygen demand
BSME	Bleached sulphite mill effluent
CLSA	Closed-Loop stripping apparatus / analysis
CTMP	Chemithermomechanical pulping
FPA	Flavour profile analysis
FRS	Flavour rating scale
FTT	Flavour threshold test
GC	Gas chromatography
GC/FID	Gas chromatograph with flame ionization detector
GC/MS	Gas chromatograph with mass selective detector
IBMP	2-isobutyl-3-methoxypyrazine
IPMP	2-isopropyl-3-methoxypyrazine
KME	Kraft mill effluent
LLE	Liquid-liquid extraction
MIB	2-methylisoborneol
MPE	Mechanical pulping mill effluent
NRBS	Northern River Basin Study
OGC	Olfactory gas chromatography
OTC	Odour threshold concentration
SME	Sulphite mill effluent
SPME	Solid phase micro-extraction
TCA	Trichloroanisole
TON	Threshold odour number
UKME	Unbleached kraft mill effluent

ACKNOWLEDGEMENTS

The authors would like to thank Nola Low of the Environmental Health Program, at the University of Alberta for her invaluable assistance in library searches, document retrieval and flavour wheel figure preparation. Dr. Brian Brownlee also provided valuable input and editorial comments during the preparation and final review of this document and his assistance is appreciated.

The information supplied in personal communications with Sonny Flett, NRBS Traditional Knowledge Group Leader; Hugh Norris, Alberta Environmental Protection St. Paul; Ron Millson, Alberta Environmental Protection Peace River; Carl Hunt, Alberta Environmental Protection Peace River; and Dennis Musselman, Alberta Environmental Protection Grande Prairie, are gratefully acknowledged.

1. INTRODUCTION

Bleached kraft pulp mill effluent and discharges related to other industrial developments are known to contain odorous compounds that have the potential to cause off-flavours in water and fish in the receiving waters. The purpose of this project is to review the literature and government records to identify the incidence of and the potential for off-flavours tainting of water and fish by compounds discharged into the Peace, Athabasca, and Slave rivers by municipalities, agriculture and industrial developments. The major emphasis has been on the tainting effects of effluents released by pulp mills, however, major review papers and more general articles dealing with municipal wastes, non-point source run-off and non-pulp mill industry discharges common to the northern river basins have also been identified. The contributions that these non-pulp mill discharges can be expected to make to the off-flavour tainting of the water and/or fish are also reviewed.

There are a number of relatively unique analytical methods used in the study of taste and odour problems and it is critical to have a basic understanding of these methods before interpreting reports of tainting incidents. A detailed review of analytical methods used for the investigation of water and fish taste and odour problems is included. The problems and limitations associated with each of these methods are discussed, and some specific applications are documented in this report.

A summary of documented historical incidents of tainting of water or fish in the northern river basins and anecdotal historical information obtained in discussions and correspondence with various regional fishery biologists in the Fish and Wildlife Division of Alberta Environmental Protection is provided. These contacts were made in response to a request by the Study Board staff and were not a part of the original contract. The concerns voiced during consultations with the NRBS Traditional Knowledge Group Leader are included as well.

A number of review articles and books covering the same general subjects have already been published. These include a very complete summary of the identification and treatment of tastes and odours in drinking water by Mallevialle and Suffet (1987). This book, although limited to the taste and odour problems associated with drinking water, is comprised of detailed sections on the chemical and sensory methods of detecting odorous compounds, sources of tastes and odours in drinking water, identification of tastes and odours, treatment for the removal of taste and odour compounds, and the physiological mechanisms involved in olfaction and taste. There have been a few reviews which deal specifically with the treatment methods for the control or removal of off-flavour compounds from water (McGuire and Gaston, 1988; Monteil, 1983; Sävenhed, 1986; Hrubec and de Kruiff, 1983). Also of use in developing an understanding of taste and odour problems in the northern river basins of Alberta, are reviews which deal with the tainting of fish. Persson (1984) compiled a lengthy review of environmentally occurring compounds which have been found to be responsible for the tainting of fish, and Motohiro (1983) has detailed the incidence of fish tainting caused by petroleum compounds. A review prepared for Alberta Environment by Hrudey and Nelson (1986) summarized the fish tainting potential of oil sands tailing ponds, a source of off-flavours specifically related to the Athabasca River. There have also been three volumes of research publications directly related to off-flavours in the aquatic environment that have been published by the Taste and Odour Committee of the International Association on Water Pollution Research and Control (Water Science and Technology volume 15, 1983; volume 20(8/9), 1988; and volume 25(2), 1992). All three of these compilations document the proceedings of International Symposia on Off-Flavours in the Aquatic Environment and contain the research findings of the international community of researchers devoted to the study of adverse tastes and odours in drinking water and aquatic food sources.

2. CHEMICAL METHODS OF TASTE AND ODOUR COMPOUND ANALYSES

For the purposes of this review the analyses used for the determination of the presence of compounds which contribute to taste and odour in water or fish, have been divided into two categories. The first has been labeled as chemical methods and includes the discussion of methods used for isolation (extraction, adsorption and desorption), fractionation, and instrumental separation and identification. The second section refers to the sensory methods of detection, quantitation and description. There are some cases of direct overlap between the two categories, such as chromatographic sniffing which involves the use of an analytical instrument for the chromatographic separation and requires the human nose as a detector. This method is discussed in the sensory section.

The chemical analyses for taste and odour compounds in water or fish has been developing for a number of years and has been driven by the continuing requirement for detection limits below the odour threshold of the various compounds commonly responsible for taste and odour problems. In the case of geosmin, one of the most common sources of off-flavours in water, the reported threshold for olfactory detection ranges from 4 ng/L to 200 ng/L, with current workers in the field expecting an odour detection threshold of 10 to 20 ng/L for most individuals (Hrudey and Low, 1992). Such low detection limit requirements have made the development of adequate analytical methods an ongoing challenge.

Generally the first concern in such analyses is isolating the compounds of interest from the water phase so that they may be concentrated. This can be done in a number of ways, including, liquid-liquid extraction, continuous liquid-liquid extraction, closed-loop stripping analysis, steam distillation extraction, solid phase extraction, adsorption onto carbon or resins, with subsequent organic solvent desorption, and purge trap types of extraction. The goal with these isolation methods has been two-fold; clean-up to remove interferences, and concentration to allow detection of compounds present in water at ng/L levels. A number of reviews and compilations of papers provide detailed summaries of extraction and analytical methods used for analyses of off-flavour compounds (Veijanen, *et al.*, 1983; Mallevialle and Suffet, 1987; Blok *et al.* 1983, Lundgren, *et al.* 1988; Leenheer, 1984, Keith, 1979). Kopfler and co-workers compared seven different methods of extracting and concentrating organics from environmental water supplies (Kopfler, *et al.*, 1986). This comparative study was based on methods that could be used to preparatively isolate gram quantities of specific organic compounds. No one method was superior for all compounds studied.

2.1 LIQUID-LIQUID EXTRACTION

Liquid-liquid extraction (LLE) has been used for the isolation of organics from aqueous samples for many years. The basic principal of the liquid-liquid extraction method is the partitioning of solutes (compounds of interest) between two immiscible liquids. In the case of aqueous environmental samples the two liquids are generally water and an organic solvent that is immiscible with water. The organic compounds of interest often have a greater tendency to be associated with the organic phase than the aqueous phase and can be transferred to an organic solvent by a simple contact mixing extraction. Extraction efficiencies for various compounds can be expressed as a partition coefficients (K) or as *p* -values (Mallevialle and Suffet , 1987). They defined the *p* -value as the distribution of a solute between two phases at equilibrium at a 1:1 water-to-solvent ratio. They also defined the fraction of solute that partitions into the organic phase as the *E*- value, where:

$$E = \frac{A_n}{A_s} \times \frac{V_n}{V_s}$$

Where:

A_n = amount of analyte in organic phase
A_s = amount of analyte in water phase
V_n = final volume of organic solvent at equilibrium
V_s = initial volume of organic solvent
$\frac{V_n}{V_s}$ = fractional recovery of organic solvent after equilibrium is reached.

The *E* -value is calculated for any solvent water ratio and then is normalized back to a 1:1 ratio to obtain the *p* -value:

$$p = \frac{E}{\alpha - [E(\alpha - 1)]}$$

Where:

α = the equilibrated solvent-to-water ratio:

$$\alpha = \frac{V_n}{V_p}$$

Where:

V_p = the final volume of water phase at equilibrium.

The *p* -value can also be expressed fractionally as a partition coefficient, *K* :

$$K = \frac{p}{1-p}$$

The greater the *p* -value the greater the amount of the analyte in the organic phase, or in other words the more efficient the extraction from the water phase. The LLE method is commonly used for the determination of compounds that readily partition into an organic solvent and are amenable to gas chromatography (APHA, AWWA, WEF, 1992). This standard method has also been adapted for use with pH adjustment so that acid and base/neutral extractables can be separated. However, there is a possibility of chemical reactions when the pH is adjusted and that may yield compounds not present in the original sample. These reaction products will contaminate the final extract. Ibrahim and co-workers compiled a good summary of compounds that may be present as contaminants when using dichloromethane as solvent for acid or base/neutral liquid-liquid extractions of water which has been chlorinated. These compounds are reported to be free radical chlorination and oxidation products of cyclohexene (which is added as a preservative to the dichloromethane) (Ibrahim, *et al.*, 1987a). Brownlee and co-workers reported extraction efficiencies for geosmin and 2-methylisoborneol using a simple liquid-liquid extraction method and their study documents implementation of the LLE method on site at a water treatment facility with relatively inexperienced personnel and without elaborate equipment (Brownlee, *et al.*, 1988). The basic LLE procedure has also been adapted for use with large volume samples (Fox, 1986) and microbiological cultures (Johnsen and Kuan, 1987).

The major limitation of the LLE procedure is the requirement for concentration of the organic phase after extraction. Often, the analyte concentration in the extraction solvent is too low to allow a direct analytical determination and there must be a concentration step to reduce the samples volume and increase the relative concentration. This is generally done using a Kuderna-Danish (K-D) evaporator (APHA, AWWA, WEF, 1992), automated evaporative concentration system (Ibrahim, *et al.*, 1987b) , or rotary evaporator and blow-down with nitrogen (Brownlee, *et al.* 1988). All of these methods assume that the organic solvent has a vapour pressure substantially greater than the compounds of interest. If this is not the case then there will be substantial losses of the volatiles during the final concentration (Blomberg, 1988). Internal standards are often added to the sample prior to extraction so as to allow for accurate quantitation despite these losses (Wood and Snoeyink, 1977; Brownlee, *et al.*, 1988 and Brownlee *et al.* 1993).

Other types of larger volume LLE systems have also been developed. These include a continuous liquid-liquid extractor (Baker and Suffet, 1987), steam distillation with continuous liquid-liquid extraction for the recovery of steam volatile compounds (Likens and Nickerson, 1964; Lee, 1988), and the "Goulden extractor" which was developed for extraction of large volumes samples at rates of up to 500 mL per minute (Goulden and Anthony, 1985). The "Goulden Extractor" utilizes a mixed settler design, where the water is stirred with the dichloromethane (DCM) extraction solvent. The solvent settles to the bottom while the water is passed through the mixing chamber. Solvent is also added at a flow of about 2% of the water flow (to replace the small fraction of solvent that is miscible with the water) This mixing chamber is combined with a counter-current packed column that breaks emulsions, and scrubs out solvent droplets from the effluent water. The use of continually added surrogate standards and the operation of large "Goulden extractor" onboard a sample collection ship have also been reported (Neilson *et al.*, 1988).

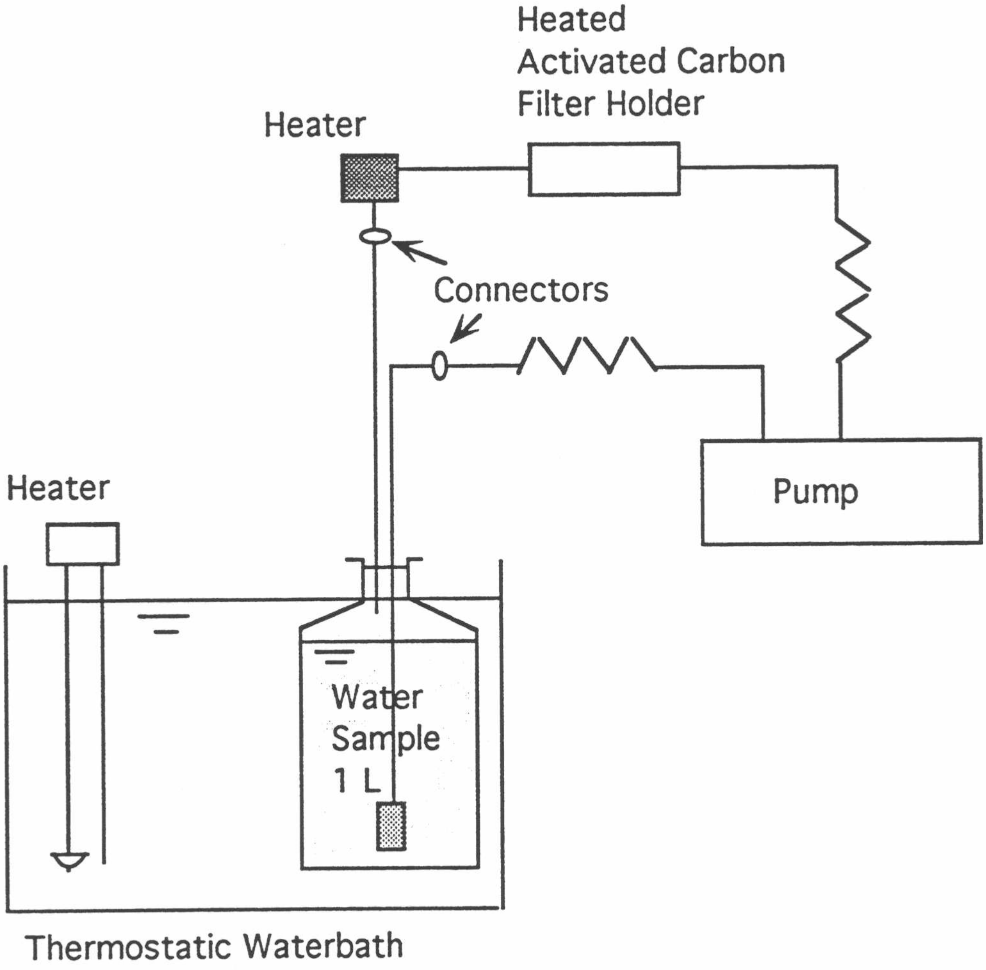
2.2 CLOSED-LOOP STRIPPING ANALYSIS

An isolation technique known as closed-loop stripping analysis (CLSA) has also been developed specifically for the isolation and concentration of non-polar, volatile compounds that are easily purged from the water phase to the air phase. The CLSA extraction method developed by Grob and co-workers is based on a combination of purging volatile compounds from a sample with a recirculating gas (usually headspace air) and adsorbing the volatile organics onto an activated carbon trap with subsequent desorption using an organic solvent (Grob, 1973; Grob and Grob, 1974; Grob *et al.*, 1975; Grob and Zürcher, 1976). The solvent extract is then analyzed using GC/MS. The commercially available CLSA system manufactured by Breuchbuhler is designed to purge the volatiles from a one litre sample and trap them on a 1.5 mg activated carbon trap. The carbon trap is then extracted with 25µL of an appropriate solvent mixture. The basic CLSA system is shown in Figure 2.1.

The main advantage of the CLSA method is the concentration of analytes of interest from an initial volume of one litre down to a final solvent extract volume of 25µL (a 40,000 fold concentration) without the need for an evaporation step. This allows detection of trace organics at nanogram-per-litre levels. Technical and detailed summaries of standard methods used for CLSA, GC/MS analysis are detailed in Standard Methods for the Examination of Water and Wastewater (APHA-AWWA-WEF, 1992). The standard method indicates use of a sample temperature of 25°C, a carbon trap temperature of 45-50°C, purge time of 2 hours, and elution with carbon disulfide in three sequential extraction using 10, 10, and 5 µL volumes for a final extraction volume of about 25µL. An internal standard mixture of chloroalkanes added to the original one litre sample prior to purging allows for quantitation of the analytes of interest. The extract is then well suited for capillary gas chromatography using standard methyl silicone liquid phase columns.

Considerable study has gone into the standardization and optimization of the CLSA method. The first discussion of the closed loop stripping analysis method developed by Grob was a report of a method for transferring substances from water at room temperature onto an absorbent filter by a closed-loop air stream (Grob, 1973). Site specific applications of the CLSA system with reported concentrations of contaminants in the ng/L range were later reported (Grob and Grob, 1974). A comparative study of closed-loop stripping and liquid extraction techniques was also undertaken by Grob and co-workers. Factors compared included; overall sensitivity, substance specific sensitivity, quantitative reproducibility, routine use suitability, time requirements, turbid water samples, GC column life, and equipment availability (Grob, *et al.*, 1975). Detailed description of the equipment

Figure 2.1 Schematic of closed-loop stripping apparatus



and practical procedures for routine application of the closed-loop stripping apparatus was reported in 1976 (Grob and Zürcher). After these initial developments in Grob's laboratories, others opened the discussion of improving closed-loop stripping analysis contamination problems by using an open stripping system (Borén, *et al.*, 1982). These discussions centered around reducing the contaminant interference problems of CLSA, expanding the range of extractable compounds, and improving recoveries. Grob responded with a study outlining typical sources of contamination of the CLSA system and the carbon filters used and offered useful suggestions for cleaning of the system and the filters (Grob, *et al.*, 1984) and a short communication of results relating techniques used to extract compounds adsorbed to the micro-sized, activated carbon filter used for closed-loop stripping (Habich and Grob, 1984).

Methods of improving closed-loop stripping recoveries, including elevated stripping temperatures, prolonged stripping times and use of open stripping have been studied (Sävenhed, *et al.*, 1983). Optimum purging temperatures and desorption solvents were also studied (Borén, *et al.*, 1985) and results demonstrated that the use of open-loop stripping temperatures between 30 and 90°C could be used to meet specific analytical demands. In particular, stripping was most successful when performed at a temperature of 90°C except when very volatile compounds are being studied. This study also demonstrated that carbon disulfide with 10% acetone gave the best results for the combination of recovery and chromatography, and addition of a small percentage of a polar solvent such as methanol, was shown to increase recoveries of polar compounds such as phenols. Others also studied the optimization of closed-loop stripping efficiencies by varying purging times and temperatures and found that optimum recoveries were achieved after 2 hour purging at 40°C (Coleman, *et al.*, 1983). Hwang and co-workers (1984) reported the effect of carbon trap resistance, measured as flow, on recovery of 2-methylisoborneol (MIB), 2-isopropyl-3-methoxypyrazine (IPMP), 2-isobutyl-3-methoxypyrazine (IBMP), geosmin, and the chlorodecane standards and concluded that filters used for replicate analyses must have flows that are matched within 0.1mL/min. They also included a complete discussion of optimizing recoveries using the "salted" closed-loop stripping method of 100 g/L sodium sulfate additions prior to stripping.

Although the CLSA method was originally developed for use in the analyses of musty-earthy compounds such as geosmin and MIB, there were ongoing recovery and quantitation problems associated with the method. A summary of Henry's law constants for geosmin, MIB, IPMP, IBMP, and 2,3,6-trichloroanisole (2,3,6-TCA) was presented by Lalezary and co-workers in 1984. Results presented indicate that only the trichloroanisoles can be effectively removed using air stripping. This indicates that geosmin, MIB, IPMP and IBMP may have volatilities low enough to cause poor and inconsistent recoveries with CLSA which depends on air stripping of the compounds from the aqueous phase. This suggests that relative recovery factors should be used for accurate quantitation using the chloroalkane standards. Such factors have seldom been used and the CLSA method was generally acknowledged as semi-quantitative until Korth developed the synthesis of deuterated geosmin and deuterated methylisoborneol for use as internal standards with closed loop stripping (Korth, *et al.*, 1991; 1992). Internal standards with very similar Henry's law constants must be used for each compound being quantified (Kenefick, *et al.* 1993).

There has also been application-specific use of closed-loop stripping extraction for volatile compound analyses. The CLSA technique has been used as a tool in solving taste and odour problems at water treatment plants during seasonal off-flavour problems (McGuire *et al.*, 1981). A method for analysis of 2-methylisoborneol produced by cyanobacterial cultures has been developed (McGuire, *et al.*, 1983) and methods have been developed for modified CLSA analysis of geosmin, MIB, IPMP, IBMP, and 2,3,6-TCA from various matrices, including water, sediment and algal cultures (Krasner, *et al.*, 1983). Open stripping concentration methods were studied and a specific case of a chlorobleaching pulp mill discharge was used to demonstrate the source identification for odorous river water (Sävenhed, *et al.*, 1985).

2.3 PURGE AND TRAP

Another chemical method which has been used successfully for the analyses of odorous compounds is known as the purge and trap technique. The purge and trap method, like the CLSA method, is based on a combination of laboratory operations. The volatiles are purged from an aqueous solution, adsorbed onto a synthetic resin and then thermally desorbed onto the head of a capillary GC column. An outline of a purge-trap system combined with direct gas chromatographic analysis was presented by Bellar and Lichtenberg in 1974. This report described modification and assembly of commercially available components and early developmental uses for volatile compound analyses. The method has since been used in the standard protocols developed by the U.S.E.P.A. (methods 502, 503, 524, 601, 602. and 624) and also is used as a standard method for the analyses of trihalomethanes (APHA-AWWA-WEF, 1992). The purge and trap method is limited to nonpolar, low-molecular-weight, low water solubility, volatile compounds (Mallevialle and Suffet, 1987) and as such is not always suitable for the broad range of odorous compounds which may be problematic. Mallevialle and Suffet also report that the purge and trap method is not as sensitive as the CLSA method because much smaller sample volumes are extracted. The method is also limited to samples collected without headspace. In general, odour samples should be collected without headspace as grab samples. Cline and Severin (1989) reported that commercially available composite water samplers are inadequate for the collection of aqueous streams when the volatile component of these waters is of concern. Compounds with Henry's constants between 0.045 - 0.95 (dimensionless) were studied. Dynamic headspace purge and trap methods have also been used successfully for the extraction of odorous compounds from solid matrices (Ramstad and Walker, 1992).

2.4 SOLID PHASE ADSORBENTS

Extraction using various solid phase adsorbents is commonly used in the extraction of trace organics from aqueous samples. The most common solid phase adsorbents can be separated into two categories; activated carbon and synthetic resins. Junk and other researchers reported the use of porous polymer resins for the accurate analyses of organic solutes in water covering a concentration range of 50 mg/L to 20 ng/L (1974). Their report detailed an extensive study of proper resin clean-up, standard preparation, extraction pH, extraction solvent, drying of solvent after extraction, and concentration of eluate. Investigations into activated carbon adsorption of odorous compounds have often been the focus of treatment or removal studies, but the carbon can also be used as a concentration method in analytical work. Optimum conditions for concentration of MIB and geosmin through adsorption onto activated carbon were reported by Herzing and co-workers in 1977. It was found that most adsorbed compounds could be recovered by extraction with dioxane. Solution pH had no major effect on adsorption, but the presence of humic substances significantly reduced adsorption (Herzing *et al.*, 1977). Such solid phase extraction methods require solvent desorption and in order to obtain concentrated solvent extracts, the volume of water passed through the adsorbent must be relatively large. Often thousands of litres are used. Results of large scale (1300L) isolation of organics using resins and activated carbon, followed desorption by ethyl ether and dichloromethane respectively, demonstrated that more than 220 compounds could be extracted and identified (Infante, *et al.*, 1993). Jüttner (1988b) reported a specific application of solid phase adsorbent in the quantitative analysis of volatile organic compounds found in cyanobacteria. The method used involves a trap (containing the solid adsorbent) which is placed at the exit port of the aeration gas flow used during cyanobacterial culturing.

Many studies comparing the extraction efficiencies of various sorbents have been carried out. The results of a comparison of pre-concentration using one resin and one activated carbon in the isolation of organic compounds from water were reported by Chriswell, *et al.* (1977). Filtrasorb 300 activated carbon was compared to Amberlite XAD-2 resin in determining recoveries for 100 model compounds. Results indicated that the resin was superior to the activated carbon for isolating identifiable taste and odour compounds from water. In another comparative study, extraction recoveries for six adsorbents relative to Likens-Nickerson solvent extraction and continuous liquid-

liquid extraction were investigated (Blok, *et al.*, 1983). Solvent extraction methods were shown to give higher recoveries than the adsorbents as well as yielding lower levels of impurities than the adsorbents.

A recent development in solid phase adsorption may also be effective in taste and odour compound analyses. Pawliszyn and co-workers have developed the technique which is called solid-phase microextraction (SPME). It is a solvent-free sample preparation in which a fused silica fiber coated with polymeric organic liquid is exposed to the aqueous sample or the headspace above a sealed sample (Arthur, *et al.*, 1992a; 1992b; 1992c; Potter and Pawliszyn, 1992; Zhang and Pawliszyn, 1993). Organic analytes adsorb to the phase on the coated fiber and after adsorption equilibrium is reached, the fiber is inserted directly into the heated GC injection port where the analytes are thermally desorbed onto the head of the column. The method shows promise for the analysis of odorous compounds because SPME requires no separate extraction and concentration steps and all of the analytes extracted are transferred directly to the GC column.

2.5 GAS CHROMATOGRAPHY

A final consideration for the chemical methods of odorous compound extraction and analyses is the chromatographic separation and compound detection. Because of the low molecular weight and relative volatility of most taste and odour compounds, gas chromatography is generally used. Standard capillary chromatography columns are used almost exclusively and detection is possible with a mass selective detector (MSD) or a flame ionization detector (FID). In the analyses of these relatively volatile compounds, the injection technique is also of considerable importance. Grob and Grob give a valuable description of proper cool-on-column injection techniques that are very useful with volatile compounds (1978) and other studies have also detailed the benefits of the cool-on-column injection method for pg/μL levels of organics (Onuska, *et al.*, 1983). Wajon reported that cool-on-column injections were critical for the analyses of various dimethyl polysulphides. This study reported decomposition and disproportionation reactions with heated active surfaces such as a vaporizing injection port (Wajon, *et al.*, 1985a; 1985b). Without cool-on-column injection it has been suggested that a silylated glass wool plug in the injector insert results in improved quantitation of samples of volatile compounds due to improved transfer of the volatiles and retention of high boiling materials (Grob and Neukom, 1984).

3. SENSORY METHODS OF TASTE AND ODOUR COMPOUND ANALYSES

A very useful introduction, general discussion, and review of aquatic off-flavours is given by Persson (1983). In this basic outline he discusses the significance of off-flavours in water and fisheries management and emphasizes the need for integration of the sensory methods of analyses with more commonly accepted instrumental methods. An updated general discussion of the challenges associated with the study of aquatic off- flavours was again provided by Persson in 1988. The basic principles of this challenging area of study are outlined in a chapter on sensory analyses in Mallevialle and Suffet (1987). The sensory analyses methods have been established to accurately describe the taste and odour of a water or fish sample and then use this information to establish a relationship between the concentration of an organic contaminant and the intensity of the taste or odour of the samples as determined by a trained panel. Once this relationship has been established, it becomes possible to begin to define the cause of a taste and odour problem. Another detailed summary of the sensory assessment of water quality also critically reviews the earlier methods used in sensory analysis (Zoeteman, 1980) and again emphasizes the viewpoint that sensory analysis is best used as an screening step, to be followed by chemical analyses when further information is required.

The determination of the taste or odour of a water or fish sample is, by definition, a sensory analysis, and because it is generally thought that sensory analyses can only be carried out by humans, the analytical methods involved are often considered to be largely subjective. Persson (1983) objected to categorizing all sensory analyses as subjective because the sensation of an odour is an objective fact, but also acknowledged that there is a very large range of variation in sensitivity among individuals. A very detailed and lengthy review of the research from 1955 to 1975 that dealt with human perception and evaluation of the quality of water destined for uses which involve personal contact or consumption has been prepared (Bruvold, 1975) and the subjective nature of the determinations was constantly emphasized. Despite this controversy, a number of standard methods have been developed for the evaluation of taste and odour qualities and quantities in water samples. The standardized methods include; the threshold odour number test (TON), the flavour threshold test (FTT), the flavour rating scale (FRS), and flavour profile analysis (FPA) (APHA-AWWA-WEF, 1992). A lengthy review of the three standard methods for evaluating taste and odour with the FTT, FRS, and FPA has been published which describes the strengths and weaknesses of each test method. (Bruvold, 1989).

The basic chemical properties of what "makes" compounds odorous have been summarized in an early study by Boelans and co-workers (1978). The properties discussed include, volatility, hydrophilicity, lipophilicity, concentration, functional groups, chirality (size and shape), and molecular flexibility (rotation and vibration at molecular bonds). A report of odour similarities in structurally related odorants (Polak, *et al.*, 1978, 1992) indicated that seven alcohols have "earthy" odours similar to geosmin, but their slight differences were not easily described. An investigation into the possibilities of classifying odours based on physiochemical data was carried out in 1981. The odour classifications were found to be related to the polarizability of the molecule, the oil/water partition coefficient and the acid-base properties. The shape of the molecule was not a reliable indicator (Chastrette, 1981). Another early study into the relationship between molecular structure or physicochemical properties and odour intensity indicated that there was a direct relationship between hydrophobicity (octanol water partition coefficient) and odour intensity, but there was no such relationship with steric or polar parameters. (Greenberg, 1979).

3.1 THRESHOLD ODOUR NUMBER

The threshold odour number is related to the odour threshold concentration (OTC) which is the lowest concentration at which an odour can still be perceived. The threshold odour number is reported as the number of dilutions required to obtain the OTC (APHA-AWWA-WEF, 1992). This test is limited to aqueous samples. Compound specific taste and odour threshold data is listed in a number of different sources and has been listed by code number, alphabetically, by molecular weight, and by Wiswesser Line Notation permutation (Fazzalari, 1978) or alphabetically in compound class groups. (van Gemert and Nettenbreijer, 1977). There is also a collation of odour threshold data for approximately 450 chemical substances which is presented along with any reported irritation thresholds for humans. (Ruth, 1986).

Some of the limitations of this test include the very subjective nature of the test, the possibility of olfactory fatigue, and the fact that the results give an overall intensity and they fail to provide information useful for identifying the cause of the odour. Zoeteman (1980) expanded the list of shortcomings of this method and included neglect of sub threshold effects, an ascending dilution series may impair results, and results may be strongly influenced by the quality of dilution water.

The perception of odours varies significantly from person to person and to help overcome the subjective limitation of this method a panel of at least five people is suggested (APHA-AWWA-WEF, 1992). The threshold odour number reported is then an average. An early study summarizing the training of flavour panels to determine odour threshold concentrations for 32 chemicals (Baker, 1963) documented the large ranges which can be expected when using a number of panelists. The most sensitive panelist gave phenol a threshold odour concentration of 16 µg/L, while the least sensitive panelist had a threshold of detection of phenol at a concentration of 17 mg/L which is a thousand-fold range. While this finding presents a problem for an analytical technique it also reveals the reality that the perception of problems among public consumers will be highly variable.

Threshold odour and taste concentrations of geosmin and MIB were found to be functions of water temperature and residual chlorine concentrations but in general complaints from consumers were found to be related to the aqueous odour compound concentrations. (Ito, *et al.*, 1988). The TON has been used to direct corrective action in water treatment plants and a study in Japan demonstrated that there was a log-log relationship (Stevens model) between the threshold odour number and the odorant concentration for geosmin and MIB (Tomita, *et al.*, 1988). However, the TON values were again found to vary remarkably with water temperature and the presence of masking compounds. Another study using TON analyses reported that the intensity of the muddy odour compounds as perceived by a panel was proportional to the logarithm of the concentrations. (Persson, 1980). Mallevialle and Suffet (1987) report that the TON can not be used alone to direct operations in a water treatment plant and suggest that a combination of different sensory methods is the most effective.

The problem of olfactory fatigue is often compound specific and cannot be predicted. Krasner, Hwang and McGuire (1981) have stated that, in general, most types of sensory analyses are prone to fatigue. The strength, character and odour threshold values for a series of chloroanisoles was studied (Griffiths, 1974) and their results demonstrated a rapid odour fatigue with substitution of chlorine in the 2- and 6- positions on the ring. The fact that the TON gives a measure of total intensity and cannot be used for compound specific characterizations is unavoidable, but in many applications the TON is still a useful value. The "total odour" is often more characteristic than any of the separate odour compounds present. A study has shown that the original complex odour of certain surface water samples may not be re-created after the odorous compounds have been isolated and recombined (Lundgren, *et al.*, 1988).

3.2 FLAVOUR PROFILE ANALYSIS

In the flavour-profile analysis (FPA) method, a panel consisting of at least four members describe the character and intensity of taste and/or odour of a sample. This method is not limited to water and is often used in the food industry (Krasner, *et al.*, 1985; ASTM 1968). The basics of the test method have been listed in a number of sources (ASTM, 1968; Bartels, *et al.*, 1987; Krasner, 1988; Suffet, *et al.*, 1988; Levi, *et al.*, 1990) and these have been standardized in "Standard Methods" (APHA-AWWA-WEF, 1992). Briefly, a 60 mL sample for each member of the panel is placed in a 250 mL borosilicate, Erlenmeyer flask with a ground glass stopper. The samples are brought to room temperature by heating in a 45°C water bath for 15 minutes. The panelist meet in a clean odour-free room and are not allowed to smoke, eat or drink for 30 minutes prior to the testing. Each panelist swirls the sample slightly, brings the flask near the nose and then removes the stopper. The character and intensity of the odour is then determined and recorded.

One of the greatest limitations of this method is the varying descriptors used to describe the same odour. The Taste and Odour Committee of the International Association on Water Pollution Research and Control (IAWPRC) has prepared a "flavour wheel" for characterizing natural waters (Mallevialle and Suffet, 1987). The initial flavour wheel used is shown in Figure 3.1. A discussion of the further development of the "flavour wheel" for the classification and identification of odours in flavour profile analysis (Suffet, *et al.*, 1988) presented a more simplistic wheel which is shown in Figure 3.2. and the finalized version now used is presented in Figure 3.3. The compounds listed outside the wheel are some possible causes of the descriptors shown. However, these lists are not intended to be complete, given the large number of odour-causing chemicals possible.

Flavour profile analysis data have been evaluated for the quality of the qualitative descriptions and the quantitative response for each descriptor. Qualitative descriptions are more consistent after training of panelists using the common language of an odour wheel. Quantitative responses were quite variable and the use of replicates was recommended. (Meng and Suffet, 1992). Other lists have been prepared including one quite extensive list of 126 different odour descriptors corresponding to 1573 organic compounds that can serve as a standard set to be used by flavour panels (Abe, *et al.*, 1990). This still may not eliminate the subjective nature of the sensory analyses, as indicated by a study which suggests a very limited association between odours and their verbal descriptors, especially when choosing between similar odour descriptors. (Engen, 1987).

When establishing a flavour profile panel, the panel must be trained to describe the same odour in the same way. General discussion of the feasibility of developing a standardization of sensory analysis in the water treatment industry by using a panel well-trained in the flavour panel analysis method was discussed by Bartels and co-workers (1986) and demonstrated that the selection and training of panelists is the most important aspect to be considered if a flavour panel is to be relied upon. In order to facilitate rapid panelist selection and training, a series of 19 common odorants are now available as a test kit for odour type and strength comparisons. The squeeze-bottles test kits are prepared using polypropylene bottles with flip-top caps, containing the chosen odorant dissolved in virtually odorless mineral oil which is absorbed in polypropylene felt (Amoore, 1992).

A few studies have been done with flavour panel methods other than FPA. Sensory panel test results have been reported for site specific evaluations of the tainting potential of two pulp mill discharges. Results obtained using a triangle test method of comparison indicated that there was no tainting potential at one site, and there was limited tainting potential for one species of fish at another site (Jardine, 1992). Another flavour panel study into the tainting potential of twelve select compounds found in oil sands wastewater indicated that the trained sensory panel found three compounds (naphthalene, benzothiophene, and 2,5-dimethylphenol) to have the lower odour thresholds and a strong capability for causing taint in fish (Jardine, and Hrudey, 1988).

Figure 3.1 IAWPRC Proposed Flavour Wheel (Mallevialle and Suffet, 1987).

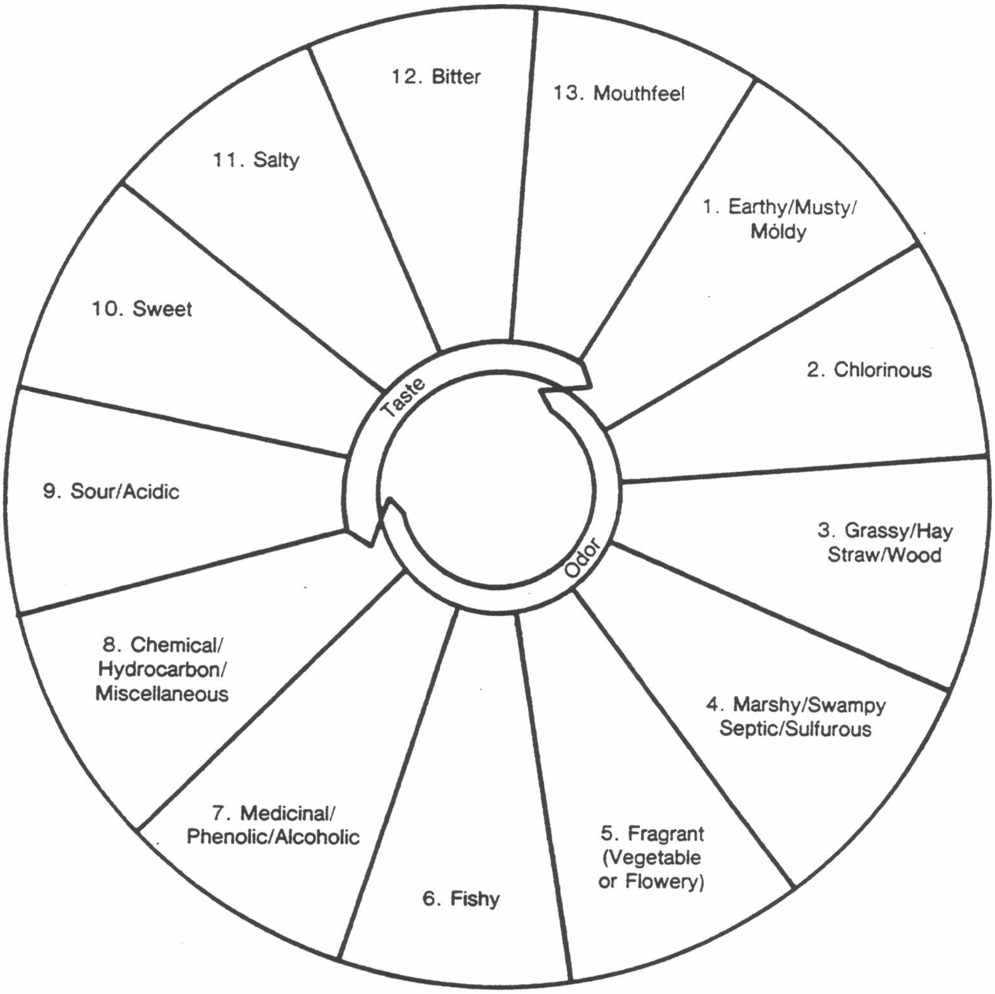


Figure 3.2 Flavour Wheel as Suggested by Suffet *et al.* (1988).

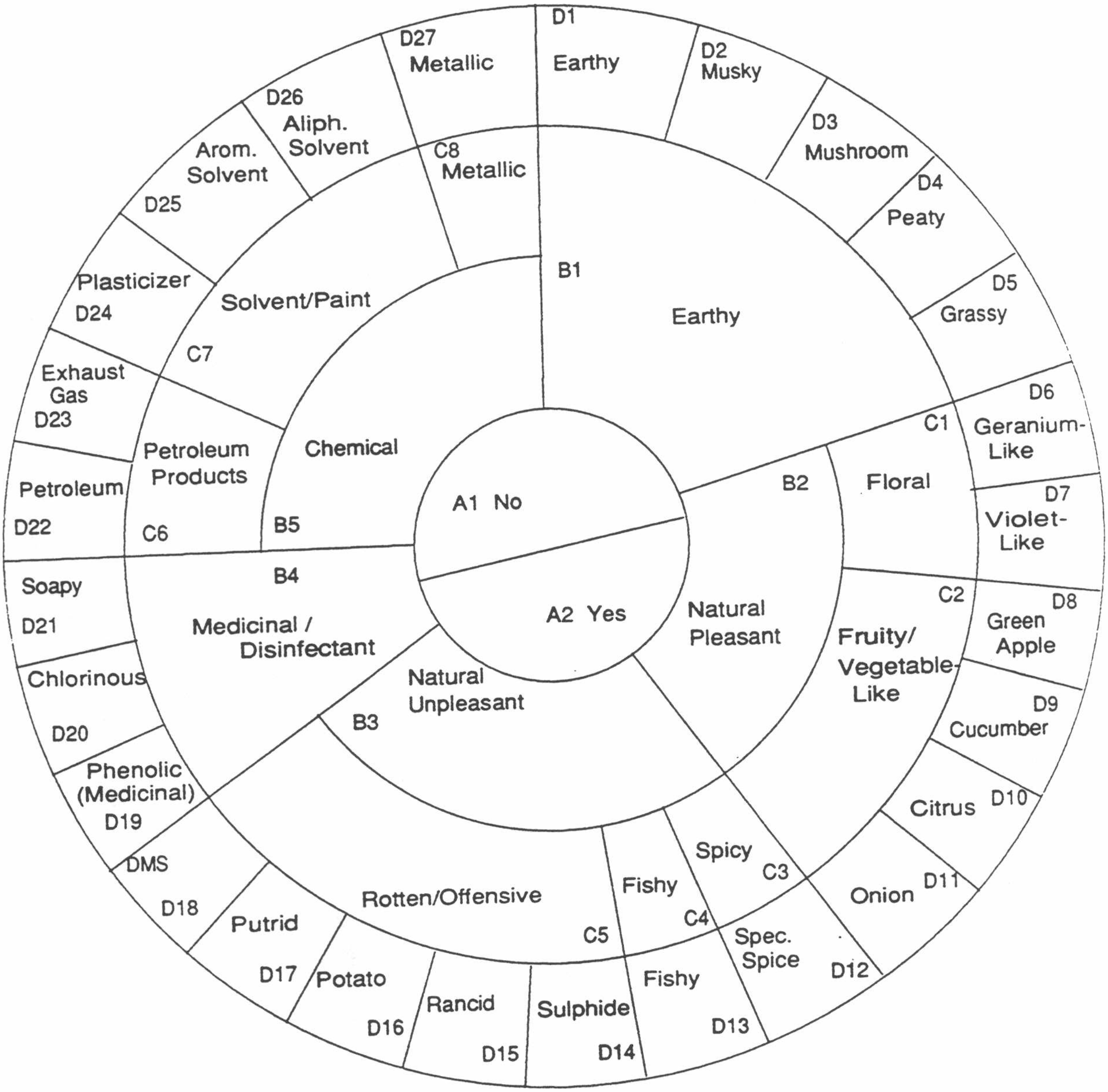
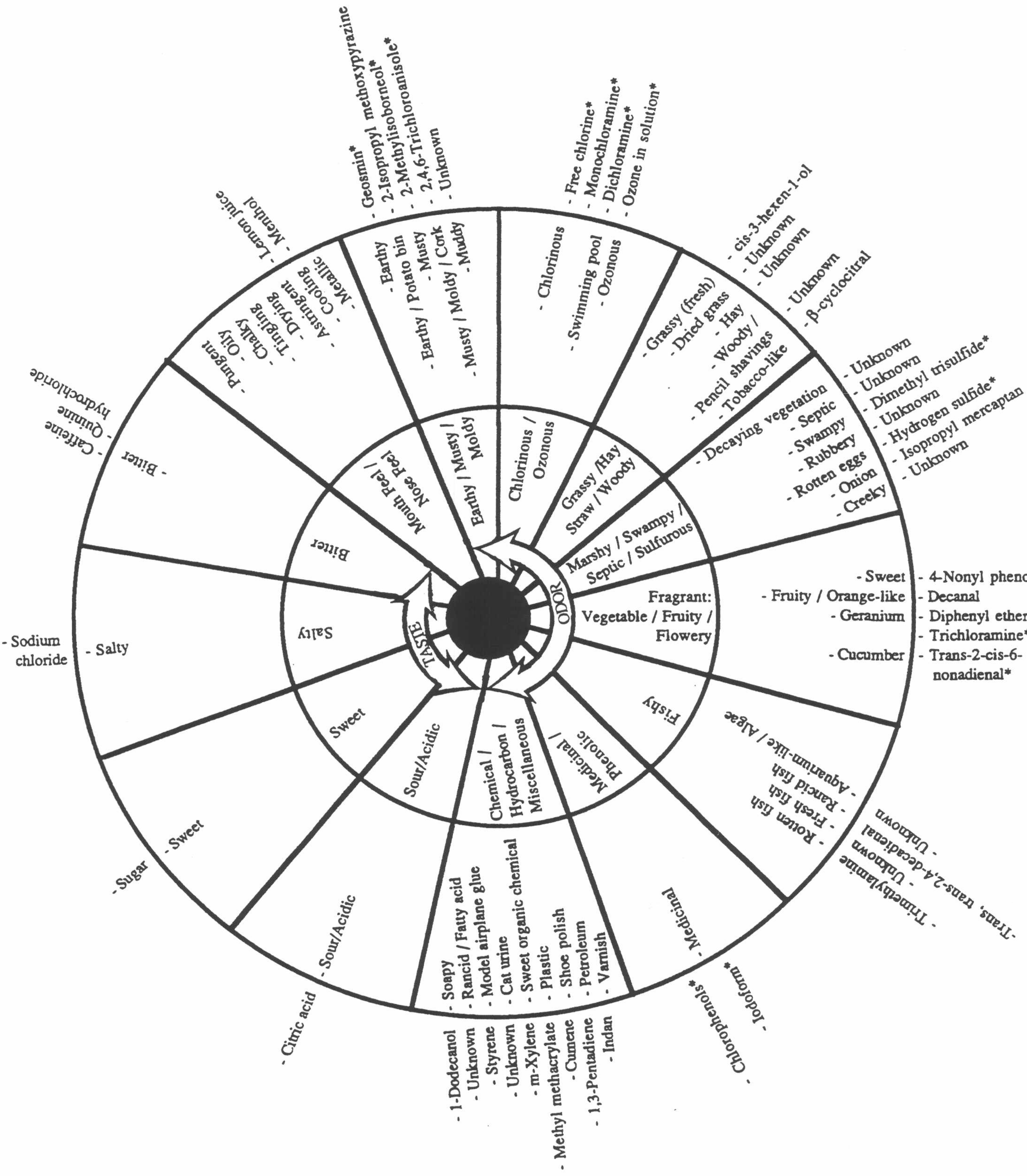


Figure 3.3 Current IAWPRC Flavour Wheel



3.3 OLFACTORY GAS CHROMATOGRAPHY

Most researchers in the field of taste and odour analyses recognize the utility of combining both sensory and chemical methods of analyses in the study of a specific odour problem. There has been general agreement that the sensory analysis provides a qualitative description of the taste and odour of the water or fish and the chemical analyses can provide an indication of the presence of specific chemicals and the quantities they are present in (Suffet, *et al.*, 1988). However, in order to confirm that specific chemicals present are responsible for specific odours in the sample requires a confirmatory combination of the sensory and chemical techniques. Without the parallel determinations, it is only a presumption that certain compounds are responsible for certain odours. This is outlined clearly in the triangular schematic diagram of relationships between instrumental and sensory techniques given by Meng (1991) are shown in Figure 3.4. In this schematic the combined chemical and sensory analyses are referred to as sensory GC, but it has also been called olfactory GC (OGC) analysis. The basic principle behind OGC is the use of the human nose as the detector at the outlet of a GC column. OGC has been used in the food industry and the perfume industry (Khiari, *et al.*, 1992) and has since been developed for specific use in the analysis of extracts prepared from odorous water or fish (Zoeteman and Piet, 1972/73; Meng, 1991; Khiari, *et al.*, 1992, Veijanen, 1990). This sensory GC analysis is carried out by having a trained operator continuously smell the effluent at the exit port of a capillary GC system while recording the odour descriptors and intensities detected with corresponding retention time. A parallel GC system with an FID or mass selective detector is used to instrumentally determine the compounds present in the sample. Figure 3.5 shows the comparison of an FID chromatogram and an "odourgram" obtained using OGC. In this case a standard mixture (200 ng/L) of odorous compounds was extracted using CLSA and then analyzed with sensory GC and GC/FID. In one summary of the development of a sensory gas chromatography procedure the researchers reported that they were able to quantitatively evaluate ng/L concentrations of mixtures of odorous compounds. Their method allowed for the fractionation of a mixture of odours and a corresponding description and quantitation of each odour (Khiari, *et al.*, 1992). They also reported that the sensory GC method was used to detect compounds present at levels of 20 ng/L while the FID detector showed little response (see Figure 3.6). It was noted that a correlation between sensory panel response and OGC results for compounds present near thresholdodour concentration levels was not always apparent. This was reported to be due to concentration differences of the chemicals in the mixture (masking) and synergistic or antagonistic effects.

The sensory GC analyses allows for the description of separate odours in a sample whereas the FPA type of analyses allowed only the description of the combined odour character of a sample. With OGC, chemical and sensory methods have been combined into an improved technique for the source identification of odorous compounds in drinking water. The combination of chemical and sensory methods was also used as a technique for the source identification of odorous compounds in river water. It was a chlorobleaching pulp mill discharge which was found to cause a significant increase in the odour of the river water (Sävenhed, *et al.*, 1985). Other site specific applications of gas chromatography / mass spectrometry combined with sensory odour analysis methods in the identification of tainting substances have also been reported (Veijanen, *et al.*, 1988; Hrudey *et al.*, 1992). Preparative gas chromatography for fractionation of odour compounds in extracts has been studied as well (Lundgren, *et al.*, 1989). In this study it was reported that the original complex odour of certain surface water samples may be re-created from dichloromethane extracts but not from the corresponding stripping extracts. The Principal Component Factor Analysis method (Meng, *et al.*, 1992) has been used to demonstrate the correlation between sensory response and chemical concentration in water. However, OGC has not yet been developed into a standardized method.

Figure 3.4 Schematic Diagram of Relationships Between Instrumental and Sensory Techniques (Meng, 1991)

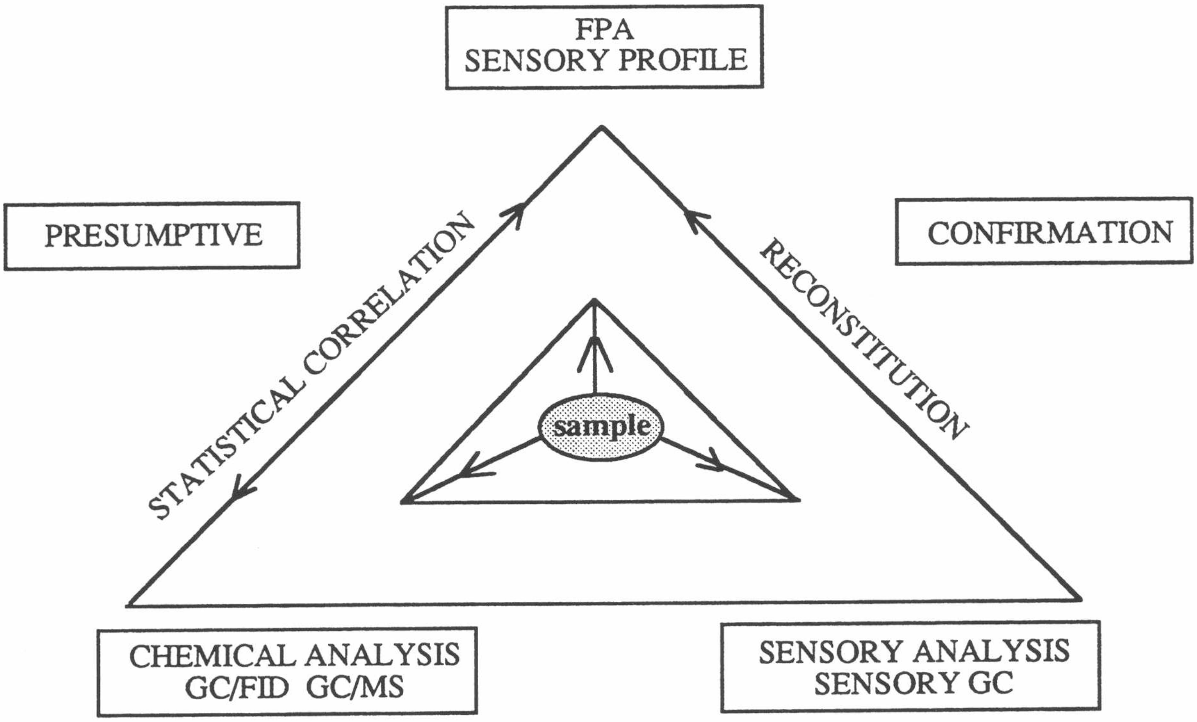


Figure 3.5 Comparison of an FID chromatogram and "odourgram" of a CLSA extract of a synthetic mixture (200 ng/L each) (Khiari, *et al.*, 1992)

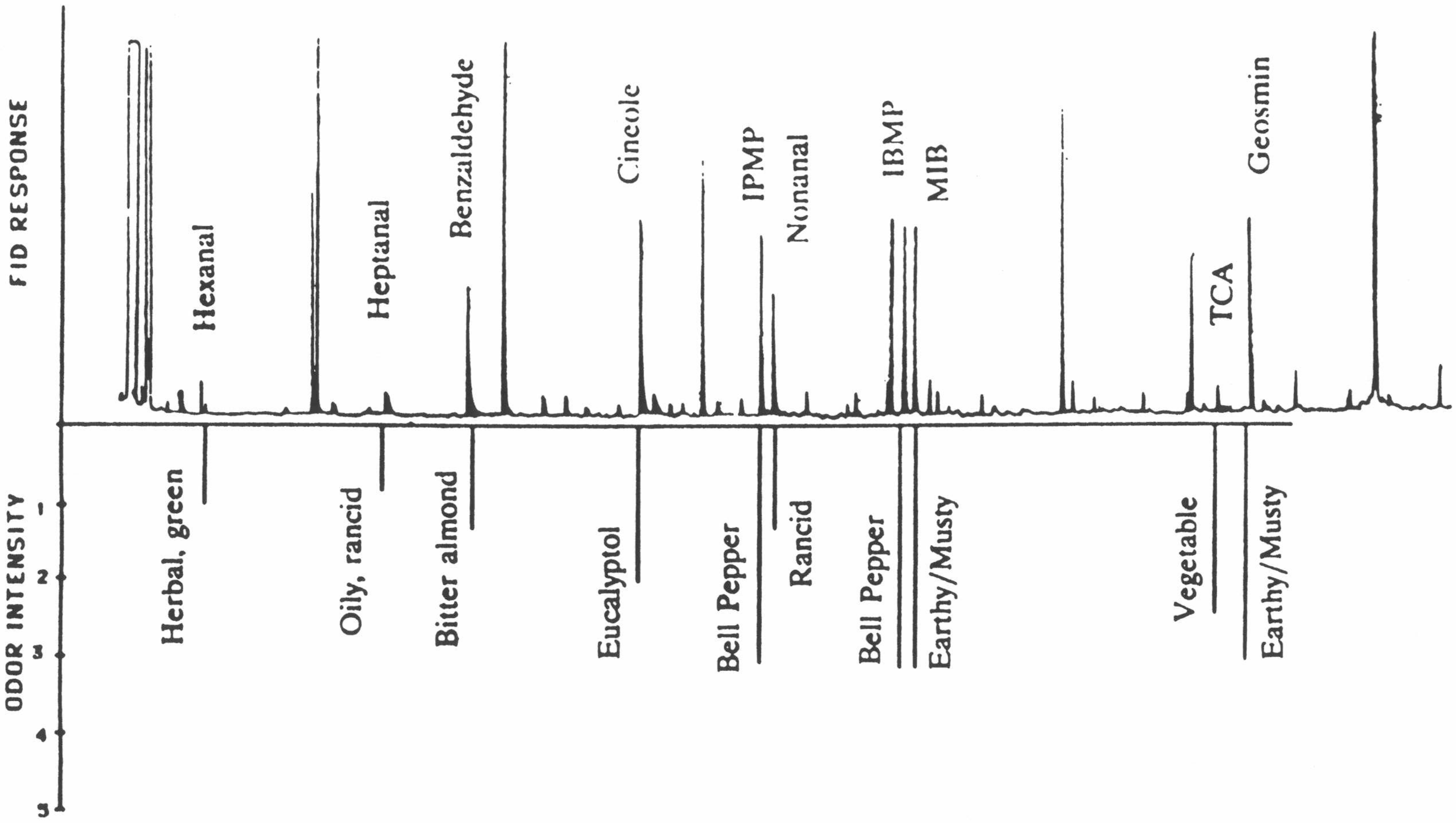
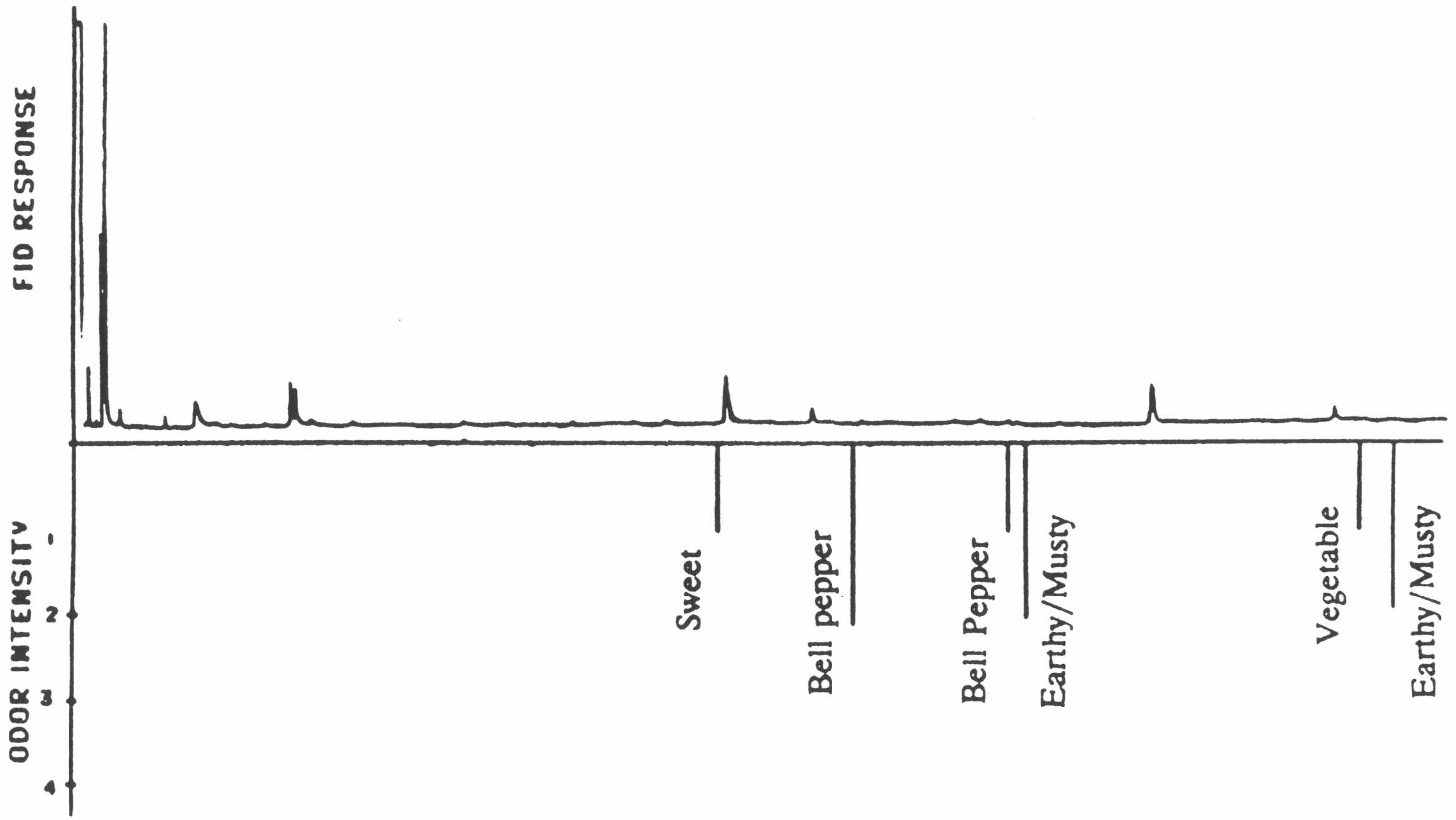


Figure 3.6 Comparison of an FID chromatogram and "odourgram" of a CLSA extract of a synthetic mixture (20 ng/L each) (Khiari, *et al.*, 1992)



3.4 OTHER

There have been a number of attempts at the development of instrumental detectors which could "sense" odours. One article reports the use of semiconductor gas-sensor elements for automated odour detection. Odours detectable were limited to ethereal, ethereal-minty, ethereal-pungent and pungent substances (Abe, *et al.*, 1987). Another report gives a general discussion of the various instrumental methods used for the detection of odorous compounds. These include the "Warwick Nose" array of SnO2 sensors and the Naval Research Laboratory surface acoustic wave sensors for chemical warfare gases. (Newman, 1991). None of the detectors are used routinely in taste and odour analyses as they do not have adequate sensitivity to compete with the nose.

4. TAINTING OF FISH

Bleached kraft pulp mill effluent and discharges related to other industrial developments are known to contain odorous compounds that have the potential to cause off-flavours in fish. There have been many complaints about tainted fish caught in the NRBS areas over the years (see Appendix 1 & 2), but the current state of the problem is not as well defined. In a personal communication with Sonny Flett, the NRBS Traditional Knowledge group leader, it was apparent that although there have been complaints of tainted fish being caught downstream of industrial discharges, in general native people are no longer fishing the Athabasca River because the taste of the fish is often quite unpalatable and they are not able to find a market for the fish (see Appendix A1). The accidental release of oily substances from the Suncor Oil Sands Plant during the winter of 1981/82 that caused the tainting of fish (petroleum taint) is still remembered by people and they avoid fish from the Athabasca River. Other discussions with people from near Garden River also indicated that in general people no longer eat the fish caught in the Peace/Wapiti river systems because of the long history of unpalatable fish caught there.

In other anecdotal historical information obtained in discussions and correspondence with various regional fishery biologists in the Fish and Wildlife Division of Alberta Environment it was again apparent that in general people do not eat the fish caught on the Wapiti, Smoky or Peace Rivers. Over the years there have been a number of warnings in fishing guides and in news releases about the safety of consuming fish species caught in waters in the NRBS area, and it seems that people no longer consume fish from these waters (see Appendix 1 & 2). Where the fish are still eaten, Alberta Fish and Wildlife officers receive a few complaints of tainted fish each year. In discussions with various informed sources it rapidly became apparent that the tainting of fish by anthropogenic sources such as pulp mill discharges is a real concern and a consequence of expectations of tainting from prior experience.

4.1 PULP MILL EFFLUENTS

There have been incidents of tainted fish that were thought to have been tainted by exposure to pulp mill effluents. Thomas (1973) has documented many cases of the tainting of fish flesh by chemicals or effluents, including effluents from the paper industry. A good review of the sublethal effects of untreated and biologically treated bleached kraft mill effluents in freshwater fish in Canada was published by Kovacs in 1986. The summary includes a section reviewing literature surrounding tainting of fish attributed to pulp mill discharges. Another review requested by Environment Canada in 1987 (McLeay and Associates Ltd., 1987) also summarizes the published literature relating to the aquatic toxicity of pulp and paper mill effluents and includes a section on off-flavours in fish. A summary of the findings of these review articles is tabulated in Table 4.1. and 4.2. A more recent study by Jardine (1992) provides a summary of the public evaluation of fish tainting from various pulp and paper mill discharges. This study demonstrates that although one mill discharge still caused fish flesh tainting, another mill discharge no longer results in fish tainting.

A detailed study undertaken by Domtar Fine Papers (1971) was set up to study the effects that various pulp mill effluents had on the taste and odour of water and fish. Their objectives included locating the sources of tainting and determining practical treatment options to protect the receiving waters and the fish therein. The paper company reports results of a detailed sensory evaluation of the effects of pulp and paper mill effluents on tainting of fish. Very basic sensory evaluations methods were used, but a study of the tainting potential of all process streams was carried out. Their study found that the kraft mill evaporator condensates, digester primary and secondary foul condensates and the recovery furnace flue gas condensates were the main sources of tainting compounds. In a sulphite plant, the sulphite waste liquor was found to be a major contributor to the odour of receiving waters, but was not implicated in the tainting of fish. The study was also documented by Cook and co-workers (1973) in a more concise technical paper. Tainting was evaluated in caged perch exposed to specific components of kraft mill effluents (Farmer, *et al.*, 1973). The study found that off-flavours were found in fish exposed to evaporator condensates, foul water condensate, and the recovery furnace flue gas condensate. However, environmental exposure studies with fish taken from downstream of the total effluent showed no flavour tainting when compared to fish taken upstream of the effluent, but such findings are difficult to interpret without knowledge of receiving water dilution rates or mixing characteristics. A study of biological treatment methods demonstrated that jack coho salmon exposed to untreated kraft mill effluent for 96 hours demonstrated measurable flavour impairment while salmon exposed to biologically treated KME for up to 96 hours did not demonstrate any flavour impairment at levels of 2.9% by volume (Shumway and Chadwick, 1971).

The compounds responsible for these tainting incidences have not been well established, although alkylphenols, thiophenol (Heil and Lindsay, 1988a), chlorophenols, guaiacol and catechol (Paasivirta, *et al.*, 1987), chlorinated acids, phenols (Paasivirta, *et al.*, 1983), organochlorine compounds (Vuorinen, *et al.*, 1985), chloroanisoles, and veratroles (Brownlee, *et al.*, 1993; Paasivirta, *et al.*, 1987; 1992) have been implicated as possible fish-tainting compounds which persist downstream of pulp bleaching effluent discharges. Microbial metabolism of chlorophenols resulted in the formation of chloroanisoles which are even more likely to cause fish-tainting (Paasivirta, *et al.*, 1983).In a series of site specific studies of fish tainting in Wisconsin, Lindsay and Heil (1992; Heil and Lindsay, 1988c) reported that the compounds responsible for the pronounced off-flavours in Walleye pike and other sport fish in the Upper Wisconsin River were alkylphenols (2-isopropylphenol, 3-isopropylphenol, 4- isopropylphenol, 2,4,-diisopropylphenol, 2,5-diisopropylphenol, 3,5-diisopropylphenol, carvacrol and thymol) and thiophenol. The research also demonstrated that the food chain was involved in the transport of the alkylphenols from the environment to the fish (alkylphenols and thiophenols were found in the river sediment and benthic organisms) (Heil and Lindsay, 1988a). Sources of the alkylphenols and thiophenols were studied

Table 4.1 Reported Studies of Off-Flavours in Fish as a Result of Pulp Mill Discharges (adapted in part from McLeay and Associates, 1987 and Kovacs, 1986)

Effluent type	Receiving water type	Fish Species	Distance from outfall (km)	Significant Off-flavour	Reference
**BSME	river	salmon	-	yes	Berg, 1983
KME	lake, river	pike, cod, perch	-	yes*	Kuusi & Suihko,1983
BKME	river	perch	-	no	Liem, *et al.*, 1977
BKME/MPE	lake	whitefish whitefish	1.5 10	yes no	Wells 1967
BKME/MPE	river	pike, pickerel	-	yes	Swabey, 1965
BKME	river	trout, whitefish	-	yes*	Langford, 1974
BKME/MPE /**BSME	river	pike	-	yes*	Weinbauer, *et al.*, 1980
BKME	river	trout	<1	yes*	Langer & Nassichuk, 1975
BKME	river	whitefish	-	yes*	Kovacs, 1982
KME	lake	perch, bream pike, roach	0-17.5	yes	Persson, 1988
BKME	river	trout	1	no	Jardine, 1992
BKME	river	trout	2	no	Jardine, 1992
BKME	river	perch	12	yes	Jardine, 1992

BKME - bleached kraft whole mill effluent
**BSME - bleached sulphite whole mill effluent (old process never used in Alberta)
KME - kraft mill effluent
MPE - mechanical pulping mill effluent
* tainting was not or was not demonstrated to be attributable to pulp mill discharge(s)

Table 4.2 Induction of Off-Flavours in Fish by Controlled Exposure to Pulp and Paper Whole Mill Effluents (adapted in part from McLeay and Associates, 1987)

Effluent	concentration (%v/v)	Fish species	Exposure time (days)	Significant off-flavour	ETTC* (%v/v)	Reference
UKME (untreated)	0.2-2.6	salmon	3-4	yes	1.5	Shumway Chadwick, 1971
UKME (biotreated)	0.7-2.9	salmon	4	no	>2.9	Shumway Chadwick, 1971
BKME/ **BSME	0.3	perch	44	no	>0.3	Cook *et al.* 1973
BKME/ **BSME	5-10	perch	7	yes	>5<10	Cook *et al.* 1973
KME	1.4-100	trout	2	yes	>8<20	Shumway Palensky, 1973
**SME	1-67	trout	2	yes	>34<51	Shumway Palensky, 1973
BKME/MPE	3-18	trout	2	yes	3	Whittle Flood, 1977
BKME/MPE	2-3	trout	6	yes	3	Whittle Flood, 1977
BKME	0.3-80	trout	4	yes	5	Liem *et al.* 1977
BKME	0.1-10	trout	4	yes	1	Liem *et al.* 1977
BKME	0.1-10	perch	4	yes	5	Liem *et al.* 1977
BKME	0.5-4	trout	0.3-2	no	>4	Langer Nassichuk, 1975
BKME	0.1-4	trout	0.2-1	yes	0.2-0.8	Gordon *et al.*, 1980
BKME	0.7-10	trout	0.2-1	yes	2.0-2.9	Gordon *et al.*, 1980

UKME - unbleached kraft whole mill effluent
BKME - bleached kraft whole mill effluent
KME - kraft whole mill effluent (not indicated if bleached or not)
**SME - sulphite whole mill effluent (old process never used in Alberta)
**BSME - bleached sulphite whole mill effluent (old process never used in Alberta)
MPE mechanical pulping effluent
*Estimated threshold tainting concentration (lowest %v/v where significant taint noted)

and it was found that degraded aspen wood is capable of contributing to alkylphenol concentrations. Strong flavour tainting was found in trout exposed to 100 µg/L concentrations of five added alkylphenols and thiophenols. Mixtures of 1-2 µg/L concentration of the same compounds did not result in flavour tainting (Heil and Lindsay, 1988a).

Caution is necessary when interpreting compound-specific research results. The identification of specific compounds is often limited by analytical methods and sample interferences. Consequently, the tendency of various compounds like the chlorophenols or alkyl phenols to be reported in fish tainting studies does not assure that these identifiable compounds were primarily responsible for causing the taint. One study by NCASI (1973) into the significance of phenolic substances in pulp mill effluents found that such chlorinated phenolics were not the cause of taste and odour in pulp mill effluents.

Investigation into tainting of fish flesh downstream of pulp mill discharges in the NRBS area has shown few documented tainting incidents, although the public perception appears to indicate that the problem has long been recognized. After receiving a number of public complaints a study of fish tainting potential was undertaken downstream of the Proctor and Gamble Mill on the Wapiti River was undertaken in 1990. The study results indicated that the pulp mill effluent resulted in fish tainting, but a cause/effect tainting relationship could not be established with chemical analyses (Millson, 1993). This study also highlights some of the limitations of a short term study without seasonal variation. The caged fish studies were intended to be carried out prior to the high flow of spring runoff but ended up being affected by the spring high flows. In contrast, tainted fish complaints were received during low, winter flows. Fetterolf (1963) noted that complaints about tainted fish caught from lakes that received pulp mill and other industry wastes were highest during the winter months but that most off-flavours in fish taken from rivers receiving similar wastes occurred in the spring Heil and Lindsay found that the anthropogenic alkylphenols and thiophenols were identified as the principal contributors to flavour-tainting in fish during low flow, while naturally occurring geosmin and MIB were major contributors to off-flavours in fish captured during high flow. Fish from a section of the Upper Wisconsin River that is influenced by several pulp and paper mills, municipalities and other industry were found to be tainted (Heil and Lindsay, 1988b, 1988c, 1988d). Alkylphenols and thiophenols were identified as the principal contributors to flavour-tainting in fish during the spring, while geosmin and MIB were major contributors in fish captured in the late summer (Heil and Lindsay, 1988a). Similar naturally occurring, seasonal odour events during spring run-off, high flow conditions have also been observed on the North Saskatchewan River (Hrudey, *et al.*, 1992). Although there have been other recorded complaints of tainted fish being caught downstream of pulp mill discharges in the NRBS area (see Appendix 1 & 2) there is little documented information on the study of these problems.

A number of researchers have also documented findings from treatment studies in which the removal of fish tainting substances was studied. One set of treatment studies into the effect of various treatment methods on the fish tainting propensity of bleached kraft mill effluents indicated that the most effective effluent treatments, in increasing order of value, were turbulent contact absorber (to provide a counter-current flow of gas and liquid), activated sludge (with clarification and activated sludge recovery) and aerated lagoon (low intensity) (Farmer *et al.*, 1973). Another study found that biotreatment did not reduce the tainting propensity of bleached kraft whole mill effluent as effectively as it reduced BOD and acute toxicity. Apparently some of the constituents in BKME responsible for fish flesh tainting are not readily biodegradable (Gordon, *et al.*, 1980). Treatment studies by Cook and co-workers (1973) indicated that extensive aeration and steam stripping as well as aerated lagoons and activated sludge processes were somewhat effective in removing compounds present in BKME that are responsible for taste and odour problems. However, the aeration and stripping processes transfer the odorous compounds into the atmosphere and general odour complaints in the vicinity of the treatment facilities are likely.

4.2 NON-PULP MILL EFFLUENTS

There have been a large number of instances where fish flesh has been tainted after the fish have been exposed to non-pulp mill types of contaminants in the aquatic environment. A number of industrial discharges or non-point source run-off could result the introduction of substances capable of leading to off-flavours in fish, but the scale of such activities is relatively limited in the NRBS areas. The NRBS effluent summary given in Appendix 3 shows that sewage effluents predominate with only 5 industrial, non-pulp mill effluents. However, a recent review reported that bioaccumulation can lead to higher concentrations of some tainting compounds in fish flesh than in the water (Hrudey and Nelson, 1986) so small scale discharges may still result in tainting depending upon exposure conditions. In the relevant literature the most common cause of fish tainting is exposure to oil or petroleum types of contamination. Stansby (1978), Malins and Hodgins (1981), McIntyre (1982), Motohiro (1983), Persson (1984), and Vandermeulen (1986) summarized such instances in reviews of tainted fish caused by petroleum compounds. Another review of literature specifically relevant to the tainting of fish by petroleum-derived compounds found in oil sands extraction and upgrading process wastewaters has also been prepared (Hrudey and Nelson, 1986). In this review, it was reported that tainted fish from the Athabasca River in 1982 were found to contain detectable levels of a range of polynuclear aromatic hydrocarbons, and polynuclear aromatic sulphur heterocycles, but a direct correlation between the presence of these compounds and the sensory taint was not established. Public complaints were received after a release of oily substances greatly in excess of licensed wastewater discharge limits from the Suncor Oil Sands Plant during the winter of 1982 (Appendix 1 & 2) and further investigations were carried out to determine what compounds were responsible.

Jardine and Hrudey (1988) investigated the tainting potential of a number of individual compounds associated with oil sands extraction and upgrading operations. The selected contaminants were spiked in walleye flesh and the fish were then screened for tainting by a trained sensory panel. The panel found that, of the compounds tested, naphthalene, benzothiophene and 2,5-dimethylphenol had the lowest detection thresholds and therefore demonstrated the strongest capability for causing an off-flavour in fish. In another study of the specific tainting potential of the oil sands wastewaters, rainbow trout were exposed to four different tailings pond wastewaters for a period of 24 hours, followed by sensory panel evaluations (Koning and Hrudey, 1992). The flesh of the fish was found to be tainted after exposure to any of the four wastewaters. Chemical analyses of the tainted fish flesh revealed the presence of alkylated benzenes and phenols well above reported odour thresholds for these compounds. These researchers also identified organic sulphur compounds in the wastewater which may also have contributed to the taint of the fish, but these sulfur compounds were not resolved in fish flesh analysis, so a correlation could not be confirmed. This was attributable to analytical difficulties in isolating and identifying these sulfur compounds in tissue extracts. However, there was a correlation between the levels of organosulfur compounds in exposure waters and the level of taint detected.

In another laboratory study rainbow trout were exposed to several different concentrations of diesel using a flow through system with the used water discharged to waste. The threshold of exposure concentration which led to tainting by diesel fuel was found to be 0.08 mg/L (Davis, *et al.*, 1992). Caged fish have been used to study the tainting potential of a large number of industries which discharge into the Ohio River (Thomas, 1973). It was reported that in this study region petroleum related industries did not discharge substances that produced off-flavours in fish, while paper industry discharges did. The sewage treatment plant discharge on the Ohio River caused an off-flavour in caged catfish immediately downstream and caused extreme tainting 1.6 km downstream. Industries producing chemicals or related products resulted in tainting of fish for the longest distances downstream of their discharges. Wastes discharged from metals industries or synthetic rubber production also resulted in an off-flavour in the test fish. In this study, effluent regulations were not at all strict and effluents not only caused tainting, but in many cases were also toxic and therefore are not completely relevant under current effluent regulations. Another multieffluent study was carried out in Finland from 1969 to 1981 (Kuusi and Suihko, 1983). In this study, only fish suspected of being tainted were accepted and only the character of the taint was reported. The flavour panel found large numbers of fish that were described as tainted by oil, kraft pulp mill effluent, or sewage types of off-flavours. Shumway and Palensky (1973) reported that municipal sewage effluents induced off-flavours in fish. An early study by Roubal and co-workers (1978) reported that low molecular weight aromatic hydrocarbons from crude oil accumulate in both coho salmon and starry flounder. They also found that the demersal (bottom dwelling) flounder accumulated more hydrocarbons than the salmon and that the salmon depurated within one week in clean water while the flounder retained detectable levels of substituted benzenes and naphthalenes over the same time period. Tidmarsh and Ackman (1986) studied fish tainting by petroleum based hydrocarbons and found that organisms present in shallow protected waters where circulation was limited were more likely to be tainted.

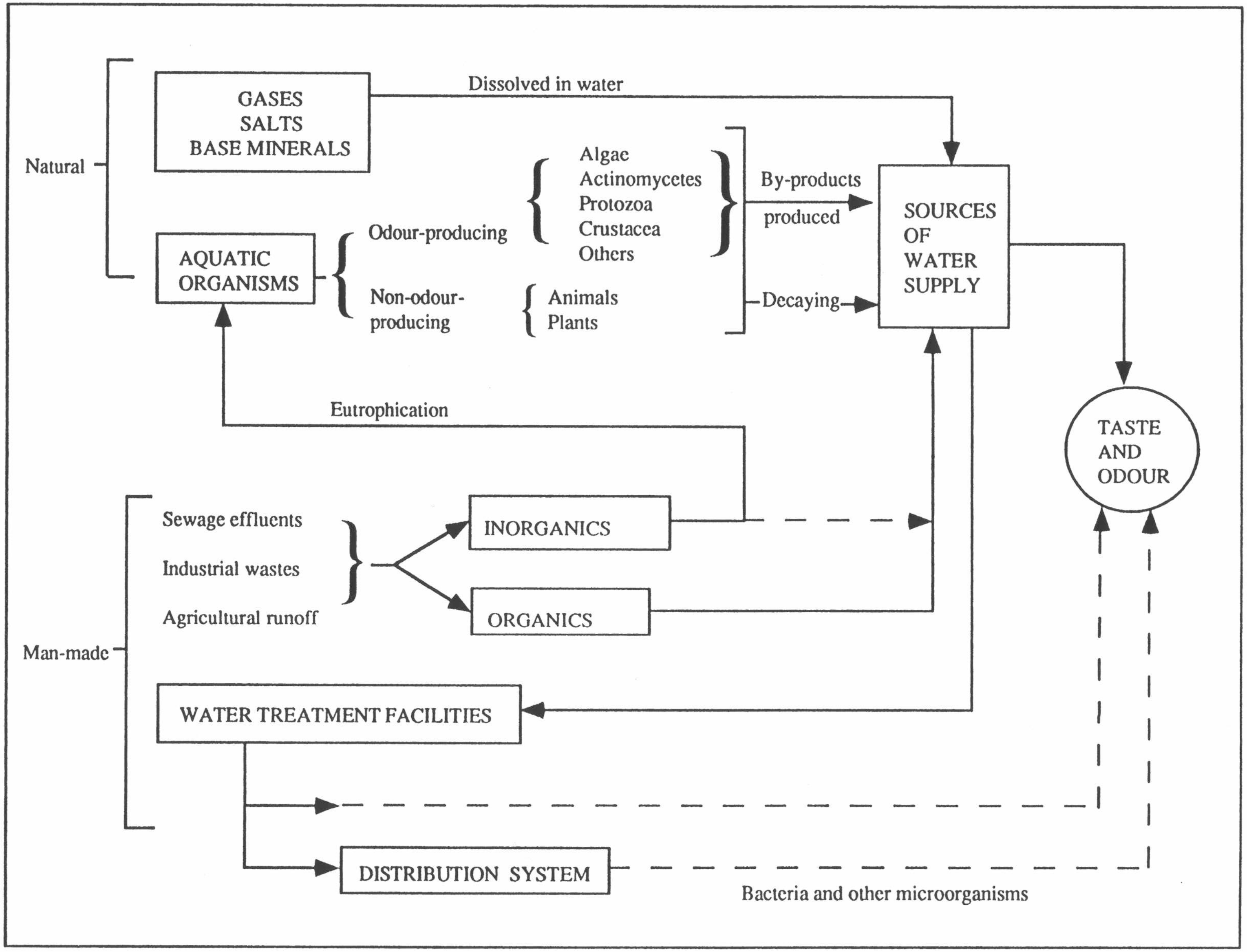
There are often reports of tainted fish in the absence of any industrial discharges and it is important to understand that tainting is not necessarily caused by anthropogenic contaminants. A review of natural chemical compounds present in lakes and rivers that may impair flavour of fish is included in a review by Persson (1984) although much of the literature reported is not recent. Reports of "muddy" or "earthy" tasting fish are often found in the literature (Persson, 1980; 1984, 1985). In one such report, the naturally occurring compounds, geosmin and MIB were found to cause a strong muddy flavour in fish and the presence of these compounds caused the closure of a commercial fishing season in Manitoba (Yurkowski and Tabachek, 1980). Placing of muddy flavored trout in clean water resulted in reduction of flavour to non-tainting levels within 3 to 5 days (Yurkowski and Tabachek, 1974). Geosmin was also responsible for the earthy flavour found in rainbow trout in Denmark (From and Hørlyck, 1984). The geosmin was reportedly acquired most rapidly via the gills, followed in descending order by the skin, small intestine, and stomach. Musty off-flavours in catfish were found to be due to contributions by MIB, 2- methyleneborane and 2-methyl-2-bornene (Martin, *et al.*, 1988). Martin and co-workers found that MIB was more concentrated in peritoneal fat and sub epidermal adipose tissue than in other tissues. Half-lives of 0.14 hours for distribution and 3.62 hours for elimination were found for plasma clearance of MIB. (Martin, *et al.*, 1990). Fatter fish (>2.5% fat) accumulated nearly 3 times more MIB than lean fish (<2%) (Johnsen and Lloyd, 1992).

5. TAINTING OF WATER

Bleached kraft pulp mill effluent and discharges related to other industrial developments are known to contain odorous compounds that have the potential to cause taste and odour problems for downstream users. The significance of off-flavours in water has been reviewed and discussed by a number of researchers (Persson, 1983; Zoeteman, 1980; Mallevialle and Suffet, 1987). There have been complaints about odorous water in the NRBS areas over the years (see Appendix 1 & 2), and there have been a number of discharge-specific odour incidents.

In an early review of odour compounds in water and their sources, Cees and co-workers indicated that there are two basic sources of odour substances (Cees, *et al.*, 1974). All odorous compounds were said to be present in water due to either industrial and municipal sewage effluents or from biological activities of algae and heterotrophic micro-organisms. Lin categorized the two sources of tastes and odours in water as natural or man-made and emphasized that off-flavour events may develop from either one or the other, or a combination of both (Lin, 1976a). The categories Lin set forth are detailed in Figure 6.1. Taste and Odour problems can results from the presence of gases, salts, minerals, aquatic organisms, wastewater treatment plant effluent, industrial discharges, non-point source run-off, water treatment processes and organisms in water distribution systems. Lin also goes on to point out that naturally occurring taste and odour compounds are the most common, but objectionable situations created by man-made sources are often the most troublesome. Mallevialle and Suffet divide the taste and odour compound sources into anthropogenic and biological categories (Mallevialle and Suffet, 1987) and these
will be used in this report.

Figure 5.1 Sources of Taste and Odour in Water (Lin, 1976a)



5.1 PULP MILL EFFLUENTS

Taste and odour problems as a result of pulp mill discharges have to be addressed at downstream water treatment facilities. There were some early studies into the effects that various sulphate (kraft) mill effluents have on treatment parameters at water treatment plant downstream of the discharge (Bouveng, and Lundtstedt, 1966; 1968, Hedberg, *et al.*, 1969) and appropriate treatment modifications have been developed. Cook and co-workers (1973) evaluated the effluent sources from a pulp mill for their water and fish tainting potential in a study carried out for Domtar Fine Papers Ltd. in Cornwall. They concluded that the kraft mill was the largest odour contributor and the sulphite mill was the main source of taste imparted by the mill effluent to the river water. They suggested however, that the deleterious odours and tastes imparted by the pulp mill effluents were diluted, absorbed, reacted or volatilized within a relatively short distance of the mill. In a second study, mill effluents with a wide range of odour thresholds were selected for various treatment evaluations. Diluted BKME samples were found to cause odour impairment at effluent concentrations from 0.1 to 0.4%. However, no correlation was found between the original odour of the effluent and the drinking water impairment (Kovacs and Voss, 1986). They also reported that the concentration of residual chlorine in a finished drinking water sample greatly influenced the degree of taste impairment. Wong *et al.* (1985) reported threshold odour levels ranging from 0.08 to 0.28 (%v/v) for biologically treated BKME and drinking water produced from BKME sources were impaired at effluent concentrations as low as 0.4% for odour and 0.6% for taste. Hamilton, *et al.*, (1987) reported that there have been no documented aquatic taste and odour studies published that address the impacts of CTMP effluents on drinking water treatment processes.

Although there has been considerable research into the health effects of chlorinated organics and other compounds that are found in pulp mill effluents (Wigilius, *et al.*, 1988), the specific compounds responsible for off-flavours in waters as a result of pulp mill discharges are not well understood . A summary of the organic compounds found in spent liquors from pulp bleaching (Kringstad and Lindström, 1984) lists a number of compounds that are possible contributors to odour problems in water. The compounds present in softwood pulping effluents include; chlorinated phenols, chlorinated guaiacols, dichlorovanillin, and chlorinated catechols. These were also reported to be present in hardwood pulping effluents but chlorinated syringols and chlorinated syringaldehydes were also found. In one study, persistent chlorinated acids, phenols, anisoles and veratroles were attributed to be possible tainting compounds downstream of pulp bleaching and chlorodisinfection plants (Paasivirta, *et al.*, 1983). In a study designed to determine the compounds present in BKME that are responsible for taste and odour problems, biologically treated effluent samples from 9 Canadian bleached softwood mills were analyzed for chlorinated phenolics content. Odour thresholds were determined for selected chlorinated phenolics and for a synthetic mixture of these compounds. Results of this study indicated that chlorinated phenolics present in effluent discharged from bleached kraft mills are not expected to contribute an off-odour to recipient waters (Kovacs, *et al.*, 1984). However, microbial metabolism of chlorophenols has been shown to cause the formation of chloroanisoles from chlorinated phenols and these anisoles are much more odorous and more likely to cause tainting (Paasivirta, *et al.*, 1983, Griffiths and Fenwick, 1977). Chlorinated anisoles and veratroles have been implicated in other studies of taste and odour problems related to BKME discharges (Brownlee, *et al.* 1993).

A number of brief annual reports summarizing the analyses carried out on the Athabasca River, Peace River, and Wapiti-Smoky River system waters for the various agencies in the Alberta government have documented the changes in odour levels over the last 25 years. The early reports indicated an increase in odour levels downstream of Hinton followed by steady decrease in levels to Athabasca (Ferguson, 1962; 1963). In 1963-64, high threshold odour numbers were recorded at Obed with a maximum value of 50 detected twice. Odour levels dropped to an average of 8 by Whitecourt (Ferguson, 1964). In 1966-67, threshold odour numbers increased from 1 upstream of Hinton to values ranging from 16 to 64 at Obed. This "wood resin" type of odour prevailed downstream until Tar Island, although values were 8 or lower at locations downstream from

Whitecourt (Province of Alberta, 1967). There were still reports of a large increase in "wood, resin" type odour levels downstream of Hinton and then gradual decreases until Fort McMurray in 1967-68. Below Fort McMurray , a chemical type of odour was found with threshold odour levels as high as 16 (Province of Alberta, 1968). A threshold odour number of 100 was reported at Obed on November 1968. This "wood resin" type of odour prevailed farther downstream for sample sets collected during the winter months (Province of Alberta, 1969). In 1969-70, threshold odour numbers ranging from 16 to 32 were reported at Obed. This "wood resin" type of odour prevailed further downstream for sample sets collected during the winter months. One sample taken at Fort McMurray had a reported odour threshold value of 64 with a hydrocarbon odour (Province of Alberta, 1970). In 1970-71, samples collected above Hinton had minimal threshold odour numbers minimal but were reported as having a "wood-resin" or "musty" type of odour. At the next sample site (Whitecourt), values ranged from 4 to 16 and at Fort McMurray, "chemical" and "hydrocarbon" type odours were often recorded (Simpson, K. J., 1971). In the next year a five year summary report indicated that the Hinton combined effluent imparted considerable odour to the river water (Clayton, 1972). Only once in the five year study period was the threshold odour number below 16 at Obed. The odour effect was still noticeable at Whitecourt but by Smith the threshold odour numbers were at or below the recommended limit of 8. The summary report also indicated that the threshold odour numbers were often above 8 at Tar Island (downstream of Fort McMurray). There were two reports of the water quality criteria maximum odour number violation during the winter of 1972 for a sampling site 117 river miles downstream of Hinton at Whitecourt (Konopasek, 1973). Reports of field evaluations of odour with respect to intensity and nature of odour continued annually and in samples collected downstream of Hinton, a "pulp mill-like" odour was always reported. In 1988 odour in the river water persisted to the vicinity of Smith, while in 1989, the odour persisted to the lower-most reaches of the Athabasca River (at least 500 km) (Noton, 1989). It was also reported that none of the other individual effluents on the River appeared to cause any notable or consistent increase in river odour (Noton and Shaw, 1989). More detailed documentation of measurable concentrations of odorous compounds attributed to BKME as far as 1100 km downstream from the Hinton combined effluent discharge has been published (Brownlee, *et al.*, 1993).

Reports of the odour component of water quality studies for the Wapiti-Smoky River system indicate a noticeable increase in odour attributed to kraft mill effluents during low flows. The odour was reported to persist under ice the length of the Smoky River (Noton, 1989). There were also reports of a large increase in odour of Wapiti River water attributable to pulp mill effluent during the fall and winter of years from 1987 to 1991. In the Smoky River a "pulp" mill odour persisted in the river to the mouth, during the fall and winter of years from 1987 to 1991 (Noton, 1992). In a 1983 study odour incidents were sporadic with only one instance of a measured odour exceeding background levels in the river water during a May sampling (Noton, *et al.*, 1989). However, during this 1983 study an odour characteristic of pulp mills was detectable during sampling in some instances, but by the time the sample reached the laboratory the odour was no longer detectable. In 1988 there were no reported adverse odours attributable to the four mills in the Peace River Basin. However, in previous years, odour problems during winter on the Peace River near the Town of Peace River were attributed to effluent from the Procter and Gamble mill at Grande Prairie (Shaw and Noton, 1989; personal communications in Appendix 2).

Although most of the compound specific work on pulp mill effluent odours has focused on chlorinated compounds, the literature does not provide a reliable basis to attribute odour problems primarily to chlorinated compounds. The work by Domtar Fine Papers (1971) to identify the most odorous process streams did not identify specific compounds but the process streams identified are rich in odorous organosulfur compounds. The production of these compounds is independent of bleaching practices and their control depends on good in-plant spill control and efficient wastewater treatment. Headley (1987) has found sulphones, sulphides and thiophenes in pulp mill effluents. He reported that sulphones and sulphides are present in biologically treated kraft mill effluents and the thiophenes were reported to originate from the cooking process of lignin, in which sodium sulphide and sodium hydroxide are used.

5.2 NON-PULP MILL EFFLUENTS

5.2.1 Biological Sources of Taste and Odour Compounds in Water

The biological compounds that cause taste and odour problems can be "produced' through the decay of dead organisms, or through the activities of living organisms. The odours caused by decaying vegetation have been characterized as musty, earthy, moldy, swampy, fishy, and grassy (Lin, 1976b). Mallevialle and Suffet (1987) cite a number of references which indicate that the odours of decay are due to decomposition of vegetation by bacteria or fungi which produce odorous compounds. The microorganisms which have been reported to produce taste and odour compounds are summarized in Table 5.1.

Table 5.1 Biological Sources of Taste and Odour in Water

Organism	Odour Descriptor	Reference
green algae	grassy	Jüttner, 1983
	musty	Mallevialle & Suffet, 1987
	fishy	Mallevialle & Suffet, 1987
blue-green algae	grassy, vegetable	Slater and Blok, 1983
	earthy, musty	Slater and Blok, 1983
	rotten, sulphur	Jenkins, *et al.*, 1967
diatoms	spicy	Mallevialle & Suffet, 1987
	geranium	Mallevialle & Suffet, 1987
	grassy	Mallevialle & Suffet, 1987
flagellates	cucumber	Lin, 1977
	fishy	Lin, 1977
	violet	Lin, 1977
	musty, grassy	Lin, 1977
fungi	earthy	Wood *et al.*, 1983
bacteria	sulphur	Whitfield & Freeman, 1983
	rotten egg	Lin, 1976b
actinomycetes	earthy, musty	Gerber, 1983
	potato-bin	Gerber, 1983

There have been a large number of articles which discuss the biological production of odorous compounds and they are too numerous for an exhaustive discussion, but a small number have been summarized in the following. Lin discusses the "natural sources of tastes and odours" in water in a series of articles. The natural sources listed include algae, actinomycetes, decaying vegetation, bacteria, and zooplankton. The compounds produced by organisms such as algae, often have very low odour thresholds and thus are a significant problems in many water bodies (Persson and Jüttner, 1983). The natural presence of hydrogen sulfide and some taste imparting inorganic constituents are also summarized (Lin, 1976a; 1976b; 1977).

Gerber has reviewed the odorous compounds produced by actinomycetes, including those substances responsible for earthy, muddy, woody, and potato-bin odours. The specific organisms which produce these "natural" odours and the specific compounds responsible were detailed (Gerber, 1983). Slater and Blok published a detailed review of odorous compounds that are produced by blue-green algae, including geosmin, MIB, β-cyclocitral, hydrocarbons, aromatics, fatty acids, amines, sulfur compounds, terpenoids, and esters. (Slater and Blok, 1983). Brownlee and co-workers (1984) reported the presence of geosmin in the composite raw water and 3- methylindole, dimethyl tetrasulfide and dimethyl pentasulfide from water surrounding decomposing algal blooms. Another report also identifies a number of odorous sulfur compounds produced in decaying blue-green algal cultures and reservoir waters containing blue-green algal blooms, including methylmercaptan, dimethyl sulfide, isobutyl mercaptan, and n-butyl mercaptan. (Jenkins, *et al.*, 1967). Weete reported that butyric acid was the principal metabolite of the actinomycete Streptomyces sp. that represents the greatest potential as an odorous water pollutant under natural conditions. (Weete, 1979). The possible significance of various microbes, including actinomycetes, cyanobacteria and fungi, in the production of earthy tastes and odours in water has been discussed (Wood, *et al.*, 1983) and Wnorowski and Scott reported that both geosmin and dimethyl trisulphide were detected in waters containing large populations of cyanobacteria (Wnorowski and Scott, 1992).

Jüttner (1988a) listed nor-carotenoids, unsaturated hydrocarbons, aldehydes, ketones, alcohols, thio compounds, terpenoids, phenols and aromatic hydrocarbons as compounds which are important biogenic odour compounds frequently found in freshwater. Veijanen and co-workers reported that although there were industrial discharges into Finnish freshwaters, the off-flavours in water were most often found to be geosmin, MIB and various aldehydes (Veijanen, *et al.*, 1988). A study of the taste and odour problem that occurred in the Seine and Marne rivers during the severe drought of 1976 found that geosmin and MIB contributed to odour problems but were not the only explanation. The presence of organic matter seemed to be essential to the development of a moldy taste (Rizet and Mouchet, 1982). Jüttner and co-workers emphasize the dynamic nature of biogenic odour compounds in a report on research carried out in a shallow eutrophic lake. (Jüttner, *et al.*, 1986). Geosmin and MIB were identified by sensory and analytical means as likely major contributors to the raw water odour arising from spring thaw for an ice- covered upland river source (Hrudey, *et al.*, 1992). Although a large number of organisms have been shown to be responsible for odour incidents, Mallevialle and Suffet (1987) point out that each odour problem is seldom due to one organism and thus the taste or odour is often quite complex.

5.2.2 Non-Pulp Mill Anthropogenic Sources of Taste and Odour Compounds

The anthropogenic sources of tastes and odours in water have been summarized in a number of reviews (Mallevialle and Suffet, 1987; Lin, 1976a; 1976b; 1977; Cees, *et al.*, 1974). Common components of man-made odour contributors include petroleum based hydrocarbons, phenols, pesticides, alkyl benzene sulfonates (ABS), water treatment chemicals and disinfection byproducts. (Lin, 1976b). Zoeteman. et al. (1971) reported a list of crude oil and oil product OTC values which indicated that the petroleum products could cause odour problems at µg/L levels. Fok, *et al.* (1984) reported periodic taste and odour episodes in treated drinking water. They suggested that the organic contaminants responsible for these episodes could have been hydrocarbons and phenolics introduced to the raw water source river as a result of urban street runoff. Combinations of odorous compounds can result in interactions between the odours of each component. The resulting odour is often greater than the sum of all components. (Rosen, *et al.*, 1963). Such a phenomenon is known as odour synergism.

There are many taste and odour problems associated with the drinking water treatment processes. Hrudey and co-workers observed more intense odours in treated water than in the raw water and suggested the likelihood that compounds were formed during oxidative reactions resulting from disinfection processes. Four odorous, low molecular weight aldehydes were found (2-methyl propanal, 2-methyl butanal, 3-methyl butanal, and phenylacetaldehyde). (Hrudey, *et al.*, 1988). A summary of a survey conducted to determine practices and problems associated with the use of chlorine dioxide by U.S. water utilities indicates that overall, odour complaints were more numerous and more diverse when chlorine dioxide was used (Dietrich, *et al.*, 1992). The negative effect of chlorination in treated water was noted in an early discussion of the taste and odour problems associated with phenol and chlorinated phenols. Increased chlorination has long been known to intensify taste and odour problems due to the formation of chlorinated phenols from chlorination of the trace levels of phenols which are often present in raw water supplies (Burttschell, *et al.*, 1959). There has also been a study into the effect that many materials that come into contact with drinking water (pipes, reservoir linings, tanks, etc.) have on the taste and odour of the water (Rigal, 1992). It was found that rubber, polyurethanes, epoxydic resins and PVC pipes can cause off-flavours in water that was relatively odourless after treatment. Anselme, *et al.* (1985) reported severe odour problems caused by defective polyethylene water pipes. They found that 20% of 264 pipes tested were defective. Problems were mainly attributed to defective manufacture resulting in release of polymer additives like alkyl phenols and oxidation of pipe surfaces causing the release of polar compounds such as aldehydes and ketones.

Shumway and Palensky (1973) reported that primary, secondary and disinfected secondary effluents from a municipal sewage treatment plant all have the potential to cause tainting in water and fish at levels ranging from 11 to 26 % (v/v). Burlingame et al. (1976) reported the presence of a number of chlorinated anisoles (trichloroanisoles, tetrachloroanisoles and pentachloroanisole) which cause intense odours in secondary and tertiary effluent of a waste water treatment plant. Garrison, Pope and Allen (1976) list many specific compounds that have been found in municipal wastewater treatment plant effluents. Many of these compounds, such as phenols, aromatic hydrocarbons, and chlorinated organics, are capable of tainting water.

6. CONTROL AND TREATMENT OPTIONS

There have been many treatment methods developed for the removal of taste and odour compounds in drinking water treatment plants. However no one method will be successful for all waters at all times (Lin, 1977). There have been a few reviews which deal specifically with the treatment methods for the control or removal of off-flavour compounds from water (McGuire and Gaston, 1988; Monteil, 1983; Hrubec and de Kruiff, 1983). Some treatment processes commonly in use at water treatment plants for taste and odour compound removal include; aeration, coagulation, chlorination, chlorine dioxide addition, oxidation by potassium permanganate addition, ozonation, and carbon adsorption. Lin (1977) includes a lengthy review of these treatment methods. Monteil (1983) also published a detailed review of the use of clarification, oxidation, and adsorption methods used in municipal drinking water treatment for taste and odour abatement. A more recent, detailed summary of the general study of taste and odour problems in water and methods used in treatment for their removal has been documented by Mallevialle and Suffet (1987). In a discussion of treatability for removal of tastes and odours, Naish reported that no single industry (including kraft mill effluent) or municipality was responsible for a major change in taste and odour levels but the combination of all effluents discharged into the St. Francois River resulted in an odour problem (Naish, *et al.*, 1982).

In the general study of water treatment for the control or removal of taste and odour compounds there has been significant research and a considerable number of publications on the subject. Sävenhed and co-workers (1987) found that alum coagulation / sand filtration in a treatment plant did not remove the odour compounds studied, but artificial groundwater recharge in sand and gravel ridges was found to substantially reduce odour compound concentrations. The effectiveness of coagulation, oxidation and activated carbon for removing taste and odour from drinking water were studied in the laboratory and in a full-scale water treatment plant (Baker, *et al.*, 1986). They concluded that coagulation was ineffective, oxidation was effective for some specific compounds, and activated carbon was generally but not always effective. Activated carbon was shown to be effective in taste and odour removal in very early studies (Sigworth, 1957), and has been proven effective in many studies since (Hansen, 1972; Hertzing, *et al.*, 1977; Hrubec and de Kruijf, 1983; Lalezary-Craig, *et al.*, 1988). Many of these studies focused on the use of granular activated carbon although some researchers also found that powdered activated carbon was effective. Dosages of 10 mg/L of powdered activated carbon brought geosmin and MIB levels as high as 66 ng/L down to levels below threshold odour concentrations (Lalezary-Craig, *et al.*, 1988). These researchers also found that chlorine and monochloramine residuals had an adverse effect on removal efficiencies, as did the presence of humic acids. Another study used a pilot scale water treatment plant to study removal efficiencies of geosmin and MIB using granular activated carbon and ozone. The results indicated that the GAC process showed better removal than an ozonation-GAC combination (Vik *et al.*, 1988). The undesirable effects of the contact of chlorine with GAC were also discussed, including formation of taste and odour compounds with prechlorination in a study carried by Voudrias (1986). In general the most effective treatment processes for improvement of taste and odour were reported to be ozonation and granular activated carbon (Hrubec and de Kruijf, 1983). Chlorine dioxide, ozonation and granular activated carbon were evaluated in another study for the removal a taste and odour compound (ethylbenzene) (Fok, *et al.*, 1984). At the pilot scale it was reported that ozone provided better removals that chlorine dioxide and GAC was also effective in odour compound removal. In a study carried out by Glaze *et al.*, nine oxidizing agents were evaluated for removing six taste and odour compounds (1- hexanal, 1-heptanal, dimethyltrisulfide, 2,4-decadienal, MIB, and geosmin) spiked into river water. Ozone alone and ozone with hydrogen peroxide appear to have the most potential for control of taste and odour compounds (Glaze *et al.*, 1990). Ozonation was also found to be effective for removing musty, earthy, fishy, and muddy tastes and odours from water (Anselme, *et al.*, 1988). Astringent and plastic tastes were also removed but the ozonation process was found to be responsible for the development of high-intensity fruity odours which correlated with the presence of aliphatic and aromatic aldehydes and ketones. Chloramination has been shown to have a lesser ability to oxidize odorous compounds than free chlorine (Krasner, *et al.*, 1989) and odour compound removal was optimized by varying free-chlorine dosages and contact times (Krasner, 1988).

In studies of treatment systems used in pulp mills to prevent the discharge of odorous compounds, there has been research into the use of aerated lagoons, activated sludge plants, and air stripping techniques. The behavior of selected chlorinated phenols, guaiacols, catechols, and vanillins during high-rate anaerobic treatment of segregated kraft mill bleach plant effluents was investigated (Parker, *et al.*, 1993b). Mono and di-substituted phenols were generally poorly removed, while trichlorophenol removal was >80%. Chlorinated guaiacols and vanillins removal efficiencies were >95%, while catechols removal efficiencies (except for 3,5 chlorocatechol) were >95% with cosubstrate supplementation . Two extensive mill-scale tests were performed to determine the extent of AOX and chlorinated phenolics removal (Saunamäki, *et al.*, 1991). In the extended aeration activated sludge plant there was 70-80% removal of the chlorinated phenolics. In the high-load activated sludge plant the removals were only 35-55%. Gordon and co-workers (1980) found that biotreatment did not reduce the tainting propensity of BKME as effectively as it reduced BOD and acute toxicity. Apparently some of the constituents in BKME responsible for fish flesh tainting are not readily biodegradable. The effectiveness of two biological treatment systems operating at a kraft softwood integrated pulp and paper mill were assessed in a separate study and the results indicated that the treatability of bleached kraft pulp and paper mill wastewater constituents is dependent upon the characteristics of the treatment systems and the composition of the wastewaters (Stuthridge, *et al.*, 1991). Material balances were determined at two kraft mills in Finland for total organically bound chlorine, chlorophenolics, volatile chlorine compounds and chloroacetones.

The formation of chlorinated phenolics has been studied in detail and it was reported that the chlorinated phenolics formed in hardwood pulp bleaching was only about a third of that for softwood pulp bleaching. Extended pulping of softwood and the use of chlorine dioxide substitution during hardwood bleaching reduced the formation of chlorinated phenolics. Activated sludge treatment removed 75-95% of chlorinated phenolics in kraft effluents (Gergov, *et al.*, 1988) The reduction of the formation of chlorinated phenolics has been studied in detail and Kringstad and Lindström (1984) report that partial replacement of chlorine with chlorine dioxide during the first stage of bleaching and biological treatment are well-known techniques used to reduce the levels of chlorinated phenolics bleached plant effluents. They also report that during bleaching of softwood pulp with partial replacement of chlorine by chlorine dioxide, the levels of chlorinated phenolics will actually increase until 50 % replacement. Above this percentage of chlorine dioxide substitution, the levels of chlorinated phenolics decline. In another study into the benefits chlorine dioxide substitution it was reported that when chlorine was completely replaced with chlorine dioxide, the only chlorinated phenolic detected in the spent liquor was 6-chlorovanillin (Voss *et al.*, 1981).

Woods and co-workers reported the ability of anaerobic consortia to reductively dechlorinate highly chlorinated compounds. Once dechlorinated, lesser chlorinated phenols were readily degraded in aerobic treatment processes (Woods, *et al.*, 1989). A discussion of the possible nucleophilic substitution of sulfide for chloride, on compounds which might not otherwise be biodegradable in an anaerobic kraft mill bleach plant treatment system, has been published (Parker, *et al.*, 1993a).

Because the compounds responsible for taste and odour problems are generally quite volatile, it is quite possible that they can be physically removed through purging or stripping processes. The Henry's law constants determined for IPMP, IBMP, MIB, 2,3,6-TCA and geosmin indicate insignificant volatilization at neutral pH for IPMP, IBMP, MIB and geosmin. Air stripping may be effective for the removal of TCA (Lalezary, *et al.*, 1984).

The work on treatability of pulp mill effluents has emphasized chlorinated compounds. Unfortunately, there is little information on the ability of treatment systems to remove other sources of odour compounds like the organosulfur compounds present in foul condensate (one of the most odorous process streams) (Domtar Fine Papers, 1971). Because the levels of chlorinated compounds produced are being substantially reduced by chlorine dioxide substitution, the role of odorous substances from the kraft process before bleaching is important.

7. CONCLUSIONS

A review of the available technical reports, government documents, books and periodical articles that document the current knowledge about water and fish tainting in the Northern river basins of Alberta has been performed. The purpose of this review was to identify incidence of or the potential for off-flavour tainting of fish and water by components discharged into the Peace, Athabasca, and Slave rivers from various sources. The main focus was the tainting effects of effluents released by pulp mills, but the role that other industrial or municipal waste discharges and non-point source run-off may play in causing off-flavours was also briefly covered. There are a number of relatively unique analytical methods used in the study of taste and odour problems and it is critical to have a basic understanding of these methods before interpreting reports of tainting incidents. In an attempt to assist in the development of this understanding, a detailed review of analytical methods used for the investigation of water and fish taste and odour problems was included. This review has summarized a useful cross-section of the literature that can be used to gain a through understanding of the problems associated with off-flavours in water and fish, especially as they relate to pulp mills in the Northern Alberta river basins. The basic conclusions that were reached are summarized below.

- There are a variety of chemical methods used for the isolation (extraction, adsorption and desorption, removal of interferences), fractionation, and instrumental separation and identification of the compounds responsible for taste and odour problems in water and fish. Those used most often and most effectively include:
    - liquid-liquid extraction with GC analysis
    - closed loop stripping with GC/MS analysis
    - purge and trap with GC analysis
    - solid phase adsorption / desorption with GC analysis

  The problems and limitations associated with each of these methods have been discussed, and it is clear that each of these methods are useful in specific taste and odour or volatile compound analyses. However, it is critical to select the most appropriate method in each case in order to obtain meaningful results.

- The sensory analytical techniques used for the study of taste and odour are even more unique and in general are subject to variabilities caused by differing human olfactory sensitivities. Those sensory techniques which are used most often in water and fish tainting studies include:
    - threshold odour number determination
    - flavour profile analysis
    - chromatographic sniffing

  Sensory analyses are the only method of providing a qualitative description of the tastes or odours present in a sample. However in order to confirm that specific chemicals are responsible for specific odours in a sample, a confirmatory combination of sensory and instrumental techniques is required. Chromatographic sniffing (olfactory GC) is the current method of choice when integrating sensory and common instrumental techniques.

- Fish tainting as a result of pulp mill discharges is well documented and there have been a number of comprehensive reviews on the subject. Many of the reports of fish tainting have shown that the tainting could not be linked to any specific compounds and that the off-flavours were often a result of the combined effects of a number of different industrial discharges into the same water body. The current state of the fish tainting problem in Northern Alberta river basins is relatively undefined. There are 10 pulp / paper mills in the Athabasca and Peace River basins and but other industrial discharges are limited to 5 non-pulp mill industrial effluents, along with numerous small municipal sewage discharges. The combined tainting effects of multiple, diverse industrial effluents which arise in heavily industrialized regions is probably not a concern in these rivers. Instead, concerns are limited to the pulp mill discharges, and non-industrial tainting substances. Biogenic substances must also be considered as potential causes of tainting problems.

- Anecdotal, historical information obtained in discussions and correspondence with various regional fishery biologists in the Fish and Wildlife division of Alberta Environmental Protection, repeatedly indicated that, in general, people do not eat the fish caught in the northern Alberta river basins. Similar avoidance by the First Nation's peoples was confirmed in communications with the NRBS Traditional Knowledge group leader. It is apparent that the concern surrounding tainting of fish by anthropogenic sources such as pulp mill discharges and accidental oil sands wastewater spills, is not only based on documented problems but is also founded on expectations of tainting associated with the existence of industrial discharges.

- Although the specific compounds responsible for taste and odour problems in water downstream of pulp mill effluent discharges are not well defined, the historical water tainting problems in the Northern Alberta river basins have largely been attributed to pulp mill effluents. Samples collected downstream from the pulp mill in Hinton have exceeded odour compliance levels for up to half the length of the Athabasca River under winter ice since the mill was constructed. Occasional taste and odour problems at the town of Peace River have also been attributed to upstream pulp mills. Reports of the odour component of water quality studies for the Wapiti-Smoky River system indicate a noticeable increase in odour due to kraft mill effluents during low flows (under ice).

- Biological or natural sources of tastes and odours in water must be considered as potential causes of off-flavours and the possibility of odour synergism when these natural compounds are combined with anthropogenic tainting compounds is likely.

- Thorough (and costly) drinking water treatment processes can be effective in removing most taste and odour compounds, provided the nature of the odour problem is understood and the treatment processes can be optimized for the types of problems that are identified.

- The removal of many of the relatively volatile, chlorinated organics responsible for tainting problems has been demonstrated in pulp mill wastewater treatment system studies, but removal efficiencies vary and systems must be constantly operated under optimum conditions if all tainting incidents are to be avoided. The preferred control method is prevention of the formation of these odorous chlorinated organics. Chlorine dioxide substitution significantly reduces the formation of such compounds, as does the use of hardwood. Where non chlorine bleaching processes such as CTMP are used there are no documented taste and odour incidents.

- The literature is not very helpful in elaborating the role of specific odour compounds produced from the kraft pulping process, prior to bleaching. Early work identified a number of process streams that contributed the most to overall mill effluent odour. The importance of these odour sources will be dependent upon in-plant spill control and wastewater treatment efficiency but will be independent of recent improvements to the bleaching processes.

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APPENDIX A1.0 PERSONAL COMMUNICATIONS

Sonny Flett

NRBS Traditional Knowledge Group Leader
(403) 697-3733

Personal Communication on November 30, 1993

In general the First Nation's people using water and or fish from the Athabasca River have noticed poorer water quality over the last 10 years. The water quality has become so poor that they no longer drink the water. Mr. Flett expressed concern about fish tainting studies and the lack of panel members from the communities involved. He suggested that a special project be set up for training certain members of the public for flavour profile analysis of fish caught upstream and downstream of effluent in the Northern River Basins. Mr. Flett also suggested that he would go out and discuss the taste and odour issue with the people involved and call back with their comments within the next few weeks.

Personal Communication on January 19, 1994

In discussion with a number of people Mr. Flett heard complaints of "muddy" tasting pickerel caught during low flow conditions on the Athabasca River. In general native people are no longer fishing the Athabasca River because the taste of the fish is often quite unpalatable and they are not able to find a market for the fish. The accidental spill from Suncor in the early 1980's that caused the tainting of fish (petroleum taint) is still remembered by people and they avoid fish from the Athabasca River. Another person commented on the white fish flesh having a poor taste and being "softer" than it used to be. Discussions with people from near Garden River also indicated that in general people no longer eat the fish caught in the Peace/Wapiti river systems because of the long history of unpalatable fish caught there.

Mr. Flett again voiced concern about the lack of community based fish tainting studies.

Hugh Norris

Alberta Environmental Protection
Fish and Wildlife Division
St. Paul
(430) 645-6313

Personal Communication on January 18, 1994

Mr. Norris indicated that there has only been one specific problem of fish tainting that resulted in significant numbers of public complaints. This occurred in the early 80's during an accidental spill at Suncor. Fish caught on the Athabasca River near Ft. McMurray had a very strong hydrocarbon taint. Mr. Norris also explained that he has not received any complaints relating to pulp mill types of odour or tainting in fish.

Ron Millson

Alberta Environmental Protection
Fish and Wildlife Division
Peace River
(403) 624-6405

Personal Communication on December 07, 1993

Mr. Millson indicated that there has been a long standing feeling among area residents that the fish caught from the Wapiti or Smoky Rivers are not edible. Even with the mill upgrades at the Grande Prairie mill, people still do not eat the fish. There has also been concern with tainted fish being caught on the Peace River, but he has no documented complaints. There have been a number of complaints received concerning taste and odour problems with the Peace River drinking water. These problems were thought to be a result of the Procter and Gamble pulp mill discharge to the Wapiti-Smoky River system, as these odour complaints ceased after the raw water intake was moved upstream of the Smoky confluence with the Peace River. However, there is still a negative feeling in general concerning the pulp mill.

Carl Hunt

Alberta Environmental Protection
Fish and Wildlife Division
Peace River
(403) 723-8244

Personal Communication on December 07, 1993

Approximately 18 years ago Mr. Hunt received a series of letters and verbal complaints (attached in Appendix A2) from area residents about taste and odour problems in trout from the Athabasca River between Hinton and the mouth of the Berland River. These complaints were forwarded to Alberta Environment officials. Several additional complaints were not recorded. The general consensus among the anglers was that the foul taste was caused by effluent from the Hinton pulp mill (North West Pulp and Power at the time). There was also a strong "sulfur" odour from the river water.

More recently, not many people fish the Athabasca River any more, so that may be the reason for fewer complaints. However, there has been a strong lobby by one long time fisherman and Edson area resident for formal fish tainting studies.

Dennis Musselman

Alberta Environmental Protection
Fish and Wildlife Division
Grande Prairie
(403) 538-5265

Personal Communication on January 18, 1994

Over the years there have been a number of complaints from one area resident about inedible fish caught on the Wapiti River. In general people do not eat the fish caught on the Wapiti, Smoky or Peace Rivers. There have also been some reports of illness due to the consumption of tainted fish. In recent years consultants hired by Proctor and Gamble caught and tasted the fish (pickerel) and found them to be palatable. There are still many anglers who fish for sport (catch and release of trophy sized fish) on the Wapiti River.

APPENDIX A2.0 COMPLAINT DOCUMENTATION

Alberta
~~FORESTRY, LANDS AND WILDLIFE~~ ENVIRONMENTAL PROTECTION
Fish and Wildlife Division

December 29, 1993

Ms. Sandra Kenefick
Room 332
Newton Research Building
University of Alberta
Edmonton, Alberta T6G 2C2

Dear Ms. Kenefick,

RE: NORTHERN RIVER BASINS STUDY:
FISH TASTE PROJECT

From your recent request for information regarding the number of public complaints received on fish taste problems in the rivers and tributaries of the Northern River Basins Study area, I would provide you with the following reply.

The major comment I received from all parts of the study area was that the people do not eat the fish anymore. The Alberta Fishing Guide and many news releases over the past several years have issued warnings to the general public about the safety of consuming various fish species in various waters of Alberta. The NRBS area has had several waters in which warnings have been issued. This was the predominant response.

Although many people no longer eat the fish, there are still some people who do. In the Fort Chipewyan District office, we receive one or two complaints per year. A similar number of complaints are received in the Fort McMurray office each year. In 1982 and 1983, following the Suncor spill of various materials into the Athabasca River, major complaints of poor fish taste did occur. The Alberta Fish and Wildlife Division carried out a number of fish taste tests during that period of time. After the obvious "petrochemical" taste disappeared, no further taste tests were undertaken. The commercial fishery in lake Athabasca was closed following the Suncor spill until the "off taste" disappeared.

A number of fish taste complaints came from the Wapiti River a few years ago. Dr. Hrudey was directly involved with this case. During the last few years, it seems that the public no longer consume fish from this river.

Also, for several years prior to 1985 in the Upper Athabasca River (Hinton area) frequent public complaints were received regarding the poor taste of fish (particularly rainbow trout). Many people attributed the taste to the Hinton pulp and paper mill. Again, in more recent years, it appears that the people are no longer consuming the fish.

R Makowecki

Ray Makowecki

cc - P. Long
D. Giggs
F. Cardinal
C. Hunt
M. Barrett - B. Stubbs
K. Crutchfield

Alberta
RECREATION, PARKS
AND WILDLIFE

MEMORANDUM

FISH AND WILDLIFE DIVISION

FROM Roger Everett
Aquatic Habitat Protection Biologist

OUR FILE REFERENCE

YOUR FILE REFERENCE

TO Carl Hunt
Regional Fishery Biologist
Edson

DATE December 29, 1975

TELEPHONE 427-6766

SUBJECT FLAVOUR OF RAINBOW TROUT FROM THE ATHABASCA RIVER

At the time these fish were taken from the river, North West Pulp and Power were dumping their sewage into the river after only primary treatment, as they were then renovating their secondary treatment facilities. NWP & P had received permission from Environment to do this.

I have forwarded a copy of the correspondence to Akio Masuda, Pollution Control Division, who has indicated that he will respond to the complainants.

As for tests that should be conducted upon the fish, Mr. Masuda indicated that the only one he could think of was a taste test. To do this, one first cooks the fish and then has a panel of tasters sample the fish to identify the nature and extent of the pulp mill flavour.

Roger Everett
Aquatic Habitat Protection Biologist

RE/md

Alberta MEMORANDUM
LANDS AND FORESTS

FROM Carl W. Hunt
Regional Fishery Biologist
Box 1390, Edson, Alberta

OUR FILE REFERENCE

YOUR FILE REFERENCE

DATE December 15, 1975

TO Dave G. Buchwald
Habitat Protection Biologist
Fish & Wildlife Division
Edmonton, Alberta

TELEPHONE

SUBJECT Re: Athabasca River

During the past summer and fall I have received a number of complaints from area residents about a taste and odour problem in rainbow trout from the Athabasca River between the mouth of the Berland River and Hinton. The importance of a rainbow trout fishery in the Athabasca was unknown prior to these angler reports, however the population appears to be significant.

Two letters and two complaints by telephone are attached, however several of the initial complaints were not recorded. In addition Mr. R. Gideon delivered six dressed headless rainbow trout to this office and requested an analysis to determine the cause of the disagreeable taste. I requested the habitat section to provide the name of a laboratory that could handle the analysis and also requested information about the chemical composition of North West Pulp & Power effluents. A reply has not been received.

If, as the anglers suggest, the foul taste is caused by effluent from NWP&P effort should be made to ensure the current pollution abatement project at the mill will remove or treat the compound causing a taste problem.

Since Dept. of Environment is responsible for waste water effluent and water quality standards they should be aware of and perhaps respond to the complaints.

Carl W. Hunt
Regional Fishery Biologist

CWH/jm

Box 834
Edson, Alberta,
Toe Opo.
October 28, 1975.

Mr. Carl Hunt
Fish & Wildlife Officer
Edson, Alberta.

Mr. Hunt:-

While fishing the Athabasca River in the area where the 15th Base line crosses the river, we caught some very nice rainbow trout, (2-3 pounds estimated). We could hardly stand the smell cooking them, and they were virtually impossible to eat. The taste and smell are the same as the accumulated junk (what ever it is) that North West Pulp andPower are dumping into this river.

Whether or not this will kill the fish, we don't know, but it certainly makes them unpalatable. In fact, the river water itself stinks of this "junk". A sad situation!

Hopefully something can be done about this.

Sincerely yours

D. F. Petty
Mrs. Ouita Petty

Mr. Daniel F. Petty
&
Mrs. Ouita Petty.

Marlboro, Alta.
Oct. 22, 1975.

Office of the Fish and Wildlife Division,
Edson, Alberta.

Gentlemen;

During the past two months I have had occasion to make three trips down the Athabasca River by boat from a point almost directly north of Obed about 40 or 50 miles ~~dx~~ downstream and return. During the course of these trips I caught several rainbow trout up to 2¼ lbs. in weight. I found upon cooking these fish, that they had a very objectionable flavor, similar to the odor eminating from the North-West pulp mill at Hinton and I could only conclude that this flavor was caused by the waste water from this mill being dumped in ~~x~~ the river up-stream. I tried everything I could think of that might eliminate this objectionable taste, such as, scaling, skinning, filleting,and soaking in a weak solution of vinegar and water and, although I was successful in eradicating a portion of it, I was also successful in elimanating every vestige of the natural rainbow trout flavor, and the net result was an abslutely uneatable fish.

I might add that I hae trapped along the Athabasca inthis area for the past 50 years and during that time I have, from time to time caught and eaten many trout of the rainbow and dolly varden varieties , and, prior to the operation of the North-West mill at Hinton, these fish were always of excellent flavor.

Yours truly,

D. W. Miller
Box1088, Edson, Alta.

D. Miller

COMPLAINTS

October 27, 1975

Jim Adrain - 10540 E Apache Tr.
Sp. 33
Apache Junction, Arizona
85220
U.S.A.

Caught fish in Athabasca 30 miles downstream (3 rainbow trout) nice big ones. Close to 6 lbs. and 4½ lbs.

Smelt like atmospheric fumes at Hinton when cleaning next day.

Couldn't eat them.

Dwayne Stephens - Box 1209
Edson, Alberta
TOE OPO

Reports smell of rainbow trout from Athabasca River was terrible "Pulp mill smell".

4 lbs. maybe 5 lbs., small 2 lbs.

APPENDIX A3.0 NON-PULP MILL EFFLUENTS DISCHARGED TO THE NORTHERN RIVER BASINS OF ALBERTA

Table A3.1 Continued

NAME OF EFFLUENT	DRAINAGE BASIN	EFFLUENT DESCRIPTION
Wandering River	Athabasca	municipal sewage
West Vale	Athabasca	community well
Westlock	Athabasca	municipal sewage
Westwind Mobile Home Park	Athabasca	municipal sewage
Whitecourt	Athabasca	municipal sewage
Wildwood	Athabasca	municipal sewage
Canyon Creek/Widewater	L. Slave	private sewage system
Driftpile School	L. Slave	municipal sewage, school
Enilda	L. Slave	municipal sewage
Faust	L. Slave	municipal sewage
Grouard	L. Slave	municipal sewage
High Prairie	L. Slave	municipal sewage
Hilliard's Bay Prov. Park	L. Slave	municipal sewage
Joussard	L. Slave	municipal sewage
Kinuso	L. Slave	municipal sewage
Lesser Slave Lake Prov. Park	L. Slave	municipal sewage
Nine Mile Point	L. Slave	private sewage system
Slave Lake	L. Slave	municipal sewage
Swan Hill Booster Station	L. Slave	municipal sewage
Wagner	L. Slave	municipal sewage
Alberta Forest Service	Peace	municipal sewage
Atikameg School	Peace	municipal sewage, school
Bear Canyon	Peace	municipal sewage
Beaverlodge	Peace	municipal sewage
Berwyn	Peace	municipal sewage
Bezanson	Peace	municipal sewage
Big Prairie	Peace	
Bishop Routhier School	Peace	municipal sewage, school
Bluesky	Peace	municipal sewage
Brownvale	Peace	municipal sewage
Buffalo Head Prairie School	Peace	municipal sewage, school
Cadotte Lake School	Peace	municipal sewage, school
Carson-Pegasus Prov. Park	Peace	municipal sewage
Chevron	Peace	municipal sewage
Clairmont	Peace	municipal sewage
Clairmont Trailer Park	Peace	municipal sewage
Cleardale	Peace	municipal sewage
Crooked Creek	Peace	
Deadwood	Peace	water plant
Deadwood School	Peace	municipal sewage, school
Debolt	Peace	municipal sewage
Desmarais	Peace	municipal sewage
Dixonville	Peace	municipal sewage
Donelly	Peace	municipal sewage
Dr. Mary Jackson(Keg R) School	Peace	municipal sewage, school
Eaglesham	Peace	municipal sewage
East Manning	Peace	water plant
East Rainbow Camp	Peace	municipal sewage
Eldoe's Mobile Home Park	Peace	municipal sewage
Elmsworth School	Peace	municipal sewage, school
Evergreen Park (Gr. Prairie)	Peace	municipal sewage
Fairview	Peace	municipal sewage
Falher	Peace	municipal sewage

Continued ....

Table A3.1 Continued

NAME OF EFFLUENT	DRAINAGE BASIN	EFFLUENT DESCRIPTION
Footner Lake Forestry Site	Peace	municipal sewage
Fort Vermillion	Peace	municipal sewage
Fox Creek	Peace	municipal sewage
Fox Creek Trailer Park	Peace	municipal sewage
Gift Lake	Peace	municipal sewage
Girouxville	Peace	municipal sewage
Goodwin	Peace	
Grande Cache	Peace	municipal sewage
Grande Cache Forest Industries	Peace	industrial, sawmill
Grande Prairie	Peace	municipal sewage
Grande Prairie Airport	Peace	municipal sewage
Grandview Hutterite Colony	Peace	municipal sewage
Griffen Creek	Peace	community well
Grimshaw	Peace	municipal sewage
Grovedale	Peace	municipal sewage
Guy	Peace	municipal sewage
Harmon Valley	Peace	watering point
Hawk Hills	Peace	water plant
High Level	Peace	municipal sewage
Hilltop Estates	Peace	
Hines Creek	Peace	municipal sewage
Hotchkiss	Peace	water plant
Hythe	Peace	municipal sewage
Jean Cote	Peace	municipal sewage
Keg River	Peace	watering point
La Crete	Peace	municipal sewage
La Glace	Peace	municipal sewage
Little Buffalo	Peace	municipal sewage, school
Little Buffalo Prairie School	Peace	municipal sewage, school
Little Smoky	Peace	municipal sewage
Loon Lake School	Peace	municipal sewage, school
Manning	Peace	municipal sewage
Marie-Reine	Peace	municipal sewage
McLennan	Peace	municipal sewage
Meekwap Work Camp	Peace	municipal sewage
Mitsue	Peace	
Moonshine Lake Prov. Park	Peace	municipal sewage
Nampa	Peace	municipal sewage
New Fish Creek	Peace	water treatment
North Taloree Indian Reserve	Peace	municipal sewage
Northstar	Peace	municipal sewage
Nose Creek School	Peace	municipal sewage, school
Notikewin Provincial Park	Peace	municipal sewage
P&G District II Forestry Camp	Peace	municipal sewage
Paddle Prairie	Peace	municipal sewage
Peace River	Peace	municipal sewage
Peace River Airport	Peace	municipal sewage
Peace River Corrections	Peace	municipal sewage
Peerless Lake School	Peace	municipal sewage, school
Peoria	Peace	municipal sewage
Pine Ridge School	Peace	municipal sewage
Queen Elizabeth Prov. Park	Peace	municipal sewage
Redearth Creek	Peace	municipal sewage

Continued ....

Table A3.1 Concluded

NAME OF EFFLUENT	DRAINAGE BASIN	EFFLUENT DESCRIPTION
Redearth Creek-AB Forest Serv	Peace	municipal sewage
Reinwood	Peace	watering point
Reno	Peace	
Ridge Valley	Peace	municipal sewage
Rocky Lane	Peace	municipal sewage
Royce	Peace	watering point
Rycroft	Peace	municipal sewage
Sandy Lake	Peace	municipal sewage
Saskatoon Island Prov. Park	Peace	municipal sewage
Sexsmith	Peace	municipal sewage
Shell-Peace River Complex	Peace	sewage
Spirit River	Peace	municipal sewage
St. Isadore	Peace	municipal sewage
Steen River School	Peace	municipal sewage, school
Strong Creek	Peace	well & water plant
Sturgeon Heights Community	Peace	municipal sewage, school
Sturgeon Lake Campground	Peace	municipal sewage
Sturgeon Lake Indian Reserve	Peace	municipal sewage
Sunset House	Peace	municipal sewage
Swan City Mobile Home Park	Peace	municipal sewage
Sweat House	Peace	water treatment & comm. well
T&E Mobile Home Park	Peace	municipal sewage
Tangent	Peace	municipal sewage
Teepee Creek	Peace	municipal sewage
Three Creeks	Peace	
Tompkins Landing	Peace	municipal sewage
Triple L Mobile Home Park	Peace	municipal sewage
Trout Lake School	Peace	municipal sewage, school
Utikumak	Peace	municipal sewage
Valleyview	Peace	municipal sewage
Valleyview Hutterite Colony	Peace	municipal sewage
Wabasca	Peace	municipal sewage
Wanham	Peace	municipal sewage
Watino	Peace	municipal sewage
Weberville	Peace	water co-op
Wembley	Peace	municipal sewage
Whitelaw	Peace	municipal sewage
Whitemud	Peace	water distribution
Winagami	Peace	
Woking	Peace	municipal sewage
Woodland Cree Band No. 474	Peace	municipal sewage
Worsley	Peace	municipal sewage
Young's Point Provincial Park	Peace	municipal sewage

APPENDIX A4.0 TERMS OF REFERENCE

NORTHERN RIVER BASINS STUDY

ASSIGNMENT NO. 4 - TERMS OF REFERENCE

Project 4412-C1: Synthesis Report on Water and Fish Tainting in the Northern River Basins

I. Introduction

Bleached kraft pulp mill effluent and discharges related to other industrial developments are known to contain compounds that have the potential to cause off-flavours in water and fish in the receiving waters. The purpose of this project is to review the literature, government records, etc. to identify incidence of and the potential for off-flavour tainting of water and fish by municipalities, agricultural and industrial developments. A review of possible analytical methods for investigating water and fish taste and odour problems is also to be undertaken.

II. Requirements

1. Review the literature, government records, etc. to identify incidences of or the potential for off-flavour tainting of water and/or fish in the Peace, Athabasca and Slave rivers by compounds discharges into rivers by municipalities, agriculture and industrial developments (i.e., bleached kraft and chemithermomechanical pulp mills, oil and gas developments, etc.). Important references are to be included in an annotated bibliography.

2. Review the literature to document the current knowledge about the role industrial developments found in the northern river basins may play in causing off-flavours in fish and water. The major emphasis should be on effluents released by pulp mills. However, major review papers dealing with municipals wastes, agricultural discharges and non-pulp mill industries found in the northern river basins should also be identified. This is to include an annotated bibliography of major summary reports found in the literature. The literature search and annotated bibliography will also include a review of analytical methods used to investigate taste and odour problems. Important reverences dealing with analytical methods will also be included in the annotated bibliography. The review is also to include consultation with Mr. Lloyd (Sonny) Flett ([403] 697-3733; fax [403] 697-3517), Traditional Knowledge Group Leader, NRBS, regarding native concerns with water and fish tainting in the northern river basins.

3. Based on the information compiled in 1 and 2, above, prepare a synthesis report outlining incidence of or the potential for water and/or fish tainting caused by municipalities, agriculture of industrial development in the northern river basins. The synthesis report should also include a summary of analytical methods appropriate for studying water and fish tainting in the northern river basins.

APPENDIX A5.0 ANNOTATED BIBLIOGRAPHY

INTRODUCTION

Bleached kraft pulp mill effluent and discharges related to other industrial developments are known to contain odorous compounds that have the potential to cause off-flavours in water and fish in the receiving waters. The following is an annotated list of technical reports, government documents, books and periodical articles that document the current knowledge about water and fish tainting in the northern river basins of Alberta. The purpose of this work is to identify incidence of or the potential for off-flavour tainting of fish and water by compounds discharged into the Peace, Athabasca, and Slave rivers from various sources. The major emphasis is on the tainting effects of effluents released by pulp mills, but the role other industrial developments, municipal wastes and agricultural discharges may play in causing off-flavours is also briefly covered. A detailed annotated list of possible analytical and sensory methods for the investigation of water and fish taste and odour problems is also included.

The references are grouped into 7 categories. The categories and the references in each category are listed alphabetically and some references are listed in more than one section where applicable.

The references have been grouped into the following categories:

1. Chemical Methods of Odorous Compound Analysis
2. Control or Treatment Options
3. Fish Tainting Relating to Non-Pulp Mill Sources
4. Fish Tainting Relating to Pulp Mill Effluents
5. Sensory Methods of Odorous Compound Analysis
6. Water Tainting Relating to Non-Pulp Mill Sources
7. Water Tainting Related to Pulp Mill Effluents

This list is by no means exhaustive but represents a very useful cross-section of literature that can be used to gain a thorough understanding of the problems associated with off-flavours in water and fish, especially as they relate to pulp mills in the Northern River Basins.

1.0 Chemical Methods of Odorous Compound Analysis

APHA-AWWA-WEF (1992). Standard Methods for the Examination of Water and Wastewater, 18th Edition, American Public Health Association.

Technical and detailed summaries of standard methods used for closed-loop stripping, gas-chromatographic/mass spectrometric analysis and liquid-liquid extraction gas-chromatographic/mass spectrometric analysis.

Arthur, C. L., Killam, L. M., Buchholz, K. D., Pawliszyn, J. and Berg, J. R. (1992). Automation and optimization of solid-phase microextraction. Analytical Chemistry **64**: 1960-1966.

The solid-phase microextraction technique has been adapted for automated use with a Varian model 8100 autosampler on a gas chromatograph.

Arthur, C. L., Potter, K. D., Buchholz, S., Motlagh, J. and Pawliszyn, J. (1992). Solid-Phase microextraction for the direct analysis of water: Theory and practice. LC/GC **10**(9): 656-661.

Detailed outline of the theory and use of the solid-phase microextraction system developed by Pawliszyn. Reports that the method is a fast, sensitive, inexpensive, portable, and solvent-free method for extracting organic compounds from aqueous samples.

Arthur, C. L., Pratt, K., Motlagh, S. and Pawliszyn, J. (1992). Environmental analysis of organic compounds in water using solid phase micro extraction. Journal of High Resolution Chromatography **15**: 741-744.

The solid-phase microextraction technique is applied to the analysis of substituted benzenes, polychlorinated biphenyl congeners 1-naphthol, and caffeine when present in an aqueous matrix.

Baker, R. J. and Suffet, I. H. (1987). Continuous liquid-liquid extractor for the isolation and concentration of nonpolar organic compounds for biological testing in the presence of humic materials. In Organic Pollutants in Water. Advances in Chemistry Series , American Chemical Society. 571-591.

Technical description of continuous liquid-liquid extractor.

Bellar, T. A. and Lichtenberg, J. J. (1974). Determining volatile organics at microgram per litre levels by gas chromatography. Journal AWWA (Dec.): 739-744.

Outline of a purge-trap system combined with direct gas chromatographic analysis. Describes modification and assembly of commercially available components and early developmental uses for volatile compound analyses.

Blok, V. C., Slater, G. P. and Giblin, E. M. (1983). Comparison of sorption and extraction methods for recovery of trace organics from water. Water Science and Technology **15**: 149-159.

A comparative study of extractions recoveries for six adsorbents relative to Likens-Nickerson solvent extraction and continuous liquid-liquid extraction. Solvent extraction methods were shown to give higher recoveries than the adsorbents as well as yielding lower levels of impurities than the adsorbents.

Blomberg, S. and Roeraade, J. (1988). An evaluation and comparison of micro-techniques for concentration of volatile components from dilute solutions. Chromatographia **25**(1): 21-24.

A comparative study of various concentrations techniques and their effects on recovery of volatile compounds after solvent extraction.

Borén, H., Grimvall, A., Palmborg, J. and Sävenhed, R. (1982). Modified stripping technique for the analysis of trace organics in water. Journal of Chromatography **252**: 139-146.

Discussion of improving closed-loop stripping analysis and contamination problems by using an open stripping system.

Borén, H., Grimvall, A., Palmborg, J., Sävenhed, R. and Wigilius, B. (1985). Optimization of the open stripping system for the analysis of trace organics in water. Journal of Chromatography **348**: 67-78.

Optimum purging temperatures and desorption solvents are studied.

Brownlee, B. G., Gammie, L., Gummer, W. D. and MacInnis, G. A. (1988). A simple extraction procedure for moderately volatile taste and odour compounds such as geosmin and 2-methylisoborneol – method and applications. Water Science and Technology **20**(8/9): 91-97.

A discussion of extraction efficiencies for geosmin and 2-methylisoborneol using a simple liquid-liquid extraction method. Includes implementation of method on site at a water treatment facility with relatively inexperienced personnel and without elaborate equipment.

Chriswell, C. D., Ericson, R. L., Junk, G. A., Lee, K. W., Fritz, J. S. and Svec, H. J. (1977). Comparison of macroreticular resins and activated carbon as sorbents. Journal AWWA **Dec**: 669-673.

The results of a comparison of preconcentration using one resin and one activated carbon in the isolation of organic compounds from water are reported. Filtrasorb 300 activated carbon was compared to Amberlite XAD-2 resin in determining recoveries for 100 model compounds. Results indicate that the resin is superior to the activated carbon for isolating identifiable taste and odour compounds from water.

Cline, S. M. and Severin, B. F. (1989). Volatile organic losses from a composite water sampler. Water Research **23**(4): 407-412.

Report that commercially available composite water samplers are inadequate for the collection of aqueous streams when the volatile component of these waters is of concern. Compounds with Henry's constants between 0.045 - 0.95 (dimensionless) were studied.

Coleman, W. E., Munch, J. W., Slater, R. W., Melton, R. G. and Kopfler, F. C. (1983). Optimization of purging efficiency and quantification of organic contaminants from water using a 1-L closed-loop stripping apparatus and computerized capillary column GC/MS. Environmental Science and Technology **17**(10): 571-576.

Discussion of optimization of closed-loop stripping efficiencies by varying purging times and temperatures. Optimum recoveries were achieved after 2 hour purging at 40°C.

Fox, M. E. (1986). A Practical Sampling and Extraction System for the Quantitative Analysis of Sub ng/L of Organochlorine Contaminants in Filtered Water and Suspended Solids. NWRI Contribution No. 86-41.

A detailed technical description of a large volume sampling and liquid-liquid extraction system.

Goulden, P. D. and Anthony, D. H. J. (1985). Design of a Large Sample Extractor for the Determination of Organics in Water. NWRI Contribution No. 85-121.

A detailed, technical description of a continuous liquid-liquid extractor commonly known as the "Goulden extractor".

Grob, K. (1973). Organic substances in potable water and its precursors. Part I. Methods for their determination by gas-liquid chromatography. Journal of Chromatography **84**: 255-273.

First discussion of the closed loop stripping analysis method developed by Grob. Reported as a method for transferring substances from water at room temperature onto an absorbent filter by a closed-loop air stream.

Grob, K. and Grob, G. (1974). Organic substances in potable water and its precursor. Part II. Applications in the area of Zürich. Journal of Chromatography **90**: 303-313.

Applications of the CLSA system with reported concentrations of contaminants in the ng/L range.

Grob, K., Grob, G. and Habich, A. (1984). Overcoming background contamination in closed-loop stripping analysis (CLSA). Journal of High Resolution Chromatography and Chromatographic Communications **7**: 34-342.

Outlines typical sources of contamination of the CLSA system and the carbon filters used. Also offers suggestions for cleaning of the system and the filters.

Grob, K. and Grob, K., Jr. (1978). On-column injection on to glass capillary columns. Journal of Chromatography **151**: 311-320.

Valuable description of proper cool-on-column injection techniques that are very useful with volatile compounds.

Grob, K., Grob, K. J. and Grob, G. (1975). Organic substances in potable water and in its precursor. III. The closed-loop stripping procedure compared with rapid liquid extraction. Journal of Chromatography **106**: 299-315.

A comparative study of closed-loop stripping and liquid extraction techniques. Factors compared include; overall sensitivity, substance specific sensitivity, quantitative reproducibility, routine use suitability, time requirements, turbid water samples, GC column life, and equipment availability.

Grob, K., Jr. and Neukom, H. P. (1984). Glass wool in the injector insert for quantitative analysis in splitless injection. Chromatographia **18**(9): 517-519.

Summary of the benefits of a silylated glass wool plug in the injector insert. Includes study of improved quantitation of samples of volatile compounds due to improved transfer of the volatiles and retention of high boiling materials.

Grob, K. and Zürcher, F. (1976). Stripping of trace organic substances from water. Equipment and procedure. Journal of Chromatography **117**: 285-294.

Detailed description of the equipment and practical procedures for routine application of the closed-loop stripping apparatus.

Habich, A. and Grob, K. (1984). Filter extraction in closed loop stripping analysis (CLSA). Journal of High Resolution Chromatography & Chromatography Communications **7**: 492-494.

A short communications of results relating techniques used to extract compounds adsorbed to the micro-sized, activated carbon filter used for closed-loop stripping.

Headley, J. V. (1987). GC/MS identification of organosulphur compounds in environmental samples. Biomedical and Environmental Mass Spectrometry **14**: 275-280.

Detected 21 odorous organosulphur compounds in a survey of water, industrial effluent, sediment, and fish samples. Relative retention times and relative response factors are listed for these compounds. The persistent chlorinated organosulphur compound and dichlorodimethyl sulphone was detected in fish and in biologically treated pulp and paper mill effluents.

Hertzing, D. R., Snoeyink, V. L. and Wood, N. F. (1977). Activated carbon adsorption of the odorous compounds 2-methylisoborneol and geosmin. Journal AWWA **69**: 223-228.

Reports optimum conditions for concentration of MIB and geosmin through adsorption onto activated carbon. Most adsorbed compounds could be recovered by extraction with dioxane. Solution pH had no major effect on adsorption, but the presence of humic substances significantly reduced adsorption.

Hrudey, S. E. and Low, N. (1992). Discussion of "The Effect of Disinfectants on a Geosmin-Producing Strain of *Streptomyces griseus*" by Whitmore, T.N. and Denny, S. 1992. J. Appl. Bacteriol., 72, 160-165. Journal of Applied Bacteriology **73**: 445-446.

Presents a summary of the reported thresholds for olfactory detection of geosmin. Reported values range from 4 ng/L to 200 ng/L. Indicates that current workers in the filed expect an odour detection threshold for most individuals of 10 to 20 ng/L for geosmin.

Hwang, C. J., Krasner, S. W., McGuire, M. J., Moyland, M. S. and Dale, M. S. (1984). Determination of subnanogram per liter levels of earthy-musty odorants in water by the salted closed-loop stripping method. Environmental Science and Technology **18**(7): 535-539.

Complete discussion of optimizing recoveries using the "salted" closed-loop stripping method of 100 g/L sodium sulfate additions prior to stripping. Also includes results of filter resistance study which concluded that filters used for replicate analyses must have flows that are matched within 0.1mL/min.

Ibrahim, E. A., Lippincott, R. L., Brenner, L. and Suffet, I. H. (1987). The effect of cyclohexene, a preservative in dichloromethane on the liquid-liquid extraction and analysis of chlorinated drinking water. Journal of Chromatography **393**: 237-253.

Good summary of compounds that may be present as contaminants when using dichloromethane as solvent for acid or base/neutral liquid-liquid extractions of water which has been chlorinated. These compounds are reported to be free radical chlorination and oxidation products of cyclohexene (which is added as a preservative to the dichloromethane).

Ibrahim, E. A., Suffet, I. H. and Sakla, A. B. (1987). Evaporative concentration system for trace organic analysis. Analytical Chemistry **57**: 2091-2098.

A comparison study of extraction solvent removal through the standard Kuderna-Danish technique and an automated evaporative concentration system.

Infante, A. P., Guajardo, N. C., Alonso, J. S., Navascués, M. C. M., Melero, M. P., Cortabitarte, M. S. M. and Narvión, J. L. O. (1993). GC/MS analysis of organic water pollutants isolated by XAD-2 resins and activated carbon in the Gallego River, Spain. Water Research **27**: 1167-1176.

Results of a large scale (1300L) isolation of organics using resins and activated carbon, followed by desorption with ethyl ether and dichloromethane respectively. More than 220 extracted compounds were identified and are listed.

Johnsen, P. B. and Kuan, J.-C. W. (1987). Simplified method to quantify geosmin and 2-methylisoborneol concentrations in water and microbiological cultures. Journal of Chromatography **409**: 337-342.

Details a basic liquid-liquid extraction procedure adapted for use with microbiological cultures. Would be well suited for use with deuterated standards.

Junk, G. A., Richard, J. J., Grieser, M. D., Witiak, D., Witiak, J. L., Arguello, M. D., Vick, R., Svec, H. J., Fritz, J. S. and Calder, G. V. (1974). Use of macroreticular resins in the analysis of water for trace organic contaminants. Journal of Chromatography **99**: 745-762.

Detailed discussion of the use of porous polymer resins for the accurate analyses of organic solutes in water covering a concentration range of 50 ppm to 20 ppt. Reports an extensive study of proper resin clean-up, standard preparation, extraction pH, extraction solvent, drying of solvent after extraction, and concentration of eluate.

Jüttner, F. (1988). Quantitative trace analysis of volatile organic compounds. Methods in Enzymology **167**: 609-616.

Technical description of procedure and apparatus used for the quantitative analysis of geosmin, MIB, and other "volatile organic compounds" produced by cyanobacterial cultures.

Keith, L. H., Ed. (1979). Identification and Analysis of Organic Pollutants in Water. Ann Arbor, Michigan., Ann Arbor Science Publishers Inc.

Good introduction to the technical aspects of trace organic compound analysis and identification techniques. Includes a number of chapters directly related to pulp mill effluents.

Kenefick, S. L., Brownlee, B., Hrudey, E. J., Gammie, L. and Hrudey, S. E. (1994). Water Taste and Odour Study (Athabasca River 1993). prepared for the Northern River Basins Study Board.

Reports FPA, CLSA results for a time of travel study for the presence of taste and odour compounds in the Athabasca River during February and March of 1993. The results indicate that the Hinton combined effluent (municipal wastewater treatment plant discharge and BKME) resulted in an impact on the odour of the Athabasca River for substantial distances downstream (>1000 km).

Kopfler, F. C., Ringhand, H. P. and Miller, R. G. (1986). A comparison of seven methods for concentrating organic chemicals from environmental water samples. Organic Pollutants in Water. Sampling, Analysis, and Toxicity Testing. Washington, D.C., 425-436.

General summary of various methods of concentration (removal of water) and isolation (removal of organics from water) techniques commonly used for environmental water analyses.

Korhonen, I. O. O., Knuutinen, J., Lahtiperä, M. and Klein, P. (1988). Mass spectra of methoxychlorobenzenes (chloroanisoles). Biomedical & Environmental Mass Spectrometry **17**: 449-454.

Good compilation of mass spectra of all 19 ring-substituted chlorinated anisoles (odorous compounds which are found in spent bleach liquors of pulp mills).

Korth, W., Bowmer, K. and Ellis, J. (1991). New Standards for the determination of geosmin and methylisoborneol in water by gas chromatography / mass spectrometry. Water Research **25**(3): 319-324.

Summary of the synthesis of deuterated geosmin and deuterated methylisoborneol, as well as their use as internal standards using closed loop stripping.

Korth, W., Ellis, J. and Bowmer, K. (1992). The stability of geosmin and MIB and their deuterated analogues in surface waters and organic solvents. Water Science and Technology **25**(2): 115-122.

Outlines proper storage procedures for the deuterated geosmin and 2-methylisoborneol standards.

Krasner, S. W., Hwang, C. J. and MacGuire, M. J. (1981). Development of a closed-loop stripping technique for the analysis of taste- and odour-causing substances in drinking water. Advances in the Identification and Analysis of Organic Pollutants in Water. Vol. 2. Ann Arbor, MI, Ann Arbor Science Publishers. 689-710.

Detailed discussion of the development and application of closed-loop stripping for the analysis of water samples and microbiological cultures for the determination of geosmin, MIB, IPMP, IBMP, and 2,3,6-TCA at levels below threshold odour concentrations.

Krasner, S. W., Hwang, C. J. and McGuire, M. J. (1983). A standard method for quantification of earthy-musty odorants in water, sediments, and algal cultures. Water Science and Technology **15**: 127-138.

Report of method development for extraction analysis of geosmin, 2-methylisoborneol, 2-isopropyl-3-methoxypyrazine, 2-isobutyl-3-methoxypyrazine, and 2,3,6-trichloroanisole from various matrices. Includes detailed diagrams of modified closed-loop stripping apparatus.

Lalezary, S., Pirbazari, M., McGuire, M. J. and Krasner, S. W. (1984). Air stripping of taste and odour compounds from water. Journal AWWA **76**(3): 83-86.

Summary of Henry's law constants for geosmin, 2-methylisoborneol, 2-isopropyl-3-methoxypyrazine, 2-isobutyl-3-methoxypyrazine, and 2,3,6-trichloroanisole. Results presented indicate that only the trichloroanisoles can be effectively and economically removed using air stripping.

Lee, H. B. (1988). Determination of twenty-one chloroanisoles in water and sediment samples. Journal of the Association of Official Analytical Chemistry **71**(4): 803-807.

Methods specific for the extraction of chloroanisoles are described. Useful table of the relative abundances of characteristic ions of the electron impact mass spectra of 21 chloroanisoles is included.

Leenheer, J. A. (1984). Concentration, Partitioning and Isolation Techniques for Volatiles. Water Analysis Vol. III Organic Species. Academic Press. 100-115.

General outline of the benefits and limitations of the various methods for extraction and isolation of volatiles from water.

Likens, S. T. and Nickerson, G. B. (1964). Detection of certain hop oil constituents in brewing products. American Society of Brewing Chemistry Proceedings: 5-13.

Describes the development and construction of a special distillation unit which uses a combination of steam distillation and continuous liquid-liquid extraction for the recovery of steam volatile compounds.

Lin, D. C. K., Foltz, R. L., Lucas, S. V., Petersen, B. A., Slivon, L. E. and Melton, R. G. (1979). Glass capillary gas chromatographic mass spectrometric analysis of organics in drinking water concentrates and advanced waste treatment water concentrates - II. Measurement of Organic Pollutants in Water and Wastewater, ASTM STP 686.

Summary of a basic partitioning scheme for the fractionation of various classes of organics found in water and wastewater.

Lundgren, B., Borén, H., Grimvall, A. and Sävenhed, R. (1989). Isolation of off-flavour compounds in water by chromatographic sniffing and preparative gas chromatography. Journal of Chromatography **482**: 23-34.

Describes the use preparative gas chromatography and sensory evaluation in the isolation and identification of specific odour compounds, including technical measures to guarantee optimum performance.

Lundgren, B. V., Borén, H., Grimvall, A., Sävenhed, R. and Wigilius, B. (1988). The efficiency and relevance of different concentration methods for the analysis of off-flavours in water. Water Science and Technology **20**(8/9): 81-89.

Report of results found when comparing open stripping with and without sodium chloride addition, XAD-2 adsorption and dichloromethane extraction. Preparative gas chromatography for fractionation of extracts is also discussed. The study showed that the original complex odour of certain surface water samples may be re-created from the dichloromethane extracts but not from the corresponding stripping extracts.

Mallevialle, J. and Suffet, I. H. (1987). Identification and Treatment of Tastes and Odors in Drinking Water.

Comprehensive and detailed summary of all facets of the general study of taste and odour problems in water.

McGuire, M. J., Krasner, S. W., Hwang, C. J. and Izaguirre, G. (1981). Closed-loop stripping analysis as a tool for solving taste and odor problems. Journal AWWA **73**(10): 530-537.

Site specific description of analytical method used to identify compounds responsible for taste and odour problems in southern California.

McGuire, M. J., Krasner, S. W., Hwang, C. J. and Izaguirre, G. (1983). An early warning system for detecting earthy-musty odors in reservoirs. Water Science and Technology **15**: 267-277.

Application of closed-loop stripping extraction for analysis of 2-methylisoborneol produced by cyanobacterial cultures.

Neilson, M. A., Stevens, R. J. J., Biberhofer, J., Goulden, P. D. and Anthony, D. H. J. (1988). Large-Sample Extractor for Determining Organic Contaminants in the Great Lakes. Environment Canada Technical Bulletin No. 157.

Description of the construction and operation of large scale sample extractors onboard a sample collection ship are described. The use of the "Goulden extractor" with continually added surrogate standards is detailed.

Onuska, F. I., Kominar, R. J. and Terry, K. (1983). An evaluation of splitless and on-column injection techniques for the determination of priority micropollutants. Journal of Chromatographic Science **21**: 512-518.

Detailed discussion of benefits of cool-on-column injection method for pg/µL levels of organics.

Potter, D. W. and Pawliszyn, J. (1992). Detection of substituted benzenes in water at the pg/mL level using solid-phase microextraction and gas chromatography-ion trap mass spectrometry. Journal of Chromatography **625**: 247-255.

Discussion of the use of solid-phase microextraction for the extraction and analyses of aqueous for volatile compounds such as benzene, toluene, ethyl benzene, and xylene isomers (BTEX).

Ramstad, T. and Walker, J. S. (1992). Investigation of musty odour in pharmaceutical products by dynamic headspace gas chromatography. Analyst **117**: 1361-1366.

Discussion of dynamic headspace purge and trap method for the extraction of odorous compounds from solid matrices.

Sävenhed, R. (1986). Chemical and Sensory Analysis of Off-flavour Compounds in Drinking Water. Linköping University, Sweden.

Detailed discussion of analytical methods, sensory properties, identification of off-flavour compounds, and the evaluation of water treatment methods for the removal of off-flavour compounds.

Sävenhed, R., Borén, H. and Grimvall, A. (1985). Stripping analysis and chromatographic sniffing for the source identification of odorous compounds in drinking water. Journal of Chromatography **328**: 219-231.

Chemical and sensory methods have been combined into an improved technique for the source identification of odorous compounds in drinking water. Open stripping concentration methods were studied and a specific case of a chlorobleaching pulp mill discharge was used to demonstrate the source identification for odorous river water. Reports that the pulp mill discharge did cause a significant increase in the threshold odour number of the river water immediately downstream from the pulp mill The characteristic pulp mill odour profile is given.

Sävenhed, R., Borén, H., Grimvall, A. and Tjeder, A. (1983). Stripping techniques for the analysis of odorous compounds in drinking water. Water Science and Technology **15**: 139-148.

Discussion of methods of improving closed-loop stripping recoveries, including elevated stripping temperatures, prolonged stripping times and use of open stripping.

Vanderstraeten, P., Wauters, E., Muylle, E., Verduyn, G., Vanderheyden, E., and Vansant, E.F. (1988). A continuous qualitative detection method for total mercaptans, organic sulfides, H_2S, and CS_2 for odoriferous emissions. JAPCA, 38; 1271-1274.

Lists common odour descriptors for various sulfur compounds.

Veijanen, A. (1990). An Integrated Sensory and Analytical Method for Identification of Off-Flavour Compounds. University of Jyväskylä Ph.D. Dissertation. Jyväskylä, Finland.

Detailed summary of an integrated sensory and analytical approach to the study of odorous compounds in many different matrices.

Veijanen, A., Lahtiperä, M. and Paasivirta, J. (1988). Analytical methods of off-flavours in the aquatic environment. Water Science and Technology **20**(2): 183-184.

Short description of results given in Veijanen, et al. 1983.

Veijanen, A., Lahtiperä, M., Paukku, R., Kääriäinen, H. and Paasivirta, J. (1983). Recent development in analytical methods for identification of flavour compounds. Water Science and Technology **15**: 161-168.

Summary of early analytical methods used for identification of off-flavour compounds.

Wajon, J. E., Alexander, R. and Kagi, R. I. (1985). Determination of trace levels of dimethyl polysulphides by capillary gas chromatography. Journal of Chromatography **319**: 187-194.

Discussion of the analyses of various dimethyl polysulphides using cool-on-column injection. Also report decomposition and disproportionation reactions with heated active surfaces such as a vaporizing injection port.

Wood, N. F. and Snoeyink, V. L. (1977). 2-methylisoborneol, improved synthesis and a quantitative gas chromatographic method for trace concentrations producing odor in water. Journal of Chromatography **132**: 405-420.

Discussion of the synthesis of 2-methylisoborneol and the use of camphor as a representative internal standard for dichloromethane extractions of odorous water.

Zhang, Z. and Pawliszyn, J. (1993). Headspace solid-phase microextraction. Analytical Chemistry **65**: 1843-1852.

Discussion of the specific application of solid-phase microextraction for the analysis of compounds that are easily volatilized from an aqueous sample. These volatile compounds are extracted from the headspace by introducing the fused silica coated fiber into the headspace of the sealed sample vial, allowing for the volatile compounds to partition to the silica fiber and then transferring the fiber to the heated GC injection port.

2.0 Control or Treatment Options

Anselme, C., Suffet, I. H. and Mallevialle, J. (1988). Effects of ozonation on tastes and odors. Journal AWWA **80**: 45-51.

Ozonation was effective for removing musty, earthy, fishy, and muddy tastes and odors from water. Astringent and plastic tastes were removed and the ozonation process was found to be responsible for the development of high-intensity fruity odours which correlated with the presence of aliphatic and aromatic aldehydes and ketones.

Baker, R. J., Suffet, I. H., Anselme, C. and Mallevialle, J. (1986). Evaluation of conventional water treatment methods for removal of taste and odor from drinking water. AWWA WQTC: 765-786.

The effectiveness of coagulation, oxidation and activated carbon for removing taste and odour from drinking water were studied in the laboratory and in a full-scale water treatment plant. It was concluded that coagulation was ineffective, oxidation was effective for some specific compounds, and activated carbon was generally but not always effective.

Fok, N., Huck, P. M., Walker, G. S. and Smith, D. W. (1984). Evaluation of drinking water treatment alternatives for taste and odour reduction. Water Pollution Research Journal of Canada **19**(1): 119-131.

Chlorine dioxide, ozonation and granular activated carbon were evaluated at the pilot scale for the removal of a taste and odour compound (ethylbenzene).

Gergov, M., Priha, M., Talka, E., Valttila, K., A. and Kukkonen, K. (1988). Chlorinated organic compounds in effluent treatment at kraft mills. Tappi Journal **71**(12): 175-184.

Material balances were determined at two kraft mills in Finland for total organically bound chlorine, chlorophenolics, volatile chlorine compounds and chloroacetones. The formation of chlorinated phenolics in hardwood pulp bleaching was only about a third of that for softwood pulp bleaching. Extended pulping of softwood and the use of chlorine dioxide substitution during hardwood bleaching reduced the formation of chlorinated phenolics. Activated sludge treatment removed 75-95% of chlorinated phenolics in kraft effluents.

Glaze, W. H., Schep, R., Chauncey, W., Ruth, E. C., Zarnoch, J. J., Aieta, E. M., Tate, C. H. and McGuire, M. J. (1990). Evaluating oxidants for the removal of model taste and odor compounds from a municipal water supply. Journal AWWA **82**(5): 79-84.

Nine oxidizing agents were evaluated for removing six taste and odour compounds (1- hexanal, 1-heptanal, dimethyltrisulfide, 2,4-decadienal, MIB, and geosmin) spiked into river water. Ozone alone and ozone with hydrogen peroxide appear to have the most potential for control of taste and odour compounds.

Gordon, M. R., Mueller, J. C. and Walden, C. C. (1980). Effect of biotreatment on fish tainting propensity of bleached kraft whole mill effluent. Transactions of the Technical Section of the Canadian Pulp and Paper Association **6**(1): TR2-TR8.

Biotreatment did not reduce the tainting propensity of BKME as effectively as it reduced BOD and acute toxicity. Apparently some of the constituents in BKME responsible for fish flesh tainting are not readily biodegradable.

Hansen, R. E. (1972). Granular carbon filters for taste and odour removal. Journal AWWA **78**(3): 176-181.

Early report of granular carbon filters used in producing water free of tastes and odours.

Hattori, K. (1988). Water treatment systems and technology for the removal of odour compounds. Water Science and Technology **20**(8/9): 237-244.

Report raw water concentrations of 103 ng/L MIB and 362 ng/L geosmin. In subsequent removal studies the researchers found that GAC effectively removed the odour compounds, PAC gave poor results, ozonation was problematic and produced additional odour compounds, bio-film contractors removed geosmin but not MIB.

Hoehn, R. C. (1965). Biological methods for the control of tastes and odours. Southwest Water Works Journal (June): 26-30.

Presentation of the research findings which employed a new approach for the control of biogenic tastes and odours, namely, that of biological control rather than chemical treatment. An early report of the sue of microorganisms to remove various organic taste and odour compounds.

Hrubec, J. and de Kruijf, H. A. M. (1983). Treatment methods for the removal of off-flavours from heavily polluted river water in the Netherlands - A review. Water Science and Technology **15**: 301-310.

A limited review of treatment methods for the removal of taste and odour compounds from water. The most effective treatment processes for improvement of taste and odour were reported to be ozonation and granular activated carbon.

Krasner, S. W. (1988). Flavor-profile analysis; an objective sensory technique for the identification and treatment of off-flavors in drinking water. *Water Science and Technology* **20**(8/9): 31-36.

General description of the flavour profile analysis method and its use when specific causative agents had not been identified. Also includes specific application of the flavour profile analysis to resolve odour problems in a drinking water treatment plant by varying free-chlorine dosages and contact times.

Krasner, S. W., Barrett, S. E., Dale, M. S. and Hwang, C. J. (1989). Free chlorine versus monochloramine for controlling off-tastes and off-odors. *Journal AWWA* **81**(2): 86-93.

Chloramination was shown to have a lesser ability to oxidize odorous compounds than free chlorine. A combination of sensory, chemical and microbiological methods were used to analyze, monitor and control the off-flavour compounds in water.

Lalezary, S., Pirbazari, M., McGuire, M. and Krasner, S. W. (1984). Air stripping of taste and odour compounds from water. *Journal AWWA* **76**(3): 83-87.

The Henry's law constants were determined for IPMP, IBMP, MIB, 2,3,6-TCA and . The results indicate insignificant strippabilities at neutral pH for IPMP, IBMP, MIB and geosmin. Air stripping may be effective for the removal of TCA.

Lalezary-Craig, S., Pirbazari, M., Dale, M. S., Tanaka, T. S. and McGuire, M. J. (1988). Optimizing the removal of geosmin and 2-methylisoborneol by powdered activated carbon. *Journal AWWA* **78**(3): 73-80.

Powdered activated carbon dosages of 10 mg/L could bring geosmin and MIB levels of up 66 ng/L down to levels below threshold odour concentrations. Chlorine and monochloramine residuals had an adverse effect on removal efficiencies, as did the presence of humic acids.

Lin, S. D. (1977). *Tastes and odors in water supplies; a review*. Water and Sewage Works (Reference Issue): R141-R163.

Includes lengthy review of treatment and removal of taste and odour compounds.

Lundgren, B. V., Grimvall, A. and Sävenhed, R. (1988). Formation and removal of off-flavour compounds during ozonation and filtration through biologically active sand filters. *Water Science and Technology* **20**(8/9): 245-253.

Reports the formation of some odorous compounds during ozonation. These ozonation bi-products were only partly removed during biologically active sand filtration.

Mallevialle, J. and Suffet, I. H. (1987). Identification and Treatment of Tastes and Odors in Drinking Water. Denver.

Comprehensive and detailed summary of all facets of the general study of taste and odour problems in water.

McGuire, M. J. and Gaston, J. M. (1988). Overview of technology for controlling off-flavours in drinking water. Water Science and Technology **20**(8/9): 215-228.

Update of reviews on the treatment methods for the control of taste and odour in water. Stresses the use of reliable, reproducible, easy-to-use sensory analytical techniques or instrumental analytical techniques to follow removal efficiencies.

Montiel, A. J. (1983). Municipal drinking water treatment procedures for taste and odour abatement - a review. Water Science and Technology **15**: 279-289.

A review of clarification, oxidation and adsorption processes for the removal of taste and odour compounds. Discussion of taste and odour compound production at the disinfection stage of drinking water treatment.

Naish, V. A., Findlay, D. M., McLean, R. A. N., Ross, R. F., Lemieux, J. and Quesnel, M. B. (1982). Treatablility of water downstream of pulp mills on a multi-use river. Technical Section, CPPA Environment Improvement Conference, Halifax, Nova Scotia: 105-108.

Brief discussion of taste and odour contributions of kraft mills on a river receiving multiple industrial and municipal effluents. Treatability for the removal of tastes and odours was studied. Reports that no single industry (including kraft mill effluent) or municipality was responsible for a major change in taste and odour levels.

Parker, W. J., Hall, E.R., and Farquhar, G. J. (1993). Evaluation of dechlorination mechanisms during anaerobic fermentation of bleached kraft mill effluent. Water Research **27**(8): 1269-1273.

Detailed discussion of the possible nucleophilic substitution of sulfide to improve dechlorination by substitution for chloride on compounds which might not otherwise be biodegradable in an anaerobic kraft mill bleach plant treatment system.

Parker, W. J., Farquhqar, G. J. and Hall, E. R. (1993). Removal of chlorophenolics and toxicity during high-rate anaerobic treatment of segregated kraft mill bleach plant effluents. Environmental Science and Technology **27**: 1783-1789.

The behavior of selected chlorinated phenols, guaiacols, catechols, and vanillins during high-rate anaerobic treatment of segregated kraft mill bleach plant effluents was investigated. Mono and di-substituted phenols were generally poorly removed, while trichlorophenol removal was >80%. Chlorinated guaiacols and vanillins removal efficiencies were >95%, while catechols removal efficiencies (except for 3,5 chlorocatechol) were >95% with cosubstrate supplementation.

Saunamäki, R., Jokinen, K., Järvinen, R. and Savolainen, M. (1991). Factors affecting the removal and discharge of organic chlorine compounds at activated sludge treatment plants. Water Science and Technology **24**(3/4): 295-307.

Two extensive mill-scale tests were performed to find out extent of removal of AOX and chlorinated phenolics. In the extended aeration activated sludge plant there was 70-80% removal of the chlorinated phenolics. In the high-load activated sludge plant the removals were only 35-55%.

Sävenhed, R., Borén, H., Grimvall, A., Lundgren, V. B., Balmér, P. and Hedberg, T. (1987). Removal of individual off-flavour compounds in water during artificial groundwater recharge and during treatment by alum coagulation/sand filtration. Water Research **21**(3): 277-283.

Alum coagulation / sand filtration did not remove the odour compounds studied, but artificial groundwater recharge in sand and gravel ridges substantially reduced odour compound concentrations.

Sigworth, E. A. (1957). Control of odor and taste in water supplies. Journal AWWA **Dec**: 1507-1521.

Activated carbon shown to be effective in taste and odour removal.

Stuthridge, T. R., Campin, D. N., Langdon, A. G., Mackie, K. L., McFarlane, P. N. and Wilkins, A. L. (1991). Treatability of bleached kraft pulp and paper mill wastewaters in a New Zealand aerated lagoon treatment system. Water Science and Technology **24**(3/4): 309-317.

The effectiveness of two biological treatment systems operating at a kraft softwood integrated pulp and paper mill were assessed. The results indicate that the treatability of bleached kraft pulp and paper mill wastewater constituents is dependent upon the characteristics of the treatment systems and the composition of the wastewaters.

Vik, E. A., Storhaug, R., Naes, H. and Utkilen, H. C. (1988). Pilot scale studies of geosmin and 2-methylisoborneol removal. Water Science and Technology **20**(8/9): 229-236.

Used a pilot scale water treatment plant to study removal efficiencies of geosmin and MIB using granular activated carbon and ozone. The results indicate that the GAC process showed better removal than the ozonation-GAC combination.

Voss, R. H. (1983). Chlorinated neutral organics in biologically treated bleached kraft mill effluents. Environmental Science and Technology **17**(9): 530-537.

Biologically treated combined mill effluents were sampled at nine bleached kraft mills. Only four main compounds were reported (chloroform, a,a-dichlorodimethyl sulfone, a,a,a'-trichlorodimethyl sulfone and a,a,a',a'-tetrachlorodimethyl sulfone). The chlorinated sulfones were reported as relatively resistant to removal by biological treatment in an aerated lagoon.

Voudrias, E. A., Snoeyink, V. L. and Larson, R. A. (1986). Desorption of organics formed on activated carbon. Journal AWWA **78**: 82-86.

The undesirable effects of the contact of chlorine with GAC are discussed, including the formation of taste and odour compounds with prechlorination.

Wong, A., Voss, R. H., Kovacs, T. G. and Dorica, J. G. (1985). Drinking water organoleptic quality as influenced by biologically treated bleached kraft mill effluent. Journal of Pulp and Paper Science **11**(6): 161-166.

The effect of biologically treated BKME on the taste and odour of water downstream of discharges was evaluated for four different mill/river sites in western Canada. Significant differences were reported for the odour thresholds and resulting contributions to the taste and odour between various mills.

Woods, S. L., Ferguson, J. F. and Benjamen, M. M. (1989). Characterization of chlorophenol and chloromethoxybenzene biodegradation during anaerobic treatment. Environmental Science and Technology **23**(1): 62-68.

Report the ability of anaerobic consortia to reductively dechlorinate highly chlorinated compounds. Once dechlorinated, lesser chlorinated phenols may be readily degraded in aerobic treatment processes.

3.0 Fish Tainting Relating to Non-Pulp Mill Effluents

Davis, H. K., Geelhoed, E. N., MacRae, A. W. and Howgate, P. (1992). Sensory analysis of trout tainted by diesel fuel in ambient water. Water Science and Technology **25**(2): 11-18.

Rainbow trout were exposed to several different concentrations of diesel using a flow through system with the used water discharged to waste. The threshold of exposure concentration which led to tainting was found to be 0.08 mg/L

From, J. and Hørlyck, V. (1984). Sites of uptake of geosmin, a cause of earthy-flavour in rainbow trout (Salmo gaidneri). Canadian Journal of Fisheries and Aquatic Sciences **41**: 1224-1226.

The earthy flavour in rainbow trout was acquired most rapidly via the gills, followed in descending order by the skin, small intestine, and stomach.

Hrudey, S. E. and Nelson, L. R. (1986). Review of Oil Sands Tailing Pond Contaminants with Fish Tainting Potential. prepared for Research Management Division, Alberta Environment, Edmonton, RMD by Steve E. Hrudey and Associates Ltd.

Review of literature specifically relevant to the tainting of fish by petroleum-derived compounds found in oil sands wastewater.

Johnsen, P. B. and Lloyd, S. W. (1992). Influence of fat content on uptake and depuration of the off-flavor 2-methylisoborneol by channel catfish (Ictalurus punctatus). Canadian Journal of Fisheries and Aquatic Sciences **49**: 2406-2411.

Fatter fish (>2.5% muscle fat) accumulated nearly 3 times more MIB than lean fish (<2%).

Koning, C. W. and Hrudey, S. E. (1992). Sensory and chemical characterization of fish tainted by exposure to oil sand wastewaters. Water Science and Technology **25**(2): 27-34.

Reports on experiments where rainbow trout were exposed to a variety of wastewaters from oil sands plants. Found that all wastewaters tainted fish to a detectable degree based on sensory assessment by a trained panel. Analysis of fish flesh indicated that total phenols exceeded published odour threshold but organic sulphur compounds were also implicated.

Kuusi, T. and Suihko, M. (1983). Occurrence of various off-flavours in fish in Finland from 1969 to 1981. Water Science and Technology (15): 47-58.

The flavour panel used in this study reported that the most common taints were oil, kraft pulp mill effluent, and sewage types of off-flavours. The most common off-flavour was kraft pulp mill effluent which was present in 41 % of their unacceptable samples.

Lindsay, R. C. and Heil, T. P. (1992). Flavor tainting of fish in the Upper Wisconsin River caused by alkyl- and thiophenols. Water Science and Technology **25**(2): 35-40.

Alkylphenols and thiophenols were identified as the principle contributors to flavour-tainting in fish during the spring, while geosmin and MIB were major contributors in fish captured in the late summer

Malins, D. C. and Hodgins, H. O. (1981). Petroleum and Marine fishes: a review of uptake, deposition, and effects. Environmental Science and Technology **15**(11): 1273-1280.

Review of fish tainting caused by petroleum. Includes sections on uptake, bioconcentration, and deposition.

Martin, J. F., Bennett, L. W. and Graham, W. H. (1988). Off-flavor in the channel catfish (Ictalurus punctatus) due to 2-methylisoborneol and its dehydration products. Water Science and Technology **20**(8/9): 99-105.

Off-flavor in catfish was found to be due to contributions by 2-methylisoborneol, 2- methyleneborane and 2-methyl-2-bornene.

Martin, J. F., Plakas, S. M., Holley, J. H. and Kitzman, J. V. (1990). Pharmacokinetics and tissue disposition of the off-flavor compound 2- methylisoborneol in the channel catfish (Ictalurus punctatus). Canadian Journal of Fisheries and Aquatic Sciences **47**: 544-547.

MIB was more concentrated in peritoneal fat and sub-epidermal adipose tissue than in other tissues. Half-lives of 0.14 hours for distribution and 3.62 hours for elimination were found for plasma clearance of MIB.

McIntyre, A. D. (1982). Oil pollution and fisheries. Phil. Trans. R. Soc. Lond. **B 297**: 401-411.

General review of the effects that oil pollution has on the fisheries industry. Includes a short section on fish tainting problems.

Motohiro, T. (1983). Tainted fish caused by petroleum compounds - a review. Water Science and Technology **15**: 75-84.

A review of the compounds present in petroleum that can produce and "oily" flavour in fish. The general classes of these tainting compounds are; saturated and unsaturated paraffins, aromatic hydrocarbons, and sulfur compounds.

Persson, P. E. (1984). Uptake and release of environmentally occurring odorous compunds by fish. Water Research **18**(10): 1263-1271.

Good review of chemical compounds present in lakes and rivers that may impair flavour of fish.

Persson, P. E. (1980). "Muddy odour in fish from hypertrophic waters." Developments in Hydrobiology **2**: 203-208.

Investigation into tainting of fish due to natural sources of muddy odour compounds.

Roubal, W. T., Stranahan, S. I. and Malins, D. C. (1978). The accumulation of low molecular weight aromatic hydrocarbons of crude oil by coho salmon and Starry Flounder. Archives of Environmental Contaminant Toxicology **7**: 237-244.

Report of bioconcentration of various organic compounds in salmon and flounder. Indicate that bottom dwelling flounder accumulated more hydrocarbons than the salmon, and that the flounder did not depurate some compounds as readily as the salmon after being moved to clean water.

Shumway, D. L. and Palensky, J. R. (1973). Impairment of the flavour of fish by water pollutants. Department of Fisheries and Wildlife, Corvallis, Oregon. EPA-R3-73-010.

Flavour imparting capacity and estimated threshold concentrations were determined for a number of organic compounds and effluents. Results indicated that the flavour panel found large numbers of fish that were described as tainted by, kraft pulp mill effluent, or sewage types of tastes.

Stansby, M. (1978). Flavours in fish from petroleum pickup. Maritime Fisheries Review **40**: 13-17.

Review of fish tainting as a result of exposure to petroleum products.

Thomas, N. A. (1973). *Assessment of fish flesh tainting substances*. Biological methods for the Assessment of Water Quality, ASTM STP 528, American Society for Testing and Materials.

Substantial documentation of a study of tainting of fish flesh by chemicals and/or effluents discharged into the Ohio River. Caged fish were used to study the tainting potential of a large number of industries.

Tidmarsh, W. G. and Ackman, R. G. (1986). Fish tainting and hydrocarbons in the environment: A perspective. *Spill Technology Newsletter* **II**(3): 76-86.

Details a study initiated in 1984 to review possible fish tainting caused by the introduction of hydrocarbons into the marine environment as a result of oil industry activities. The study indicates that tainting is usually limited to organisms present in shallow protected coastal waters where circulation is limited.

Vandermeulen, J. H. (1986). A review of toxicity and sublethal effects of petroleum hydrocarbons on freshwater biota. *Oil and Freshwater - Chemistry, Biology and Countermeasure Technology*. Pergamon Press. 267-303.

General review of the effects of oil on freshwater biota. Includes a section on tainting of fish and shell-fish due to exposure to petroleum hydrocarbons and a discussion of the difficulties surrounding the study of fish tainting substances.

Yurkowski, M. and Tabachek, J. L. (1974). Identification, analysis, and removal of geosmin from muddy-flavored trout. *Journal of the Fisheries Resources Board of Canada* **31**: 1851-1858.

Placing of muddy flavored trout in clean water resulted in reduction of flavour to non-tainting levels within 3 to 5 days. A method for the quantitative estimation of geosmin in fish flesh is described.

Yurkowski, M. and Tabachek, J. L. (1980). Geosmin and 2-methylisoborneol implicated as a cause of muddy odor and flavor in commercial fish from Cedar Lake, Manitoba. *Canadian Journal of Fisheries and Aquatic Sciences* **37**: 1449-1450.

The naturally occurring compounds, geosmin and 2-methylisoborneol were found to cause a strong muddy flavour in fish. The presence of these compounds caused the closure of a commercial fishing season.

4.0 Fish Tainting Relating to Pulp Mill Effluents

Berg, N. (1983). Chemical and sensory analysis of off-flavours in fish from polluted rivers in Norway. Water Science and Technology **15**: 59-65.

Fish caught in water polluted with paper mill effluents were analyzed using a GC headspace technique. Tainting compounds such as terpenes and their derivatives were reported to cause strong taints. Low levels of halogenated compounds were also found.

Cook, W. H., Farmer, F. A., Kristiansen, O. E., Reid, K., Reid, J. and Rowbottom, R. (1973). The effect of pulp and paper mill effluents on the taste and odour of the receiving water and the fish therein. Pulp and Paper Magazine of Canada, Convention Issue.

The effluent sources from a pulp mill were evaluated for their water and fish tainting potential. It was concluded that the kraft mill was the largest odour contributor and the sulphite mill was the main source of taste imparted by the mill effluent to the river water. In the fish tainting studies it was concluded that condensates (i.e. evaporator condensates, digester foul condensates, and the recovery furnace flue gas condensates) were the main process waste sources of fish tainting materials.

Domtar Fine Papers Ltd. (1971). Effect of Pulp and Paper Mill Effluents on the Taste and Odour of Water and Fish. Pulp and Paper Pollution Abatement, Environment Canada.

Reports results of a detailed sensory evaluation of the effects of pulp and paper mill effluents on tainting of fish. Very basic sensory evaluations methods were used, but a thorough study of the tainting potential of all process streams was carried out.

Farmer, F. A., Neilson, H. R. and Esar, D. (1973). Flavour evaluation by triangle and hedonic scale tests of fish exposed to pulp mill effluents. Canadian Institute of Food Science Technology Journal **6**(1): 12-16.

Tainting was found in caged perch exposed to evaporator condensates, foul water condensate, and the recovery furnace flue gas condensate. The most effective effluent treatments in increasing order of value were Turbulent Contact Absorber, activated sludge and aerated lagoon. Fish taken from downstream of the total effluent showed no flavour tainting when compared to fish taken upstream of the effluent.

Fetterolf, C. M., Jr. (1963). *Purdue University Extension series* **48**(3): 174-182.

Discussion of tainting effects in fish over different seasons. It was noted that complaints about tainted fish on lakes receiving water from pulp mills and other industry were mainly by ice fishermen in late winter. However, in fish taken from rivers receiving pulp mill wastes and other industrial discharges, most tainting incidents occurred in the spring.

Gordon, M. R., Mueller, J. C. and Walden, C. C. (1980). Effect of biotreatment on fish tainting propensity of bleached kraft whole mill effluent. *Transactions of the Technical Section of the Canadian Pulp and Paper Association* **6**(1): TR2-TR8.

Biotreatment did not reduce the tainting propensity of BKME as effectively as it reduced BOD and acute toxicity. Apparently some of the constituents in BKME responsible for fish flesh tainting are not readily biodegradable.

Heil, T. P. and Lindsay, R. C. (1988a). Sensory properties of thio- and alkyl- phenols causing flavor tainting in fish from the Upper Wisconsin River. *Journal of Environmental Science and Health* **B23**(5): 361-388.

Strong flavor tainting was found in trout exposed to 100 ppb concentrations of five added alkylphenols and thiophenols. Mixtures of 1-2 ppb concentration of the same compounds did not result in flavor tainting.

Heil, T. P. and Lindsay, R. C. (1988b). A method for quantitative analysis of flavor-tainting alkylphenols and aromatic thiols in fish. *Journal of Environmental Science and Health* **B23**(5): 475-488.

Simultaneous steam distillation-extraction with GC/MS method for flavor-tainting compound analyses was effective for 2-isopropylphenol, 3-isopropylphenol, 4-isopropylphenol, 2,4,-diisopropylphenol, 2,5-diisopropylphenol, 3,5-diisopropylphenol, carvacrol and thymol.

Heil, T. P. and Lindsay, R. C. (1988c). Volatile compounds in flavour-tainted fish from the upper Wisconsin River. *Journal of Environmental Science and Health* **B23**(5): 489-512.

Fish from a section of the Upper Wisconsin River that is influenced by several pulp and paper mills, municipalities and other industry were found to be tainted. Alklyphenols and thiophenols were identified as the principal contributors to flavour-tainting in fish during the spring while geosmin and MIB were major contributors in fish captured in the late summer.

Heil, T. P. and Lindsay, R. C. (1988d). Environmental and industrial factors relating to flavor-tainting of fish in the Upper Wisconsin River. Journal of Environmental Science and Health **B23**(5): 527-552.

Sources of the alkylphenols and thiophenols were determined. Degraded aspen wood is capable of contributing to alkylphenol tainting in Wisconsin River fish. Alkylphenols and thiophenols were found in the river sediment and benthic organisms.

Kovacs, T. (1986). Effects of bleached kraft mill effluent in freshwater fish; A Canadian perspective. Water Pollution Research Journal **21**(1): 91-118.

A good review of the sublethal effects of untreated and biologically treated bleached kraft mill effluents in freshwater fish in Canada. Includes a section reviewing literature surrounding tainting of fish attributed to pulp mill discharges.

Kovacs, T. G. (1982). The Effect of Bleached Kraft Effluent on the Aquatic Environment. prepared for Weyerhaeuser Canada Ltd. Kamloops B.C., by Pulp and Paper Research Institute of Canada.

A study comparing the flavour of mountain whitefish captured in upstream tributaries and from several kilometers downstream of a pulp mill discharge. Reported tainting of whitefish in BKM effluent concentrations ranging from 0.5 to 1.0 %v/v.

Langer, O. E. and Nassichuk, M. D. (1975). Selected Biological Studies of the Thompson River System. Environment Canada. Technical Report Series No. PAC/T-75-22.

Rainbow trout were caged less than 1 kilometer downstream from a BKME and held for up to 13 days. Significant tainting of all fish developed, and the worst tainting was recorded for the fish held immediately downstream of the mill diffuser.

Langford, R. W. (1974). Data Review of Biological and Chemical Effects of the Crestbrook Pulp Mill Effluent on the Kootenay River. Fish and Wildlife Branch, B.C. Ministry of Environment. Victoria, B.C. (As referenced in McLeay and Associates, (1987) Aquatic Toxicity of Pulp and Paper Mill Effluent: A Review. Environment Canada Report # EPS 4/PF/1.)

Carried out a number of controlled flavour evaluation studies on fish caught upstream and downstream of BKME. Although fish collected upstream and downstream were of different species, there were consistent off-flavour ratings for the downstream fish.

Liem, A. J., Naish, V. A. and Rowbottom, R. S. (1977). An Evaluation of the Effect of Inplant Treatment Studies on the Abatement of Air and Water Pollution from a Hardwood Kraft Pulp Mill. Environmental Protection Service, Ottawa, Ontario.

Fish caught from a large river receiving BKME (untreated) were not reported to have an off-flavour.

McLeay, D. and Associates Ltd. (1987). Aquatic Toxicity of Pulp and Paper Mill Effluent: A Review. Environment Canada. Report EPS 4/PF/1. April.

Includes lengthy review of off flavours resulting from pulp mill effluents. Mainly discusses fish tainting propensity of these effluents.

Millson, R. (1993). Personal Communication Alberta Environmental Protection, Peace River Alberta.

Investigation into tainting of fish flesh downstream of the Proctor and Gamble Mill on the Wapiti River was undertaken in 1990. The study results indicated that the pulp mill effluent resulted in fish tainting, but a cause/effect tainting relationship could not be established with chemical analyses.

Paasivirta, J., Klein, P., Knuutila, M., Knuutinen, J., Lahtiperä, M., Paukku, R., Veijanen, A., Welling, L., Vuorinenc, M. and Vuorinen, P. J. (1987). Chlorinated anisoles and veratroles in fish. Model compounds. Instrumental and sensory determinations. Chemosphere **16**(6): 1231-1241.

Tainting of trout was correlated with chloroanisole, veratrole, chlorophenol, guaiacol and catechol levels.

Paasivirta, J., Knuutinen, J., Tarhanen, J., Kuokkanen, T., Surma-Aho, K., Paukku, R., Haariainen, H., Lahtipera, M. and Veijanen, A. (1983). Potential off-flavour compounds from chlorobleaching of pulp and chlorodisinfection of water. Water Science and Technology **15**: 97-104.

Persistent chlorinated acids, phenols and neutral compounds were attributed to be possible fish-tainting compounds downstream of pulp bleaching and chlorodisinfection plants. Microbial metabolism of chlorophenols resulted in the formation of chloroanisoles which are even more likely to cause fish-tainting.

Paasivirta, J., Rantalainen, A. L., Welling, L., Herve, S. and Heinonen, P. (1992). Organochlorines as environmental tainting substances; taste panel study and chemical analyses of incubated mussels. Water Science and Technology **25**(2): 105-113.

Chloroanisoles and chloroveratroles were indicated to form the main tainting load in the studied mussels.

Persson, P.E. (1988). Off-flavours in fish from waters around a pulp and paper mill in Finland. Verh. Internat. Verein. Limnol. **23**: 1751-1755.

Study of fish exposed to effluents from sulphite and kraft pulp mills. Reported that species of fish differ in their susceptibility to tainting (bottom feeders less tainted in general).

Persson, P.E. (1985). Off-flavours in fish from the Gulf of Finland. Finnish Fisheries Research **6**: 112-117.

Documents off-flavours in fish found in waters below kraft pulp mills.

Shumway, D. L. and Chadwick, G. G. (1971). Influence of kraft mill effluent on the flavor of salmon flesh. Water Research **5**: 997-1003.

Salmon exposed to untreated KME for up to 96 hours did not demonstrate any flavor impairment at levels 2.9% by volume.

Shumway, D. L. and Palensky, J. R. (1973). Impairment of the Flavour of Fish by Water Pollutants. Department of Fisheries and Wildlife, Corvallis, Oregon. EPA-R3-73-010.

Flavour imparting capacity and estimated threshold concentrations were determined for a number of organic compounds and effluents using caged fish studies. Results indicated that the flavour panel found large numbers of fish that were described as tainted by, kraft pulp mill effluent, or sewage types of tastes. Those fish exposed to kraft mill effluent demonstrated and estimated threshold tainting concentration (ETTC) of >8<20 %v/v, while those exposed to a sulphite mill effluent showed and ETTC of >34<51.

Swabey, Y.H. (1965). Report on a Study of the Flavour of Fish from the Spanish River and Adjacent North Channel. Ontario Wat. Res. Comm. Toronto, Ontario.(As referenced in McLeay and Associates, (1987) Aquatic Toxicity of Pulp and Paper Mill Effluent: A Review. Environment Canada Report # EPS 4/PF/1.)

Significant off-flavour in pike and pickerel from a river receiving bleached kraft mill effluent and mechanical pulping mill effluent.

Vuorinen, P. J., Paasivirta, J., Piilola, T., Surma-Aho, K. and Tarhanen, J. (1985). Organochlorine compounds in Baltic salmon and trout. I. Chlorinated hydrocarbons and chlorophenols 1982. Chemosphere **14**(11/12): 1729-1740.

Organochlorine compounds (including some potential tainting compound) levels were determined for the muscle liver and eggs of Baltic salmon and trout levels were frequently highest in the livers.

Weinbauer, J. D., Thiel, D. A., Kaczynski, V. W. and Martin, C. S. (1980). Receiving stream fisheries studies relative to secondary treated pulp and paper mill effluents. Tappi **63**(10): 121-125.

A study documenting the biological status of a 70-mile stretch of the Wisconsin River receiving treated pulp and paper mill effluents. Palatability tests of fish caught in the immediate vicinity of the pulp mill effluents indicated slight taste impairment. Other effluents and non-point source pollution entering the Wisconsin River upstream of the mills were found to have an even more adverse effect than the effluents from the mills.

Wells, D.L. (1967). Report on the Flavour of Whitefish from Nipigon Bay 1967. Ontario Wat. Res. Comm. Toronto, Ontario.(As referenced in McLeay and Associates, (1987) Aquatic Toxicity of Pulp and Paper Mill Effluent: A Review. Environment Canada Report # EPS 4/PF/1.).

Significant off-flavour in whitefish from a lake receiving bleached kraft mill effluent and mechanical pulping mill effluent.

Whittle, D. M. and Flood, K. W. (1977). Assessment of the acute toxicity, growth impairment, and flesh tainting potential of a bleached kraft mill effluent on rainbow trout (Salmo gairdneri). Journal of the Fisheries Resources Board of Canada **34**(6): 869-878.

Significant flavour impairment was found at 3% vol./vol. bleached kraft mill effluent concentration. No flavour impairment was detected at effluent concentrations of 2% vol./vol. Flavour impairment was found to be the most sensitive sublethal response measured.

5.0 Sensory Methods of Odorous Compound Analysis

Abe, H., Kanaya, S., Komukai, T., Takahashi, Y. and Sasaki, S.-I. (1990). Systemization of semantic descriptions of odors. Analytica Chimica Acta **239**: 73-85.

Extensive list of 126 different odour descriptors that can serve as a standard set to be used by flavour panels.

Abe, H., Yoshimura, T., Kanaya, S., Takahashi, Y., Miyashita, Y. and Sasaki, S.-I. (1987). Automated odor-sensing system based on plural semiconductor gas sensors and computerized pattern recognition techniques. Analytica Chimica Acta **194**: 1-9.

Limited discussion of use of semiconductor gas-sensor elements for automated odour detection. Odors detectable are limited to ethereal, ethereal-minty, ethereal-pungent and pungent substances.

Amoore, J. E. (1992). Odor standards in squeeze-bottle kits for matching quality and intensity. Water Science and Technology **25**(2): 1-9.

Description of a series of 19 common odorants available as a test kit for odour type and strength comparisons when training and standardizing a flavour profile panel. The squeeze-bottles test kits are prepared using polypropylene bottles with flip-top caps, containing the chosen odorant dissolved in virtually odorless mineral oil which is absorbed in polypropylene felt.

APHA-AWWA-WEF (1992). Standard Methods for the Examination of Water and Wastewater, 18th Edition, American Public Health Association pp 2-15 to 2-23.

Technical and detailed summaries of standard methods used for the flavour threshold test (FTT), the flavour rating scale (FRS), and the flavour profile analysis (FPA).

ASTM (1968). Manual on Sensory Testing Methods.

Early manual to guide the inexperienced panel on the correct procedures for conducting sensory tests. Summarizes many of the test forms, special applications, and statistical procedures used in the food industry for odour and taste evaluations.

Baker, R. A. (1963). Threshold odors of organic chemicals. Journal AWWA **55**(7): 913-916.

Summary of flavour panel methods used to determine odour threshold concentrations for 32 chemicals.

Bartels, J. H. M., Brady, B. M. and Suffet, I. H. (1987). Training panelists for the flavor profile analysis method. Journal AWWA **79**: 26-32.

Presents a general introduction about panel training for the flavour profile analysis method.

Bartels, J. H. M., Burlingame, G. A. and Suffet, I. H. (1986). Flavor profile analysis; taste and odor control of the future. Journal AWWA **78**(3): 50-55.

General discussion of the feasibility of developing a standardization of sensory analysis in the water treatment industry by using a panel well-trained in the flavour profile analysis method.

Boelens, H., van der Linde, L. M., de Rijke, D., de Valnois, P. J., van Dort, J. M. and Takken, H. J. (1978). Chemistry and flavour. I. Molecular structure and organoleptic quality. Chemical Society Reviews **7**: 167-176.

Basic discussion of the chemical properties of odorous compounds. The properties discussed include, volatility, hydrophobicity, lipophilicity, concentration, functional groups, chirality (size and shape), and molecular flexibility (rotation and vibration at molecular bonds).

Bruvold, W. H. (1975). Human perception and evaluation of water quality. CRC Critical Reviews in Environmental Control **March**: 153-231.

Very detailed and lengthy review of the research from 1955 to 1975 that dealt with human perception and evaluation of the quality of water destined for uses which involve personal contact or consumption.

Bruvold, W. H. (1989). A critical review of methods used for the sensory evaluation of water quality. CRC Critical Reviews in Environmental Control **19**(4): 291-308.

A lengthy review of the three standard methods for evaluating taste and odour with the flavour threshold test (FTT), the flavour rating scale (FRS), and the flavour profile analysis (FPA) Describes the strengths and weaknesses of each test method.

Bruvold, W. H. (undated). A Comparative Analysis of Research Methods Used to Assess Secondary Maximum Contaminant Levels for Taste and Odor in Water. Report prepared for the School of Public Health, University of California, Berkeley.

Description of standard sensory analysis methods for taste and odour as they relate to the USEPA secondary maximum contaminant levels of 15 groups of contaminants which adversely affect the aesthetic quality of drinking water.

Chastrette, M. (1981). An approach to a classification of odours using physicochemical parameters. *Chemical Senses* **6**(3): 157-163.

An investigation into the possibilities of classifying odors based on physiochemical data. The odour classifications were found to be related to the polarizability of the molecule, the oil/water partition coefficient and the acid-base properties. The shape of the molecule was not a reliable indicator.

Engen, T. (1987). Remembering odors and their names. *American Scientist* **75**: 497-502.

General, non-technical discussion of odour perceptions. Suggests a very limited association between odors and their verbal descriptors, especially when choosing between similar odour descriptors.

Fazzalari, F. A., Ed. (1978). *Compilation of Odor and Taste Threshold Values Data*. ASTM Data Series DS 48A. American Society for Testing and Materials.

Compound specific taste and odour threshold data. Listed by code number, alphabetically, by molecular weight, and by Wiswesser Line Notation permutation.

Greenberg, M. J. (1979). Dependence of odor intensity on the hydrophobic properties of molecules. A quantitative structure odor intensity relationship. *Journal of Agricultural and Food Chemistry* **27**(2): 347-352.

Early study into the relationship between molecular structure or physicochemical properties and odour intensity. Results indicate that there was a direct relationship between hydrophobicity (octanol water partition coefficient) and odour intensity, but there was no such relationship with steric or polar parameters.

Griffiths, N. M. (1974). Sensory properties of the chloro-anisoles. *Chemical Senses and Flavor* **1**: 187-195.

The strength, character and odour threshold values for a series of chloroanisoles was studied. Demonstrated a rapid odour fatigue with substitution of chlorine in both the 2- and 6- positions in the ring.

Griffiths, N. M. and Fenwick, G. R. (1977). Odour properties of chloroanisoles - Effects of replacing chloro- by methyl groups. *Chemical Senses and Flavor* **2**: 487-491.

Summary of a study which indicated that the predominant musty odour character of chloroanisoles was less apparent in the methylanisoles.

Ito, T., Okumura, T. and Yamamoto, M. (1988). The relationship between concentration and sensory properties of 2-methylisoborneol and geosmin in drinking water. Water Science and Technology **20**(8/9): 11-17.

Threshold odour and taste concentrations of geosmin and 2-methylisoborneol were found to be functions of water temperature and residual chlorine concentrations. Complaints from consumers were found to be related to geosmin or 2-methylisoborneol concentrations.

Jardine, C. G. (1992). Public evaluation of fish tainting from pulp and paper mill discharges. Water Science and Technology **25**(2): 57-64.

Sensory test results reported for a site specific evaluations of the tainting potential of pulp mill discharges. Results indicate that there was no tainting potential at one site, but there was tainting potential for one species of fish at another site.

Jardine, C. G. and Hrudey, S. E. (1988). Threshold detection values of potential fish tainting substances from oil sands wastewaters. Water Science and Technology **20**(8/9): 19-25.

Description of research into the tainting potential of twelve select compounds found in oil sands wastewater. The trained sensory panel found three compounds(naphthalene, benzothiophene, and 2,5-dimethylphenol) to have a strong capability for causing taint in fish.

Khiari, D., Brenner, L., Burlingame, G. A. and Suffet, I. H. (1992). Sensory gas chromatography for evaluation of taste and odor events in drinking water. Water Science and Technology **25**: 97-104.

Summary of the development of a sensory gas chromatography procedure to quantitatively evaluate ng/L concentrations of mixtures of odorous compounds. This method allows for the fractionation of a mixture of odors and a corresponding description and quantitation of each odour.

Krasner, S. W. (1988). Flavor-profile analysis: An objective sensory technique for the identification and treatment of off-flavors in drinking water. Water Science and Technology **20**(8/9): 31-36.

General description of the flavour profile analysis method and its use when specific causative agents had not been identified.

Krasner, S. W., McGuire, M. J. and Ferguson, V. B. (1985). Tastes and odors; the flavor profile method. Journal AWWA **77**(3): 34-39.

Basic discussion of the flavour profile analysis method.

Levi, Y., Cadet, J. L. and Coutant, J. P. (1990). Water flavor evaluation assistance automation. *AWWA WQTC Symposium Proceedings*, San Diego.

A non-standard flavour evaluation method.

Levi, Y. and Jestin, J. M. (1988). Offensive tastes and odors occurring after chlorine addition in water treatment processes. *Water Science and Technology* **20**(8/9): 269-277.

Report a new partially automated method for absolute threshold flavour determination called "up and down".

Mallevialle, J. and Suffet, I. H. (1987). *Identification and treatment of tastes and odors in drinking water*. Denver, CO, American Water Works Association Research Foundation and Lyonnaise des Eaux.

Very comprehensive summary of the study of tastes and odours in drinking water. Includes Discussion of the physiological mechanisms, identification based on consumer complaints, sources of tastes and odors, classifications, sensory and chemical analyses and treatment options.

Meng, A. K. (1991). *Correlation Analysis of Chromatographic and Sensory Profiles of Drinking Water* (Thesis). Drexel University, Philadelphia, Pennsylvania. (As referenced by Khiari, D., Brenner, L., Burlingame, G.A. and Suffet, I.H., (1992). Water Science and Technology. 25(2); 97-104.).

Detailed discussion of the statistical correlation that can be used for comparisons of flavour profile analyses and analytical results. The correlation between FPA and chemical analysis is a presumptive test that must be confirmed by testing the odour of the chemical identified by a GC detector (i.e., by olfactory GC).

Meng, A. K., Brenner, L. and Suffet, I. H. (1992). Correlation of chemical and sensory data by principal component factor analysis. *Water Science and Technology* **25**(2): 49-56.

A description of the Principal Component Factor Analysis method This procedure was used to demonstrate the correlation between sensory response and chemical concentration in water.

Meng, A. K. and Suffet, I. H. (1992). Assessing the quality of flavour profile analysis data. *Journal AWWA* **84**(6): 89-96.

Flavour profile analysis data was evaluated for the quality of the qualitative descriptions and the quantitative response for each descriptor. Qualitative descriptions are more consistent after training of panelists using the common language of an odour wheel. Quantitative responses were quite variable and the use of replicates was recommended.

Newman, A. R. (1991). Electronic Noses. *Analytical Chemistry* **63**(10): 585A-588A.

General discussion of the various instrumental methods used for the detection of odorous compounds. These include the "Warwick Nose" array of SnO2 sensors and the Naval Research Laboratory surface acoustic wave sensors for chemical warfare gases.

Parliment, T. H. and Epstein, M. F. (1973). Organoleptic properties of some alkyl-substituted alkoxy- and alkylthiopyrazines. *Journal of Agricultural and Food Chemistry* **21**(4): 714-716.

Results of a study looking at the odour variability in compounds similar to 2-methoxy- 3-isobutylpyrazine (potent bell pepper odour).

Persson, P. E. (1980). Sensory properties and analysis of two muddy odour compounds, geosmin and 2-methylisoborneol, in water and fish. *Water Research* **14**: 1113-1118.

Study of naturally produced odours and the sensory determination of their concentrations in water and fish. It is reported that the intensity of the muddy odour compounds as perceived by the panel was proportional to the logarithm of the concentrations.

Persson, P. E. (1983). Off-flavours in aquatic ecosystems - An introduction. *Water Science and Technology* **15**: 1-11.

General discussion and review of aquatic off-flavours.

Persson, P. E. (1988). Aquatic off-flavours - past, present and future. *Water Science and Technology* **20**: 283-288.

Updated general discussion of the challenges associated with the study of aquatic off- flavours.

Polak, E., Trotier, D. and Baliguet, E. (1978). Odor similarities in structurally related odorants. *Chemical Senses and Flavor* **3**(4): 369-381.

The "earthiness" quality of seven pure cyclic alcohols similar to geosmin were estimated.

Polak, E. H. and Provasi, J. (1992). Odor sensitivity to geosmin enantiomers. *Chemical Senses* **17**(1): 23-26.

The naturally occurring (-) geosmin enantiomer was found on average to have an odour threshold eleven times lower than the (+) enantiomer of geosmin.

Rigal, S. (1992). The use of organoleptic investigations to evaluate the quality of materials in contact with drinking water. *Water Science and Technology* **25**(2): 41-48.

An investigation into the odorous contributions of various materials used for plastic pipes, reservoir coatings, and various adhesives, greases and seals used in contact with drinking water.

Ruth, J. H. (1986). Odor thresholds and irritation levels of several chemical substances: A review. *American Industrial Hygiene Association Journal* **47**: A142-A151.

A collation of odour threshold data for approximately 450 chemical substances is presented along with any reported irritation threshold for humans.

Sävenhed, R., Borén, H. and Grimvall, A. (1985). Stripping analysis and chromatographic sniffing for the source identification of odorous compounds in drinking water. *Journal of Chromatography* **328**: 219-231.

A discussion of the combination of chemical and sensory methods as an improved technique for the source identification of odorous compounds.

Suffet, I. H., Brady, B. M., Bartels, J. H. M., Burlingame, G., Mallevialle, J. and Yohe, T. (1988). Development of the flavor profile analysis method into a standard method for sensory analysis of water. *Water Science and Technology* **20**(8/9): 1-9.

A discussion of the development of the "flavor wheel" for the classification and identification of odors in flavor profile analysis.

Tomita, M., Ichikawa, N. and Goda, T. (1988). Correlation between threshold odor numbers and the concentration of odorous substances in water. *Water Science and Technology* **20**(8/9): 27-30.

Summary of a study which demonstrated that there was a log-log relationship (Stevens model) between the threshold odour number value and the odorant concentration for geosmin and 2-methylisoborneol.

van Gemert, L. J. and Nettenbreijer, A. H., Ed. (1977). *Compilation of Odour Threshold Values in Air and Water*. Voorburg and Zeist, Netherlands, National Institute for Water Supply and Central Institute for Nutrition and Food Research TNO.

Compound specific odour threshold value data. Listed alphabetically in compound class groups.

Van Langenhove, H. R., Teerlinck, D., van Wassenhove, F. A. and Schamp, N. M. (1984). Sensory analysis of odorous water samples. Journal of the Water Pollution Control Federation **56**(4): 351-354.

Static and dynamic methods of sensory water analysis are compared using an olfactometer.

Veijanen, A., Paasivirta, J. and Lahtiperä, M. (1988). Structure and sensory analyses of tainting substances in Finnish freshwater environments. Water Science and Technology **20**(8/9): 43-48.

Site specific application of gas chromatography / mass spectrometry combined with sensory odour analysis methods to identify tainting substances.

Zoeteman, B. C. J. (1980). Sensory Assessment of Water Quality. Pergamon Press.

Critical review and detailed description of methods existing in 1979 that were used for sensory water quality assessment. Good introduction to the general aspects of sensory assessment of drinking water.

Zoeteman, B. C. J. and Piet, G. J. (1972/73). On the nature of odours in drinking water resources of the Netherlands. Science of the Total Environment **1**: 399-410.

First description of the "odorgram" technique also known as "GC sniffing.

6.0 Water Tainting Relating to Non-Pulp Mill Effluents

Anselme, C., N'Guyen, K., Bruchet, A. and Mallevialle, J. (1985). "Can polyethylene pipes impart odors in drinking water?" Environmental Technology Letters **6**: 477-488.

Severe odour problems caused by defective polyethylene water pipes. They found that 20% of 264 pipes tested were defective. Problems were mainly attributed to defective manufacture resulting in release of polymer additives like alkyl phenols and oxidation of pipe surfaces causing the release of polar compounds such as aldehydes and ketones.

Brownlee, B. G., Painter, D. S. and Boone, R. J. (1984). Identification of taste and odour compounds from Western Ontario. Water Pollution Research Journal of Canada **19**(1): 111-118.

Reports presence of geosmin in composite raw water and 3- methylindole, dimethyl tetrasulfide and dimethyl pentasulfide from water surrounding decomposing algal blooms.

Burttschell, R. H., Rosen, A. A., Middleton, F. M. and Ettinger, M. B. (1959). Chlorine derivatives of phenol causing taste and dour. Journal AWWA **February**: 205-214.

Early discussion of the taste and odour problems associated with phenol and chlorinated phenols. Increased chlorination is reported to intensify the taste and odour.

Cees, B., Zoeteman, J. and Piet, G. J. (1974). Cause and identification of taste and odour compounds in water. The Science of the Total Environment **3**: 103-115.

Review of odour compounds in water their sources. Two basic sources of odour substances are industrial and municipal sewage effluents or from biological activities of algae and heterotrophic micro-organisms.

Dietrich, A. M., Orr, M. P., Gallagher, D. L. and Heohn, R. C. (1992). Tastes and odours associated with chlorine dioxide. Journal AWWA **84**(6): 82-88.

Summary of survey conducted to determine practices and problems associated with the use of chlorine dioxide by U.S. water utilities. Odour complaints were more numerous and more diverse when chlorine dioxide was used, and increased numbers of complaints about odours were associated with the presence of new carpets in customer's homes.

Gerber, N. N. (1983). Volatile substances from Actinomycetes. Their role in the odor pollution of water. Water Science and Technology **15**: 115-125.

Detailed review of the odorous compounds produced by actinomycetes, including those substances responsible for earthy, muddy, woody, and potato-bin odours. The organisms which produce these "natural" odours and the specific compounds responsible are tabulated.

Hrudey, S. E., Gac, A. and Daignault, S. A. (1988). Potent odour-causing chemicals arising from drinking water disinfection. Water Science and Technology **20**(8/9): 55-62.

Reports observations of more intense odours in treated water than in the raw water which suggests the likelihood that compounds are formed during oxidative reactions resulting from disinfection processes. Four odorous, low molecular weight aldehydes were found (2-methyl propanal, 2-methyl butanal, 3-methyl butanal, and phenylacetaldehyde).

Hrudey, S. E., Rector, D. and Motkosky, N. (1992). Characterization of drinking water odour arising from spring thaw for an ice-covered upland river source. Water Science and Technology **25**(2): 65-72.

Geosmin and 2-methylisoborneol were identified by sensory and analytical means as likely major contributors to the raw water odour arising from spring thaw for an ice- covered upland river source.

Jenkins, D., Medsker, L. I. and Thomas, J. F. (1967). Odorous compounds in natural waters, Some sulfur compounds associated with blue- green algae. Environmental Science and Technology **1**: 731-735.

Identifies a number of odorous sulfur compounds produced in decaying blue-green algal cultures and reservoir waters containing blue-green algal blooms, including methylmercaptan, dimethyl sulfide, isobutyl mercaptan, and n-butyl mercaptan.

Jüttner, F. (1983). Volatile odorous excretion products of algae and their occurrence in the natural aquatic environment. Water Science and Technology **15**: 247-257.

Good summary of the various odour descriptors associated with algae and the compounds that have been found to be responsible for these odours.

Jüttner, F. (1988). Biochemistry of biogenic off-flavour compounds in surface waters. Water Science and Technology **20**(8/9): 107-116.

Lists nor-carotenoids, unsaturated hydrocarbons aldehydes, ketones, alcohols, thio compounds, terpenoids, phenols and aromatic hydrocarbons which are important biogenic odour compounds frequently found in freshwater.

Jüttner, F., Höflacher, B. and Wurster, K. (1986). Seasonal analysis of volatile organic biogenic substances (VOBS) in freshwater phytoplankton populations dominated by Dinobryon, microcystis and Aphanizomenon. Journal of Phycology **22**: 169-175.

Discusses the marked dynamics of volatile organic biogenic substances in a shallow eutrophic lake.

Kenefick, S. L., Brownlee, B., Hrudey, E. J., Gammie, L. and Hrudey, S. E. (1994). Water Taste and Odour Study (Athabasca River 1993). prepared for the Northern River Basins Study Board.

Reports FPA, CLSA results for a time of travel study for the presence of taste and odour compounds in the Athabasca River during February and March of 1993. The results indicate that the Hinton combined effluent (municipal wastewater treatment plant discharge and BKME) resulted in an impact on the odour of the Athabasca River for substantial distances downstream (>1000 km).

Lin, S. D. (1976a). Sources of tastes and odors in water. Part 1. Water and Sewage Works **123**(6): 101-104.

Discusses the "natural sources of tastes and odours in water. Natural sources listed include algae, actinomycetes, decaying vegetation, bacteria, and zooplankton. The natural presence of hydrogen sulfide and odorous metallic constituents are also summarized.

Lin, S. D. (1976b). Sources of tastes and odors in water. Part 2. Water and Sewage Works **123**: 64-67.

The anthropogenic sources of tastes and odours in water are discussed. Common components of man-made odour contributors include phenols, pesticides, alkyl benzene sulfonate (ABS), water treatment chemicals and disinfection byproducts.

Lin, S. D. (1977). Tastes and odors in water supplies: a review. Water and Sewage Works (Reference Issue), R-141-R-163.

Review of sources of tastes and odours, as well as analytical and sensory methods of detection, control options and a detailed summary of treatment and removal studies.

Persson, P. E. and Jüttner, F. (1983). Threshold odour concentrations of odorous algal metabolites occurring in lake water. Aqua Fennica **13**: 3-7.

Lists b-cyclocitral, 2,6,6-trimethylcyclo-2-en-1-one, 2,2,6-trimethylcyclohexanone, 1,3,3-trimethylcyclohexene, octa-1,3-diene, and 2-methylpent-2-enal as biogenic odour compounds.

Rizet, M. and Mouchet, J. (1982). Influence of discharges from storage reservoirs on the tastes and odours appearing in the Seine and Marne rivers. *Water Science and Technology* **14**(4/5): 43-55.

A study of the taste and odour problem that occurred in the Seine and Marne rivers during the severe drought of 1976. Geosmin and 2-methylisoborneol contributed to odour problems but were not the only explanation. The presence of organic matter seemed to be essential to the development of a moldy taste.

Rosen, A. A., Skeel, R. T. and Ettinger, M. B. (1963). Relationship of river water odor to specific organic contaminants. *Journal of the Water Pollution Control Federation* **35**(6): 777-782.

Early description of the phenomenon of odour synergism. Combinations of odorous compounds resulted in interactions between the odors of each component. The resulting odour was often greater than the sum of all components.

Slater, G. P. and Blok, V. C. (1983). Volatile compounds of the cyanophyceae - a review. *Water Science and Technology* **15**: 181-190.

Good review of odorous compounds that are produced by blue-green algae, including geosmin, 2-methylisoborneol, b-cyclocitral, hydrocarbons, aromatics, fatty acids, amines, sulfur compounds, terpenoids, and esters.

Wajon, J. E., Wajon, W. E., Alexander, R. and Kage, R. I. (1985). Dimethyl trisulphide and objectionable odours in potable water. *Chemosphere* **14**(1): 85-89.

The presence of dimethyl trisulphide at concentrations in excess of 10 ng/L was associated with an objectionable odour. The source of this compound was not established.

Weete, J. D. (1979). Streptomyces sp. A source of odorous substances in potable water. *Water, Air, and Soil Pollution.* **11**(2): 217-223.

Reports butyric acid as the principal metabolite of the actinomycete Streptomyces sp. that represents the greatest potential as an odorous water pollutant under natural conditions.

Whitfield, F. B. and Freeman, D. J. (1983). Off-flavours in crustaceans caught in Australian coastal waters. *Water Science and Technology* **15**(6/7): 85-95.

Discussion of the compounds responsible for off-flavours in prawn, lobster and crab. Includes a discussion of tainting that is a result of bacteria which may be found on these organisms.

Wnorowski, A. U. and Scott, W. E. (1992). Incidence of off-flavours in South African surface waters. Water Science and Technology **25**(2): 225-232.

Odorous substances detected in water were geosmin and dimethyl trisulphide.

Wood, S., Williams, S. T. and White, W. R. (1983). Microbes as a source of earthy flavours in potable water - a review. International Biodeterioration Bulletin **19**(3/4): 83-97.

The possible significance of various microbes, including actinomycetes, cyanobacteria and fungi, in the production of earthy tastes and odours in water is discussed.

Zoeteman, B.C.J., Kraayeveld, A.J.A. and Piet, G.J. (1971). Oil pollution and drinking water odour. H2O **4**(16): 367-373.

Lists crude oil and oil product OTC values which indicate that many petroleum products can cause odour problems at µg/L levels.

7.0 Water Tainting Relating to Pulp Mill Effluents

Bouveng, H. O. and Lundtstedt, K. (1966). Potable water from surface waters affected by pulp mill effluents. Part 1. Sulphate mill effluents. Svensk Papperstidning årg. **69**(16): 526-533.

Early study into the effects that various sulphate kraft mill effluents have on treatment parameters at water treatment facilities downstream of the discharge.

Bouveng, H. O. and Lundtstedt, K. (1968). Potable water from surface waters affected by pulp mill effluents. Part 2. Effect of sulphite spent liquor. Svensk Papperstidning årg. **71**(10): 405-410.

Early study into the effects that sulphite spent liquor effluents have on treatment parameters at water treatment facilities downstream of the discharge. It was reported that flocculation efficiency of the receiving water was less affected by kraft spent liquors than by sulphite spent liquors.

Brownlee, B. G., MacInnis, G. A. and Noton, L. R. (1993). Chlorinated Anisoles and Veratroles in a Canadian River Receiving Bleached Kraft Pulp Mill Effluent Identification, Distribution, and Olfactory Evaluation. Environmental Science and Technology **27**: 2450-2455.

Documentation of measurable concentrations of compounds attributed to bleached kraft mill effluent as far as 1100 km downstream from the effluent discharge. 2,4,6-trichloroanisole was found at levels near its OTC of 1 ng/L. 4,5-dichloroveratrole, 3,4,5-trichloroveratrole, and tetrachloroveratrole were found 2-4 orders of magnitude below their odour threshold concentrations.

Clayton, D. (1972). Water Quality Summary Athabasca River 1966-1971. Province of Alberta, Department of the Environment, Division of Standards and Approvals.

Reports that the pulp mill at Hinton has a drastic effect on the odour of the Athabasca River. Only once in the five years study period was the threshold odour number below 16 at Obed. The odour effect was still noticeable at Whitecourt but by Smith the threshold odour numbers were at or below the recommended limit of 8. Also found that the threshold odour numbers were often above 8 at Tar Island (downstream of Fort McMurray).

Cook, W. H., Farmer, F. A., Kristiansen, O. E., Reid, K., Reid, J. and Rowbottom, R. (1973). The effect of pulp and paper mill effluents on the taste and odour of the receiving water and the fish therein. Pulp and Paper Magazine of Canada, Convention Issue.

The effluent sources from a pulp mill were evaluated for their water and fish tainting potential. It was concluded that the kraft mill was the largest odour contributor and the sulphite mill was the main source of taste imparted by the mill effluent to the river water. In the fish tainting studies it was concluded that condensates (i.e. evaporator condensates, digester foul condensates, and the recovery furnace flue gas condensates) were the main sources of fish tainting materials.

Domtar Fine Papers Ltd. (1971). Effect of Pulp and Paper Mill Effluents on the Taste and Odour of Water and Fish. Pulp and Paper Pollution Abatement, Environment Canada.

Reports results of a detailed sensory evaluation of the effects of pulp and paper mill effluents on tainting of water and fish. Very basic sensory evaluations methods were used, but a thorough study of the tainting potential of all process streams was carried out.

Ferguson, R. H. (1962). Summary Report. Athabasca River, 1961-62. Province of Alberta, Department of the Environment, Division of Pollution Control.

Report increase in odour levels downstream of Hinton and a steady decrease in levels to Athabasca.

Ferguson, R. H. (1963). Summary Report. Athabasca River, 1962-63. Province of Alberta, Department of the Environment, Division of Pollution Control.

Report large increase in odour levels downstream of Hinton and then a steady decrease in levels to Athabasca.

Ferguson, R. H. (1964). Summary Report. Athabasca River, 1963-64. Province of Alberta, Department of the Environment, Division of Pollution Control.

High threshold odour numbers recorded at Obed with a maximum value of 50 detected twice. Odour levels dropped to an average of 8 by Whitecourt.

Hamilton, H. R., Turk, O. S., Sikes, J. E. G., McDonald, R. D. and Hrudey, S. E. (1987). Management of Effluents from Chemithermomechanical Pulp Mills. Alberta Environment.

Various treatment methods were reviewed for a study in the management of CTMP effluents. Anaerobic treatment methods were found to generate odorous sulfur compounds but such compounds were easily removed with a simple scrubbing unit. No threshold odour numbers were reported.

Hedberg, T., Bouveng, H. O. and Lundtstedt, K. (1969). Potable water from surface waters affected by pulp mill effluents. Part 2. Effect of sulphite spent liquor. Svensk Papperstidning årg. **72**(12): 39-41.

Early pilot scale study into the effects that pulp mill effluents have on treatment parameters at water treatment facilities downstream of the discharge. At the pilot scale, sulphite spent liquor exerts a considerably larger negative effect on water treatment efficiencies than does spent kraft liquor.

Konopasek, P. (1973). Water Quality. Athabasca River, 1971-1972. Report for Alberta Environmental Control Division. Edmonton, Alberta.

Report of water quality criteria maximum odour number violation twice during the winter of 1972 for a sampling site 117 river miles downstream of Hinton (Whitecourt).

Kovacs, T. G. and Voss, R. H. (1986). Factors influencing the effect of bleached kraft mill effluents on drinking water quality. Water Research **20**(9): 1185-1191.

The taste and odour of drinking waters contaminated with bleached kraft mill effluent were investigated. Diluted water samples were found to be impaired at effluent concentrations from 0.1 to 0.4%. No correlation was found between the original odour of the effluent and the impaired drinking water.

Kovacs, T. G., Voss, R. H. and Wong, A. (1984). Chlorinated phenolics of bleached kraft mill origin. Water Research **18**(7): 911-916.

Biologically treated effluent samples from 9 Canadian bleached softwood mills were analyzed for chlorinated phenolics content. Odour thresholds were determined for selected chlorinated phenolics and for a synthetic mixture of these compounds. Results indicate that chlorinated phenolics present in effluent discharged from bleached kraft mills are not expected to contribute an off-odour to recipient waters.

Kringstad, K. P. and Lindström, K. (1984). Spent liquors from pulp bleaching. Environmental Science and Technology **18**(8): 236A-248A.

Review of the chemical composition of spent liquors from pulp bleaching. Includes a useful discussion of the formation of various classes of compounds during pulp bleaching and lists many of the compounds commonly found.

McLeay, D. and Associates Ltd. (1987). Aquatic Toxicity of Pulp and Paper Mill Effluent; A Review. Environment Canada.

Includes lengthy review of off flavours resulting from pulp mill effluents. Mainly discusses fish tainting propensity of these effluents.

NCASI (1973). The Measurement of Phenolic Substances and the Significance of Their Presence in Pulp Mill Effluents on Taste and Odor in Receiving Waters. Stream Improvement Technical Bulletin No. 268. National Council of the Paper Industry for Air and Stream Improvement, Inc., New York, N.Y.

A study into the significance of phenolic substances in pulp mill effluents. It was found that such chlorinated phenolics were not the cause of taste and odour in pulp mill effluents.

Noton, L. R. (1989). The Peace and Athabasca River Systems. A Synopsis of Alberta Environment's Monitoring Programs and the Water Quality Effects of Existing Pulp Mill Effluents. Environmental Quality Monitoring Branch, Alberta Environment.

Report of odour component of water quality study for the Wapiti-Smoky River system indicate a noticeable increase in odour due to kraft mill effluents during low flows. The odour was reported to persist under ice the length of the Smoky River. It was also found that odour persisted at least 500 km in the Athabasca River.

Noton, L. R. (1992a). Water Quality in the Wapiti-Smoky River System Under Low-Flow Conditions 1987- 1991. A Synopsis of Government Surveys and Monitoring. Environmental Quality Monitoring Branch, Alberta Environment.

Reported a large increase in odour of Wapiti River water due to pulp mill effluent during the fall and winter of 1987-1991. In the Smoky River the "pulp" mill odour persisted in the river to the mouth, during the fall and winter of 1987-1991.

Noton, L. R. (1992b). Water Quality in the Wapiti-Smoky River System Under Low-Flow Conditions 1987- 1991. A Synopsis of Government Surveys and Monitoring. Supplement. Environmental Quality Monitoring Branch, Alberta Environment.

Includes a summary of the field data for evaluations of odour during river surveys of the Wapiti-Smoky River system for 1989-1991.

Noton, L. R., Anderson, A. M., Reynoldson, T. B. and Kostler, J. (1989). Water Quality in the Wapiti-Smoky River System Downstream of the Procter and Gamble Pulp Mill, 1983. Environmental Quality Monitoring Branch, Alberta Environment.

Reported only one instance (May) of a measured odour exceeding background levels in the river water during the 1983 sampling. However, an odour characteristic of pulp mills was detectable during sampling in more instances.

Noton, L. R. and Shaw, R. D. (1989). Winter Water Quality in the Athabasca River System 1988 and 1989. Environmental Quality Monitoring Branch, Alberta Environment, August.

Report of field evaluations of odour with respect to intensity and nature of odour. Directly downstream of Hinton a "pulp mill-like" odour was always present. In 1988 odour in the river water persisted to the vicinity of Smith, while in 1989, the odour persisted to the lower-most reaches of the Athabasca River. Also reported that none of the other individual effluents on the River appeared to cause any notable or consistent increase in river odour.

Paasivirta, J., Knuutinen, J., Tarhanen, J., Kuokkanen, T., Surma-Aho, K., Paukku, R., Kääriäinen, H., Lahtiperä, M. and Veijanen, A. (1983). Potential off-flavour compounds from chlorobleaching of pulp and chlorodisinfection of water. Water Science and Technology **15**: 97-104.

Persistent chlorinated acids, phenols and neutral compounds were attributed to be possible tainting compounds downstream of pulp bleaching and chlorodisinfection plants. Microbial metabolism of chlorophenols resulted in the formation of chloroanisoles which are even more likely to cause tainting.

Province of Alberta (1967). Summary Report. Athabasca River Pollution Survey, Winter 1966-67. Province of Alberta, Department of Health, Environmental Health Services Division.

Threshold odour numbers increased from 1 upstream of Hinton to values ranging from 16 to 64 at Obed. This "wood resin" type of odour prevailed downstream until Tar Island, although values were 8 or lower at locations downstream from Whitecourt.

Province of Alberta (1968). Summary Report. Athabasca River Pollution Survey, 1967-68. Province of Alberta, Department of Health, Environmental Health Services Division.

Report large increase in "wood, resin" type odour levels downstream of Hinton and then gradual decreases until Fort McMurray. Below Fort McMurray , a chemical type of odour was found with threshold odour levels as high as 16.

Province of Alberta (1969). Summary Report. Athabasca River Pollution Survey, Winter 1968-69. Province of Alberta, Department of Health, Environmental Health Services Division.

Threshold odour number of 100 reported at Obed on November 1968. This "wood resin" type of odour prevailed farther downstream for sample sets collected during the winter months.

Province of Alberta (1970). Summary Report. Athabasca River Pollution Survey, Winter 1969-70. Province of Alberta, Department of Health, Environmental Health Services Division.

Threshold odour numbers ranging from 16 to 32 reported at Obed. This "wood resin" type of odour prevailed farther downstream for sample sets collected during the winter months. One sample taken at Fort McMurray had a reported odour threshold value of 64 with a hydrocarbon odour.

Shaw, R. D. and Noton, L. R. (1989). A Preliminary Assessment of the Impact of Existing Pulp Mills on the Peace River. Environmental Quality Monitoring Branch, Alberta Environment.

In 1988 there were no reported adverse odours due to the four mills in the Peace River Basin. However, in previous years, odour problems during winter in the Peace River near the Town of Peace River were attributed to effluent from the P&G mill at Grande Prairie.

Simpson, K. J. (1971). Pollution Survey Summary. Athabasca River, 1970-71. Province of Alberta, Department of the Environment, Division of Pollution Control.

Above Hinton threshold odour numbers were minimal and generally had a "wood-resin" or "musty" type of odour. At the next sample site (Whitecourt), values ranged from 4 to 16 and at Fort McMurray, "chemical" and "hydrocarbon" type odours were often recorded.

Wigilius, B., Borén, H., Grimvall, A., Carlberg, G. E., Hagen, I. and Brögger, A. (1988). Impact of bleached kraft mill effluents on drinking water quality. Science of the Total Environment **74**: 75-96.

Chemical and bioassay study of the long-range transport of chloro-organic substances due to bleached kraft effluent discharges.
