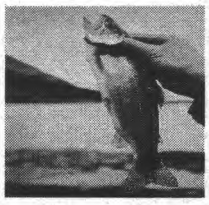
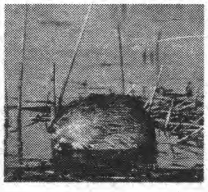
Canada Alberta Northwest Territories

Northern River Basins Study



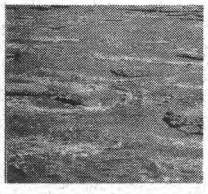


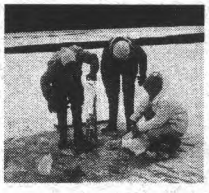





NORTHERN RIVER BASINS STUDY PROJECT REPORT NO. 42

WATER ODOUR
ATHABASCA RIVER
FEBRUARY AND MARCH, 1993



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by

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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.

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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,
IT IS THEREFORE REQUESTED BY THE STUDY OFFICE THAT;
this publication be subjected to proper and responsible review and be considered for release to the public.

(Dr. F.J. Wrona, Ph.D., Science Director) — (Date) 15 Dec 94

Whereas it is an explicit term of reference of the Science Advisory Committee "to review, for scientific content, material for publication by the Board",
IT IS HERE ADVISED BY THE SCIENCE ADVISORY COMMITTEE THAT;
this publication has been reviewed for scientific content and that the scientific practices represented in the report are acceptable given the specific purposes of the project and subject to the field conditions encountered.
SUPPLEMENTAL COMMENTARY HAS BEEN ADDED TO THIS PUBLICATION: [✓] Yes [ ] No

(Dr. P. A. Larkin, Ph.D., Chair) — (Date) Dec 15/94

Whereas the Study Board is satisfied that this publication has been reviewed for scientific content and for immediate health implications,
IT IS HERE APPROVED BY THE BOARD OF DIRECTORS THAT;
this publication be released to the public, and that this publication be designated for: **[ ] STANDARD AVAILABILITY [ ] EXPANDED AVAILABILITY**

(Lucille Partington, Co-chair) — (Date) Dec. 15/94

(Robert McLeod, Co-chair) — (Date) Jan. 03/94

WATER ODOUR, ATHABASCA RIVER, 1993

STUDY PERSPECTIVE

Public concerns have been raised that pulp mill effluent and other effluent sources are making drinking water odorous and bad tasting. A study was undertaken to analyze the water in the Athabasca River from upstream of Hinton to Fort Chipewyan, a distance of 1200 kilometres. The water at 30 sites was tested for odours by two independent flavour panels in addition to being analyzed by gas chromatography using both sensory and chemical specific detection methods. The purpose of this study was to give a preliminary indication of the extent of water odour problems in the Athabasca River and also to identify the chemical compounds likely to cause these problems.

The Northern River Basins Science Advisory Committee noted that although anisoles and veratroles, substances commonly associated with taste and odour problems, were undetected in the effluent, they may form from precursors such as guaicols which were neither tested in the effluent nor river samples. The possiblity exists that should these precoursers exist, odours could appear as latent effects of biological processes active in the aquatic ecosystem. The results of the next study (4413-C1) on off-flavour tainting should shed some light on this possibility.

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?

At Hinton the effluent of the pulp mill and town are combined and treated. This effluent was identified by all detection methods as having a significant impact on raw water odour as far downstream as Ft. McKay (1000 Km downstream of Hinton). The communities potentially affected include: Whitecourt, Athabasca, Ft. McMurray, Ft. Chipewyan.

Tributaries were found to have an insignificant effect on raw water odour. Future water odour studies were recommended to concentrate on the Athabasca River mainstem. The report concluded that flavour panels were the most appropriate way to monitor for the presence of odour-causing compounds. Chemical specific gas chromatography, although accurate, is limited in its application to only a few specific compounds.

This study provided an opportunity to compare and evaluate different odour detection methods. Most importantly, it provided baseline information on the odour characteristics of water in the Athabasca River prior to the Alberta-Pacific Pulp Mill coming on stream. A follow-up study in 1993/94 is to provide additional information on the effect of the new mill on off-flavour tainting of water.

REPORT SUMMARY

The purpose of this study was to determine the potential for off-flavour tainting of water and/or fish in the Athabasca River by compounds discharged by bleached kraft and chemi-thermomechanical pulp mills. The opportunity to determine in-stream occurrence of common tainting compounds prior to the startup of the Alberta-Pacific mill was exploited. This study combined three different analytical methods commonly used in monitoring for the presence of odorous compounds in water supplies. Two trained flavour profile panels were used to characterize the odour of the samples, two trained analysts evaluated the samples using olfactory GC and all samples were quantitatively analyzed for the presence of target odour compounds using GC/MS. These three techniques all provide quite different information and all have certain limitations.

The flavour profile panel work, involving two independent panels, clearly indicated an impact of Hinton combined effluent on the odour of the Athabasca River for substantial distances downstream. The odour contributions to the Athabasca River from tributaries were minor. The odour contributions from other effluent sources (sewage treatment plants and chemi-thermomechanical pulp mills) were less distinctive than the Hinton combined effluent and their role in affecting downstream odour is not as clear. Notwithstanding these observations, the observed impacts on raw water odour could not be detected in the treated drinking water at Ft. McMurray, possibly because of removal of odorous compounds in treatment and / or masking of the raw water odour with chlorinous odours. The raw water supply at Ft. Chipeweyan was not particularly odorous and the finished water exhibited a very strong chlorine odour that would have masked any subtle odours present.

The CLSA-GC/MS for target compounds also suggests that there was limited contribution of odour compounds from the tributaries. None of the effluent samples, including the Hinton combined effluent, contributed substantial concentrations of the target odour compounds to the Athabasca River, with the possible exception of geosmin. Notwithstanding these findings, there was a very distinctive rise in 3,4,5-trichloroveratrole and a measurable, but less distinctive rise in 2,4,6-trichloroanisole in the Athabasca River downstream of Hinton. Because neither of these compounds were present in substantial concentration in the Hinton combined effluent, and their concentrations increased downstream of Hinton, there is not a simple explanation for a possible role of this effluent source in the observed Athabasca River system concentrations for these compounds. In any case, none of the target odour compounds, by themselves would explain the odour character that was perceived by the flavour panel in the Hinton combined effluent and affected downstream samples. The OGC should have provided a separate approach to account for non-target odorous compounds that might explain the odours perceived by the flavour panel. However, there were very few extra odour peaks detected by OGC with perhaps only a sulfury / septic odour and a sulfury/mercaptan/crude oil odour that were likely to have contributed in any substantial way to the pulp mill odour character. Identifying these compounds would likely assist the odour characterization process, but there are likely other contributing compounds that have not yet been detected by the methods employed in this survey. This possibility suggests the need for a better characterization of the compounds that are primarily responsible for creating the odour of pulp mill effluents.

ACKNOWLEDGMENTS

The authors gratefully acknowledge the technical assistance provided by Terrina Perley for the closed-loop stripping extractions and Gordia MacInnis for large volume extractions. We also thank the members of the E.L. Smith flavour profile analysis panel; David Rector, Bob Andrews, Marguerite Ferguson, and Pat Melnychuk and the members of the University of Alberta flavour profile analysis panel; Nola Low, Mary Tweedie, Dianne Sergy, Margaret Wanke, and Veronica Clough. We thank Leigh Noton of Alberta Environment for his advice and assistance and the staff of the Alberta Environment Millwoods Laboratory for collection of the samples.

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1.0 INTRODUCTION

1.1 OBJECTIVE

The purpose of this study was to determine the potential for off-flavour tainting of water and/or fish in the Athabasca River by compounds discharged by bleached kraft and chemi-thermomechanical pulp mills. The opportunity to determine in-stream occurrence of common tainting compounds prior to the startup of the Alberta-Pacific mill was exploited. The project involved chemical analyses including gas chromatography / mass spectrometry (GC/MS), gas chromatography / flame ionization detection (GC/FID) and olfactory gas chromatography (OGC), as well as flavour profile panel analyses by both an experienced sensory panel and a newly trained sensory panel. These analyses were carried out on water and effluent samples collected at the time-of-travel of the Athabasca River and its tributaries in February and March of 1993.

1.2 BACKGROUND

Bleached kraft mill effluent has been reported to impair the taste and odour of drinking water at effluent concentrations ranging from 0.1 to 0.4% (Kovacs and Voss, 1986; Paasivirta et al., 1983; Wong, et al., 1985). Pulp mill effluent contributions to river water levels of phenols and trichlorophenol are also important because of the recent Swedish reports of bio-methylation of trichlorophenol to 2,4,6-trichloroanisole, an extremely potent source of musty odours in water and fish (Nyström et al., 1992). A preliminary survey (Brownlee et al., 1992) of the Athabasca River from upstream of Hinton to 120 km downstream of Ft. McMurray has found evidence of odour compounds arising immediately downstream of Hinton that persist as far downstream as Ft. McMurray.

Fish tainting by bleached kraft mill effluents has been reported in a variety of studies (Cook et al., 1973; Farmer et al., 1972; Gordon et al., 1980, Government of Canada, 1991) with tainting observed at effluent concentrations as low as 0.5%. Unbleached kraft mill effluents have also been implicated in fish tainting episodes (Hall, personal communication, 1992). Chemi-thermomechanical pulp mill effluents have received little attention with regard to fish or water off-flavour tainting.

Given the potential contributions of off-flavour compounds from various industrial and natural sources and the relative contribution of effluents during low winter flow, there was a need to evaluate the occurrence and significance of off-flavour compounds in the northern river systems. In the case of the Athabasca River, it was particularly important to obtain baseline information on the presence of off-flavour compounds before the Alberta-Pacific pulp mill came on stream.

The initial phase of this study was to be undertaken in the winter of 1993 during the months of February and March. During this sampling period the Athabasca River and the tributaries sampled were ice-covered and were at low flows (see Table 1). Because many odorous compounds are problematic at ng/L concentrations and are often quite volatile, this sampling period was purposely chosen to allow collection of river water when dilution of effluents is usually lowest and odour compound persistence is greatest.

The Northern River Basins Study Board co-chair, Mr Bev Burns, has identified a need "to reconcile science with societal values and concerns". In the case of sensory evaluations, the different values held by the scientific community and the general population were recognized as significant. This study used two sensory evaluation panels; one made up of a cross-section of the general population with adequate sensory capabilities but limited formal training, and the other made up of people trained by the water treatment plant industry and experienced in scientific evaluations. Because the U of A panel was newly established it was considered prudent to have the samples evaluated by an established panel as well, to provide a cross check.

2.0 METHODS

The water and effluent samples were collected by Alberta Environment on behalf of the Northern River Basins Study. A total of 13 Athabasca River water samples were collected between the first sampling point at Highway 40 and the final sampling point at Lake Athabasca near Fort Chipewyan. Effluent samples were collected from Hinton (bleached kraft pulp mill and municipal combined), Alberta Newsprint, Millar Western, Whitecourt Waste Water Treatment Plant, Slave Lake Pulp, Athabasca Waste Water Treatment Plant, Fort McMurray Waste Water Treatment Plant, and Suncor. Raw and finished water samples were collected from the Fort McMurray Water Treatment Plant and finished water was collected from the Fort Chipewyan Water Treatment Plant. In addition, 5 samples were collected from tributaries that feed the Athabasca River. All water and effluent samples were collected at the time-of-travel of the Athabasca River at sampling sites shown in Figure 1 and on the sampling dates listed in Table 1.

All samples were collected without headspace or preservation and were cooled until delivery to the laboratory at the University of Alberta. All samples were delivered within 24 hours of collection. A 8L water sample was collected at each sampling point and 2L effluent samples were collected and diluted 20:1 with de-ionized, odour-free water at the laboratory.

2.1 FLAVOUR PROFILE ANALYSIS

Subsamples (4L) of each water and diluted effluent were filter sterilized using pressure filtration and a pre-combusted glass-fibre filter combined with a membrane filter (0.2µm Millipore GSWP), prior to distribution to both sensory evaluation panels. Samples were subjected to flavour profile analysis by the experienced sensory panel under the direction of Dr. Les Gammie, Director of Applied Water Treatment Research for the City of Edmonton Water Branch, as well as a newly trained panel established at the University of Alberta. 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 method #2170 (APHA-AWWA-WEF, 1992). The panels used in this study were generally run in accordance with this method .

Each panel consisted of three to eight panelists trained to examine the sensory characteristics of the water and diluted effluent samples. A 100mL sample for each panel member was transferred to an odour-free 250mL Erlenmeyer flask with a ground glass stopper. The samples were brought to room temperature by heating in a 45°C water bath for 15 minutes. The heated sample was then gently swirled in a circular manner to ensure that volatile compounds were released into the headspace. The flask was brought to the nose, the stopper removed and the odour attributes were determined by sniffing at the flask opening. The odour attributes and intensities were recorded in the order perceived. The intensities were based on a multiple point scale (0=no odour, tr=trace or threshold, 1=weak, 2=moderate, 3=strong). Half units were also used by panelists to further distinguish intensities. The panelist met in a clean odour-free room and were not allowed to smoke, eat or drink for 30 minutes prior to the testing.

The panel established at the University of Alberta was selected by screening 12 potential panelists for anosmia using the scratch and sniff smell identification test produced by Sensonics Inc. (Haddon Heights, N.J.) Of the screened group, 8 suitable potential panelists were selected and further evaluated for their discrimination of the primary taste categories by means of testing solutions of sucrose (sweet), citric acid (sour), quinine (bitter) and NaCl (salty) together with distilled water. The final University of Alberta panel for any test session consisted of between 3 and 8 panelists.

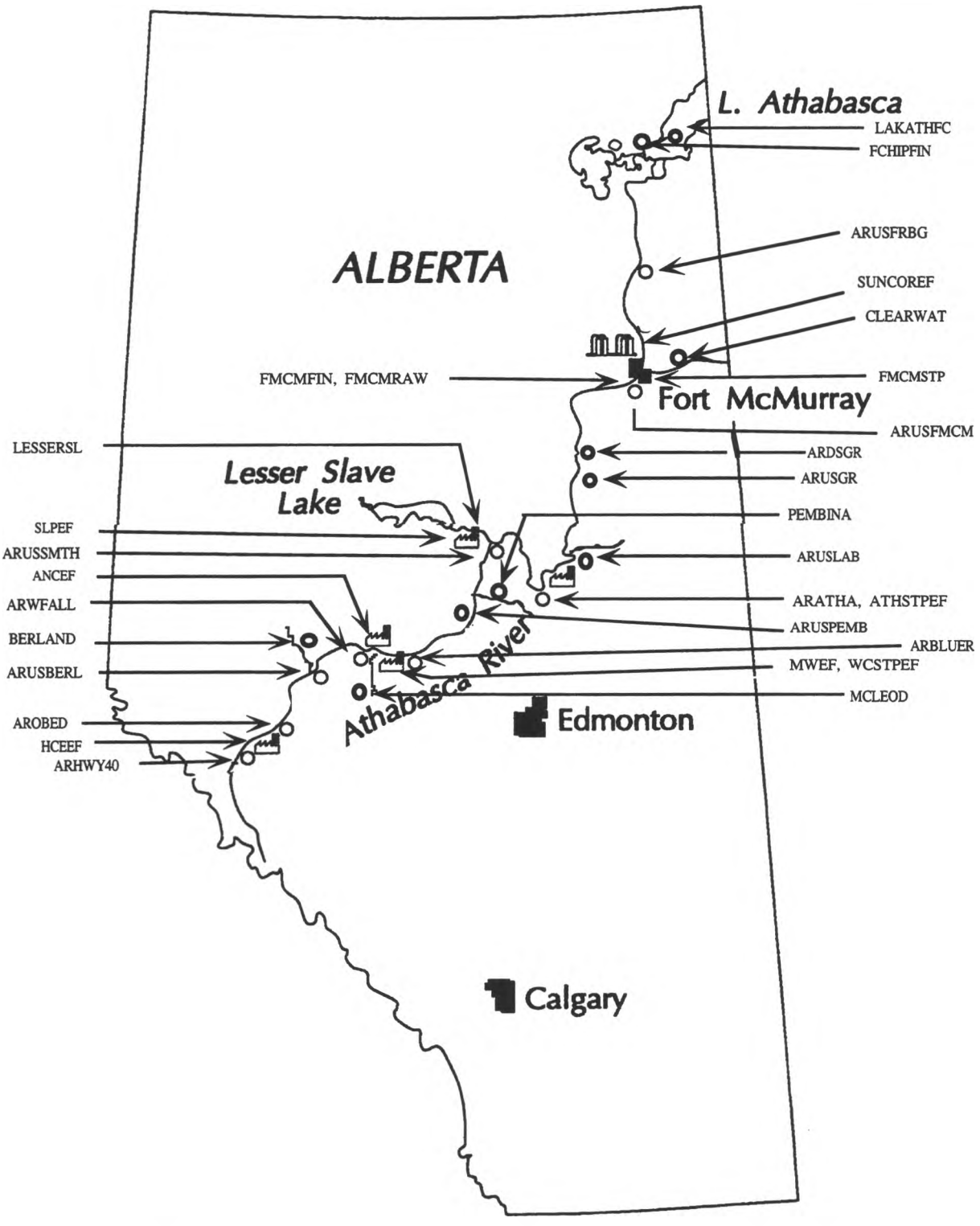

Figure 1. SAMPLING SITE MAP

Table 1.

TASTE AND ODOUR WATER SAMPLING SCHEDULE AND FLOW DATA
Athabasca River 1993

Time-of-travel (days)	Sampling Date	Site	Sample Code	Flow m3/s
1	Feb.11	ENTRANCE (Double Set)	ARHWY40	28.7[a]
1	11	Hinton Combined Effluent	HCEEF	1.2[a]
1	11	OBED	AROBED	29.9[b]
3	12	u/s BERLAND R.	ARUSBERL	38.7[b]
3	12	Berland R.	BERLAND	7.8[a]
5	16	WINDFALL	ARWFALL	43.6[a]
6	17	McLeod R.	MCLEOD	5.1[a]
6	17	Alberta Newsprint Effluent	ANCEF	0.2[a]
6	17	Millar-Western Effluent	MWEF	0.1[a]
6	17	Whitecourt STP Effluent	WCSTPEF	0.04[a]
6	17	BLUE RIDGE	ARBLUER	49.6[b]
12	23	u/s PEMBINA R.	ARUSPEMB	44.6[b]
12	23	Pembina R.	PEMBINA	1.6[a]
14	24	u/s SMITH @ HWY 2	ARUSSMTH	46.2[b]
14	24	Lesser Slave R.	LESSERSL	13.0[a]
14	24	Slave Lake Pulp Effluent	SLPEF	0.05[a]
18	26	ATHABASCA	ARATHA	62.0[a]
18	26	Athabasca STP Effluent	ATHSTPEF	0.01[a]
22	Mar.04	u/s LA BICHE R.	ARUSLAB	63.8[b]
25	09	u/s GRAND RAPIDS	ARUSGR	71.5[b]
26	09	d/s GRAND RAPIDS	ARDSGR	73.3[b]
29	10	u/s FORT McMURRAY	ARUSFMCM	65.1[a]
29	10	Fort McMurray Raw Water	FMCMRAW	-
29	10	Fort McMurray Finished Water	FMCMFIN	-
29	10	Fort McMurray STP Effluent	FMCMSTP	0.2[a]
29	10	Clearwater R.	CLEARWAT	41.7[a]
29	11	Suncor Effluent	SUNCOREF	0.3[a]
32	16	u/s FIREBAG R.	ARUSFRBG	132.0[b]
	17	L. ATHABASCA OFF FORT CHIP	LAKATHFC	138.6[b]
	17	Fort Chip Finished Water	FCHIPFIN	-

[a] Measured Flow as per Alberta Environment
[b] Estimated Flow as per Alberta Environment

Because of the short lead time available for implementing this study, the University of Alberta panel experienced limited training, involving exposure to the target odour compounds, before facing the survey samples. In the case of the City of Edmonton panel, it had been operational for over 24 months and was very experienced in judging odours commonly found in the river systems. The panels were presented with all samples coded by simple numbers that precluded identification of sample source. Hence the sensory reactions obtained were free of any expectations beyond the general knowledge that the survey was directed towards the determining odours in the Athabasca River system. Once, the septic / pulp and paper character of the Hinton combined effluent and downstream Athabasca River samples was detected and recognized by the panelists, this descriptor was applied to other downstream samples when panelists recognized the same character.

2.2 CLOSED-LOOP STRIPPING - GC / MS ANALYSIS

One litre subsamples of each sample collected were analyzed, in quadruplicate, as received. All effluent samples were diluted 20:1 with odour-free water prior to extraction. A known mass of chlorodecane was added to each 1L sample, as a recovery standard, prior to close-loop stripping analysis (CLSA). The samples were closed loop stripped onto 1.5 mg carbon filters for 2 hours (water bath temperature at 30°C and filter temperature at 50°C) using a Brechbühler AG closed loop stripping apparatus. The filters were then removed and extracted twice with 10 µL and once with 5 µL of carbon disulfide : acetone (9:1). A known mass of biphenyl-d10 was added to the extract as an injection standard. A 2 µL sample was analyzed by GC-MS using injection in the splitless mode and 10µL of doubly distilled isooctane was added prior to subsequent analyses by OGC and GC/FID. The GC-MS analysis was carried out in the selected ion monitoring (SIM) mode on an HP 5890 GC with an HP 5970 mass-selective detector and an HP 59940 Chemstation® data system. The GC-MS conditions were: 80°C to 180°C at 4°C/min and then 10°C/min to 280°C; DB-1301 column, 30m x 0.25mm ID, 0.25 µm film thickness; column head pressure 100 kPa; injector temp 250°C; detector temp 300° C. Peaks of three characteristic ions for each target compound and internal standard were monitored and the presence of a compound in an extract was confirmed if the peaks of the three ions all maximized at the same retention time and had standard intensity ratios within 20% of those of the calibration standards. Characteristic ions, their typical relative intensities and their retention times are shown in Table 2. Target compounds were chosen on the basis of known odour compounds. Many of the odour compounds in bleached kraft mill effluent were, and still remain, unidentified. This is recognized as a definite weakness in this field of study, but resolution of this problem was certainly beyond the scope and budget of this project.

The quantitation of each compound was based on the peak areas of specific quantitation ions. The internal standard 1-chlorodecane was used to calculate a response factor for each target compound from CLSA of a series of concentrations of calibration standard mixtures. The response factors are listed in Table 3. This method of quantitation results in reporting a final value which has been adjusted for recovery and therefore represents the quantity in the water sample. The biphenyl-d10 standard was used to calculate the recovery of 1-chlorodecane in each CLSA extraction. The detection limits for the CLSA extraction combined with GC/MS analysis for the target compounds are also listed in Table 3. The CLSA-GC/MS detection limits are affected by stripping efficiencies and the condition of the GC/MS. The detection limit for 2-methylisoborneol is noticeably higher than the limits for other target compounds. The loss in sensitivity was found to be related to limitations in the stripping efficiency of MIB for the standardized filters that were used. Method blanks, filter blanks and controls with known standard concentrations were also extracted using the CLSA and analyzed using GC/MS during the course of the project.

Table 2:
GC/MS DATA FOR TARGET ODOUR COMPOUNDS AND INTERNAL STANDARDS

Compound	Retention Time (min)	Quantification Mass (amu) intensities)	Characteristic Ions (with relative
1-chlorodecane	9.5	91	43(100), 91(87), 93(27)
biphenyl-d10	13.2	164	164(100), 162(38), 160(23)
2-isopropyl-3-methoxy pyrazine	5.3	137	137(100), 152(28), 24(24)
2-isobutyl-3-methoxy pyrazine	7.2	124	124(100), 95(27), 151(19)
2-methylisoborneol	7.9	95	95(100), 135(9), 168(3)
2,4,6-trichloroanisole	11.6	195	195(100), 212(88), 210(85)
2,3,6-trichloroanisole	13.1	210	210(100), 212(99), 195(43)
geosmin	14.0	112	112(100), 125(15), 182(4)
3,4,5-trichloroveratrole	21.6	240	240(100), 242(97), 225(92)

Table 3:
GC/MS RESPONSE FACTORS FOR TARGET ODOUR COMPOUNDS RELATIVE TO INTERNAL STANDARDS

Compound	*Recovery Factor Relative to 1-chlorodecane	**Response Factor Relative to Biphenyl-d10	Detection Limit (ng/L)
1-chlorodecane	-	0.293	-
2-isopropyl-3-methoxy pyrazine	0.190	0.600	0.9
2-isobutyl-3-methoxy pyrazine	0.530	0.425	1.0
2-methylisoborneol	0.080	0.357	10
2,4,6-trichloroanisole	2.12	0.196	0.10
2,3,6-trichloroanisole	1.92	0.150	1.0
geosmin	0.900	0.352	0.24
3,4,5-trichloroveratrole	0.340	0.106	1.2

* based on 24 CLSA standard extractions
** based on 12 GC/MS injections

2.3 OLFACTORY GAS CHROMATOGRAPHIC ANALYSIS

The CLSA extracts were also analyzed by olfactory gas chromatography (OGC) using a Hewlett Packard 5890 Gas Chromatograph where the GC conditions were: 80°C to 280°C at 4°C/min; DB-1301 column, 30m x 0.32mm ID, 0.25 μm film thickness; column head pressure 70 kPa; injector temp 250°C. For OGC, the column was raised through a heated transfer unit from the GC oven to a glass detection cone (Olfactory detection outlet kit, SGE International). Air drawn by a venturi from the GC oven is used to heat the transfer zone and the glass cone is purged with air that has been humidified by passing it through a reservoir containing Milli-Q water. After a 2μL sample was injected and the solvent peak had eluted, the effluent was continually monitored by time. Elution time, intensity and odour descriptor were recorded. A six point intensity scale was used (*=very weak, to ******=very strong). Control samples of known standard concentrations were used to help establish consistency of descriptors and intensity values. Method blanks and filter blanks were also checked using the OGC analytical method. The CLSA extracts were also analyzed using the GC and column described above, but with the column connected to a flame ionization detector (FID) at a detector temperature of 280°C.

3.0 RESULTS and DISCUSSION

3.1 CLOSED-LOOP STRIPPING - GC / MS

The results of the analyses of the CLSA extracts and the corresponding chlorodecane recoveries are shown in Table 4. All CLSA extractions were carried out in quadruplicate because of the inherent variability of stripping efficiencies of some compounds. The recovery of the chlorodecane in each extraction can be used as an indicator of overall recoveries and also highlights the extracts which are questionable. Any extracts with chlorodecane recoveries less than 10% were not reported. It must be emphasized that these results were obtained using GC/MS in selected ion monitoring mode in order to increases sensitivity. By doing so the analysis is limited to monitoring for chosen target compounds. However, many of the odour compounds in bleached kraft mill effluent were, and still remain, unidentified. This is recognized as a definite weakness in this field of study, but resolution of this problem was certainly beyond the scope and budget of this project.

The CLSA analytical results demonstrate a number of basic trends. The mainstem Athabasca River sample upstream of Hinton (ARHWY40) did not contain detectable levels of any of the target odour compounds. Downstream of Hinton after the combined effluent (bleached kraft pulp mill and municipal) there was consistent detection of 3,4,5-TCV in mainstem samples (including Fort McMurray raw and finished waters) all the way to the Firebag River (Figure 2). Geosmin and 2,4,6-TCA were also detected in most mainstem samples downstream of Hinton (including Fort McMurray raw and finished waters). The tributary samples (Berland, McLeod, Pembina, Lesser Slave, Clearwater) generally contained fewer odor compounds than the Athabasca River mainstem samples and in most of the tributaries only geosmin was detected (Figure 3). The diluted effluent samples contained low or non detectable levels of most of the target odour compounds.

Non-target, total ion chromatographs (TIC) were also obtained for each CLSA extract. A TIC of the target compounds is shown for comparative purposes in Figure 4. TICs for selected samples (ARHWY40, AROBED and ARUSFRBG) indicate that significantly higher levels of the non-target compounds are seen immediately downstream of Hinton (Figure 6) as compared to the levels found upstream (Figure 5). The levels of non-target compounds had diminished again by the time of the sample collection upstream of the Firebag River (Figure 7). The same trends are apparent in the FID chromatographs shown in Figure 8. Some of the non-target compounds were easily identifiable from computer aided mass spectral searches. Those with match probabilities greater that 90% are labeled in figures 5 to 7. Mass spectral interpretation from first principles for the remaining peaks was beyond the scope of the project.

Table 4:
CLOSED LOOP STRIPPING GC/MS RESULTS

Sample ID	IPMP ng/L	IBMP ng/L	MIB ng/L	246TCA ng/L	236TCA ng/L	Geosmin ng/L	345TCV ng/L	Chlorodecane Recovered
ARHWY401	ND	ND	ND	ND	ND	ND	ND	27.5%
ARHWY403	ND	ND	ND	ND	ND	ND	ND	56.5%
ARHWY404	ND	ND	ND	ND	ND	ND	ND	57.0%
HCEEF2*	ND	ND	ND	0.3	ND	ND	1.3	25.7%
HCEEF4*	1.1	ND	ND	0.1	ND	ND	1.4	45.3%
AROBED1	3.8	ND	ND	0.3	ND	0.2	4.9	74.0%
AROBED2	1.3	ND	ND	0.8	ND	ND	3.5	41.5%
AROBED4	3.8	ND	ND	1.2	ND	ND	3.7	49.6%
ARUSBERL1	ND	ND	ND	2.0	ND	1.0	15.8	19.5%
ARUSBERL2	1.7	ND	ND	1.4	ND	0.6	21.0	45.7%
ARUSBERL3	ND	ND	ND	2.2	ND	1.0	21.7	56.2%
ARUSBERL4	2.5	ND	ND	1.6	ND	1.1	22.6	47.1%
BERLAND1†	ND	ND	ND	ND	ND	0.3	ND	31.8%
BERLAND2†	ND	ND	ND	ND	ND	ND	ND	52.1%
BERLAND3†	ND	ND	ND	ND	ND	0.4	ND	43.2%
BERLAND4†	ND	ND	ND	ND	ND	ND	ND	40.2%
ARWFALL1	ND	ND	ND	3.1	ND	1.6	54.6	39.8%
ARWFALL2	4.2	ND	ND	3.4	ND	2.0	49.5	18.6%
ARWFALL3	ND	ND	ND	7.0	ND	3.9	54.3	27.0%
ARWFALL4	ND	ND	ND	7.6	ND	3.0	39.4	15.0%
MCLEOD1†	ND	ND	ND	ND	ND	1.5	ND	40.2%
MCLEOD2†	ND	ND	ND	ND	ND	1.5	ND	31.4%
MCLEOD3†	1.3	ND	ND	0.3	ND	1.7	ND	31.5%
MCLEOD4†	ND	ND	ND	0.4	ND	1.9	ND	29.7%
ANCEF2*	ND	ND	ND	ND	ND	1.3	2.0	34.2%
ANCEF4*	ND	ND	ND	ND	ND	1.7	2.1	42.8%
MWEF1*	ND	ND	ND	ND	ND	5.5	ND	36.9%
MWEF3*	ND	ND	ND	ND	ND	5.7	5.0	36.7%
WCSTPEF2*	ND	ND	ND	ND	ND	2.0	ND	40.5%
WCSTPEF4*	ND	ND	ND	ND	ND	2.2	ND	34.6%
ARBLUER1	0.9	ND	ND	3.5	ND	2.0	37.3	43.2%
ARBLUER2	ND	ND	ND	2.4	ND	2.1	25.9	27.3%
ARBLUER3	0.9	ND	ND	3.6	ND	2.3	44.5	41.2%
ARBLUER4	2.5	ND	ND	2.0	ND	2.1	24.9	51.2%

* Effluent sample diluted 20:1 (actual concentrations are 20 times greater)
† Tributary sample

continued...

Sample ID	IPMP ng/L	IBMP ng/L	MIB ng/L	246TCA ng/L	236TCA ng/L	Geosmin ng/L	345TCV ng/L	Chlorodecane Recovered
ARUSPEMB1	ND	ND	ND	3.0	ND	4.8	17.0	36.4%
ARUSPEMB2	2.6	ND	ND	3.5	ND	ND	26.8	35.4%
ARUSPEMB3	1.9	ND	ND	4.0	ND	1.4	29.5	39.5%
ARUSPEMB4	2.5	ND	ND	3.7	ND	2.3	33.2	44.4%
PEMBINA1†	ND	ND	ND	ND	ND	3.0	ND	29.8%
PEMBINA2†	ND	ND	ND	ND	ND	3.4	ND	33.5%
PEMBINA3†	ND	ND	ND	ND	ND	2.6	ND	41.2%
PEMBINA4†	ND	ND	ND	ND	ND	ND	ND	47.1%
ARUSSMTH1	2.0	ND	ND	4.9	ND	1.5	20.2	32.1%
ARUSSMTH2	2.2	ND	ND	4.7	ND	2.3	28.4	47.1%
ARUSSMTH3	2.0	ND	ND	5.2	ND	1.8	18.0	18.4%
ARUSSMTH4	1.4	ND	ND	3.9	ND	2.9	35.2	54.5%
LESSERSL1†	ND	ND	ND	ND	ND	1.6	ND	46.8%
LESSERSL2†	ND	ND	ND	ND	ND	4.0	ND	28.1%
LESSERSL3†	ND	ND	ND	ND	ND	2.5	ND	39.2%
LESSERSL4†	ND	ND	ND	ND	ND	4.3	ND	32.7%
SLPEF2*	ND	ND	ND	ND	ND	5.6	ND	23.7%
SLPEF4*	ND	ND	ND	ND	ND	5.4	ND	33.8%
ARATHA1	1.6	ND	ND	5.2	ND	1.3	19.3	38.4%
ARATHA2	1.3	ND	ND	6.3	ND	2.4	31.4	38.0%
ARATHA3	1.2	ND	ND	5.6	ND	1.7	24.5	38.7%
ARATHA4	1.2	ND	ND	6.7	ND	3.0	34.3	15.9%
ATHSTPEF2*	ND	ND	ND	ND	ND	ND	ND	39.1%
ATHSTPEF4*	ND	ND	ND	ND	ND	ND	ND	27.7%
ARUSLAB1	ND	ND	ND	6.7	ND	1.9	26.4	33.7%
ARUSLAB2	ND	ND	ND	6.3	ND	2.4	24.9	38.1%
ARUSLAB3	ND	ND	ND	7.4	ND	1.9	22.8	34.6%
ARUSLAB4	ND	ND	ND	7.1	ND	2.6	20.5	37.3%
ARUSGR1	ND	ND	ND	9.7	ND	2.1	39.3	36.1%
ARUSGR2	ND	ND	ND	10.7	ND	2.5	29.8	31.1%
ARUSGR3	ND	ND	ND	11.0	ND	2.2	38.4	33.8%
ARUSGR4	ND	ND	ND	10.5	ND	2.4	22.2	30.5%
ARDSGR1	ND	ND	ND	11.9	ND	2.0	28.9	28.8%
ARDSGR2	ND	ND	ND	10.4	ND	1.8	25.9	39.3%
ARDSGR4	ND	ND	ND	9.1	ND	2.2	24.7	30.6%
ARUSFMCM1	ND	ND	ND	9.2	ND	1.2	25.2	28.0%
ARUSFMCM2	ND	ND	ND	11.9	ND	2.2	21.9	36.4%
ARUSFMCM3	ND	ND	ND	13.9	ND	1.7	26.2	26.9%
ARUSFMCM4	ND	ND	ND	17.3	ND	3.3	30.4	24.4%

* Effluent sample diluted 20:1 (actual concentrations are 20 times greater)
† Tributary sample

continued...

Table 4. Concluded

Sample ID	IPMP ng/L	IBMP ng/L	MIB ng/L	246TCA ng/L	236TCA ng/L	Geosmin ng/L	345TCV ng/L	Chlorodecane Recovered
FMCMRAW1	ND	ND	ND	9.3	ND	0.6	13.3	42.3%
FMCMRAW2	ND	ND	ND	11.4	ND	1.6	18.5	37.9%
FMCMRAW3	ND	ND	ND	10.2	ND	0.9	11.2	35.5%
FMCMRAW4	ND	ND	ND	18.7	ND	2.7	23.1	19.9%
FMCMFIN1‡	ND	ND	ND	14.2	ND	1.7	18.8	28.0%
FMCMFIN2‡	ND	ND	ND	11.1	ND	2.1	18.5	31.8%
FMCMFIN3‡	ND	ND	ND	11.7	ND	ND	12.4	29.2%
FMCMFIN4‡	ND	ND	ND	9.9	ND	ND	11.6	40.5%
FMCMSTP2*	ND	ND	ND	ND	ND	ND	ND	27.4%
FMCMSTP4*	ND	ND	ND	ND	ND	ND	ND	33.3%
CLEARWAT3†	ND	ND	ND	ND	ND	5.1	ND	20.8%
CLEARWAT4†	ND	ND	ND	ND	ND	4.4	ND	25.9%
SUNCOREF2*	ND	ND	ND	ND	ND	ND	ND	38.7%
SUNCOREF4*	ND	ND	ND	ND	ND	ND	ND	42.3%
ARUSFBG1	ND	ND	ND	4.8	ND	1.3	7.2	45.3%
ARUSFBG2	ND	ND	ND	ND	ND	ND	ND	11.1%
ARUSFBG3	ND	ND	ND	5.4	ND	2.0	6.4	40.8%
LAKATHFC1	ND	ND	ND	ND	ND	ND	ND	31.6%
LAKATHFC2	ND	ND	ND	ND	ND	ND	ND	39.8%
LAKATHFC3	ND	ND	ND	ND	ND	ND	ND	25.1%
LAKATHFC4	ND	ND	ND	ND	ND	ND	ND	41.7%
FCHIPFIN1‡	ND	ND	ND	ND	ND	3.3	ND	29.3%
FCHIPFIN2‡	ND	ND	ND	ND	ND	ND	ND	44.4%
FCHIPFIN3‡	ND	ND	ND	ND	ND	1.3	ND	24.2%
FCHIPFIN4‡	ND	ND	ND	ND	ND	ND	ND	39.2%

* Effluent sample diluted 20:1 (actual concentrations are 20 times greater)
† Tributary sample
‡ Treated drinking water sample

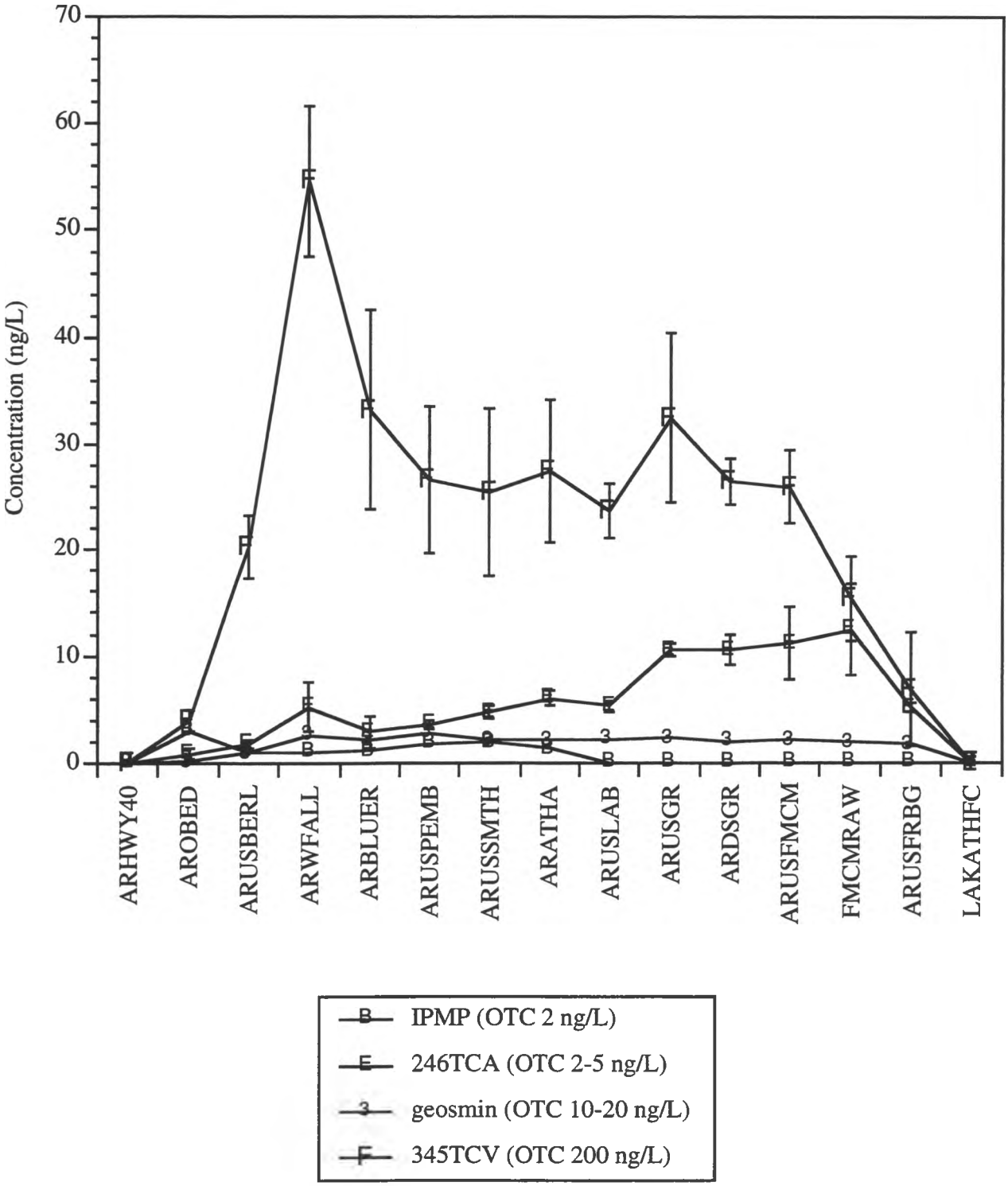

Figure 2. CLSA/GC/MS RESULTS FOR ATHABASCA RIVER TIME-OF-TRAVEL SAMPLES

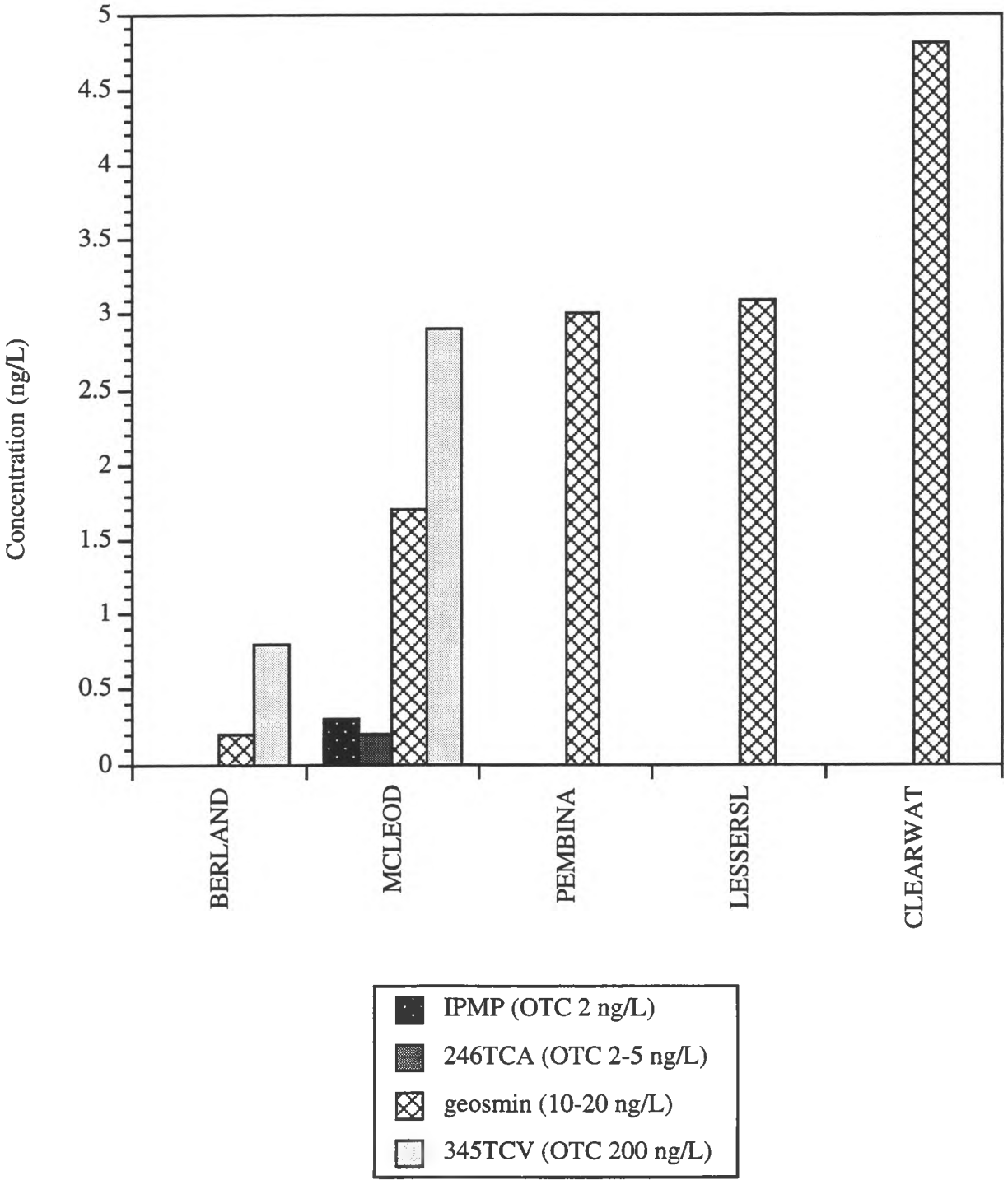

Figure 3. CLSA/GC/MS RESULTS FOR TRIBUTARY SAMPLES

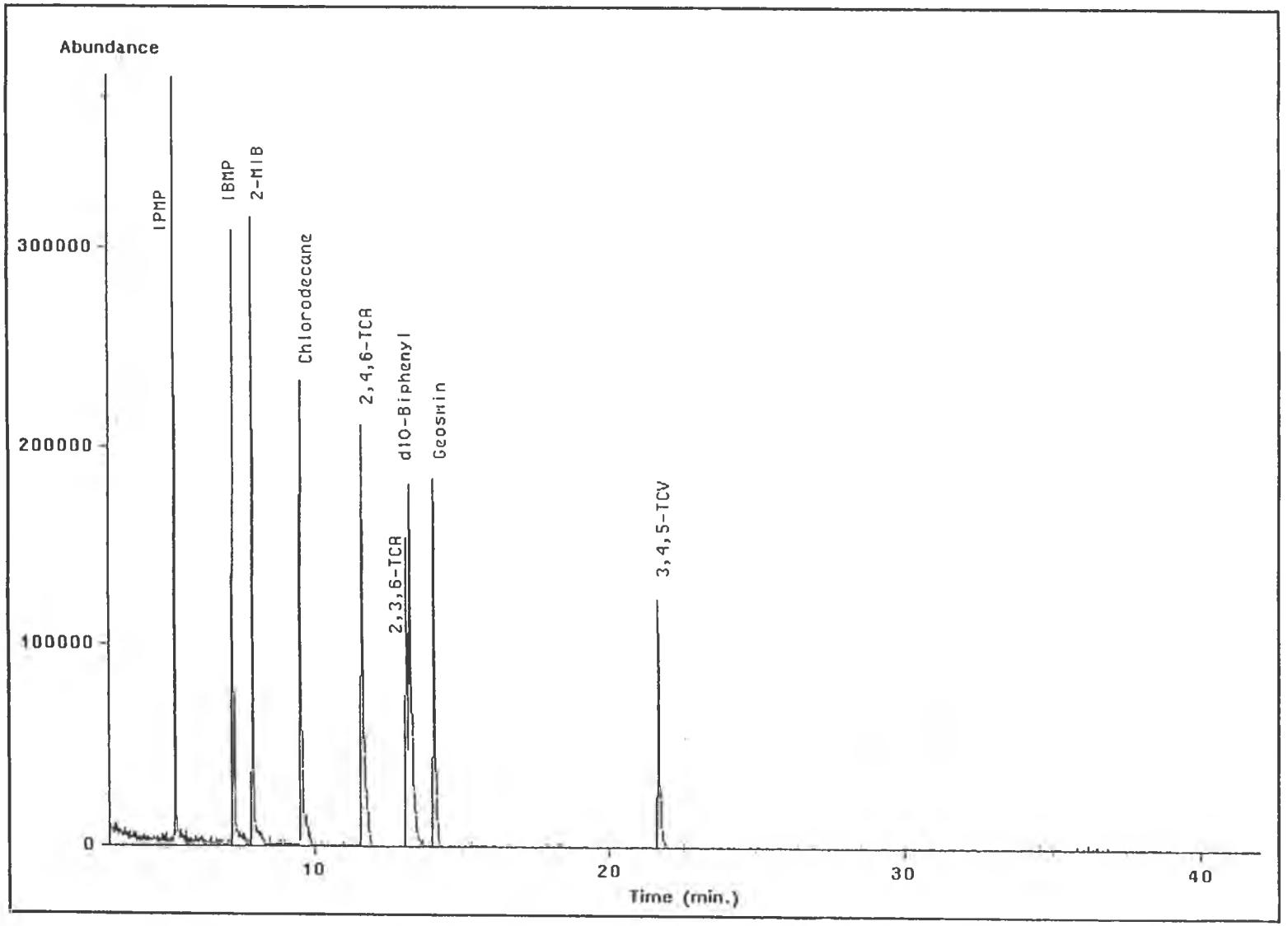

Figure 4. TOTAL ION CHROMATOGRAPH OF TARGET COMPOUND STANDARDS AND INTERNAL STANDARDS

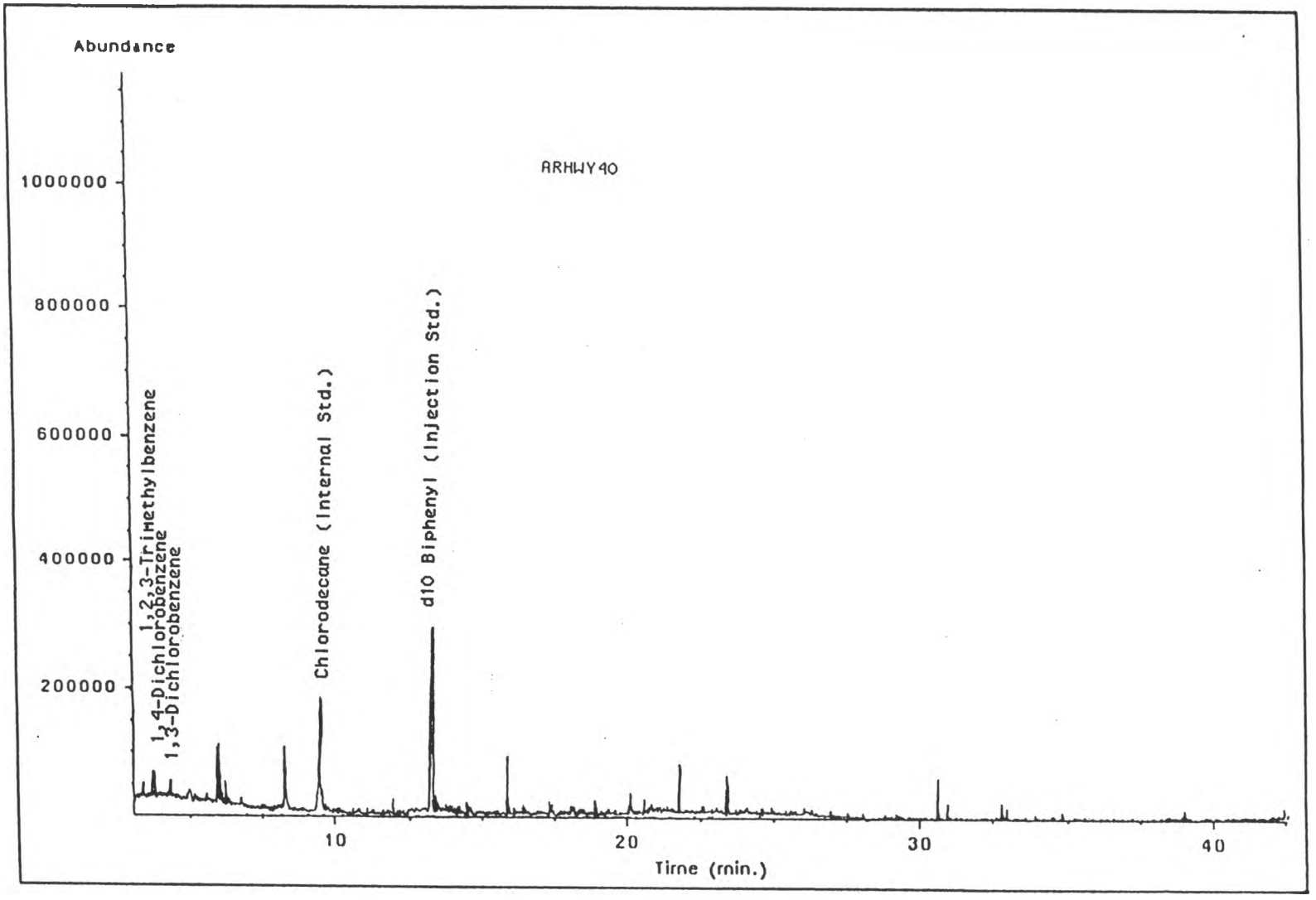

Figure 5. TOTAL ION CHROMATOGRAPH OF ARHWY40 CLSA EXTRACT

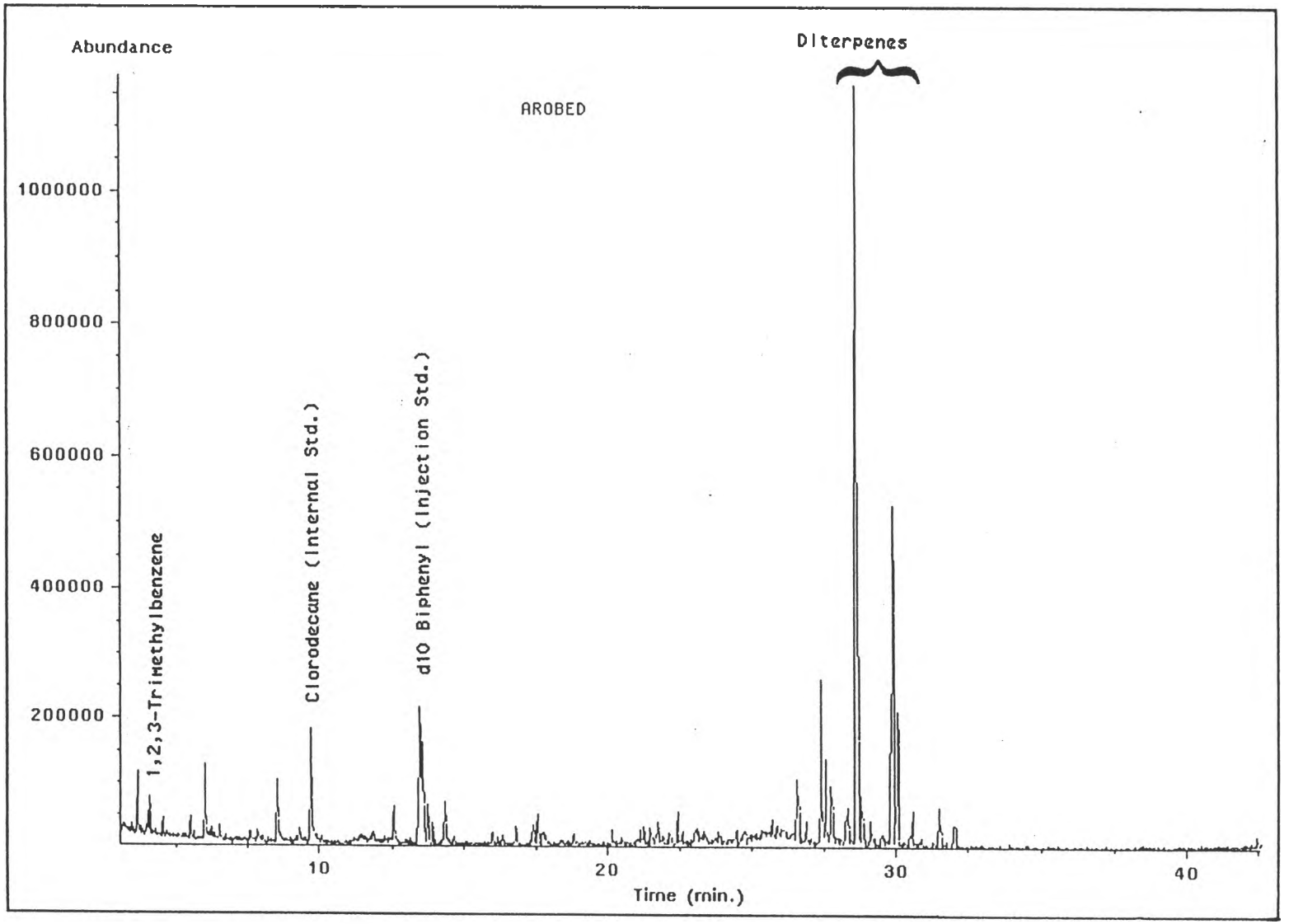

Figure 6. TOTAL ION CHROMATOGRAPH OF AROBED CLSA EXTRACT

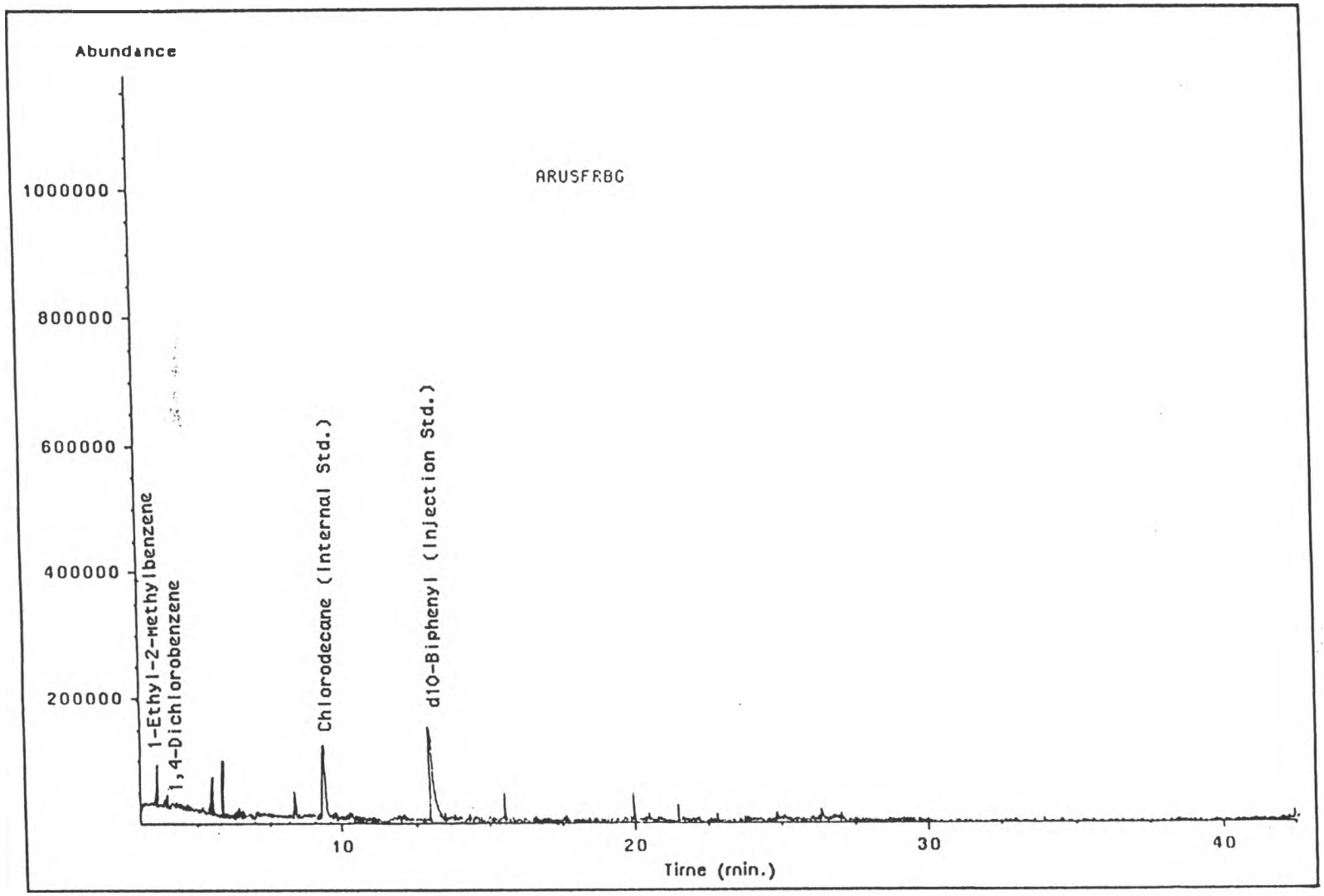

Figure 7. TOTAL ION CHROMATOGRAPH OF ARUSFRBG CLSA EXTRACT

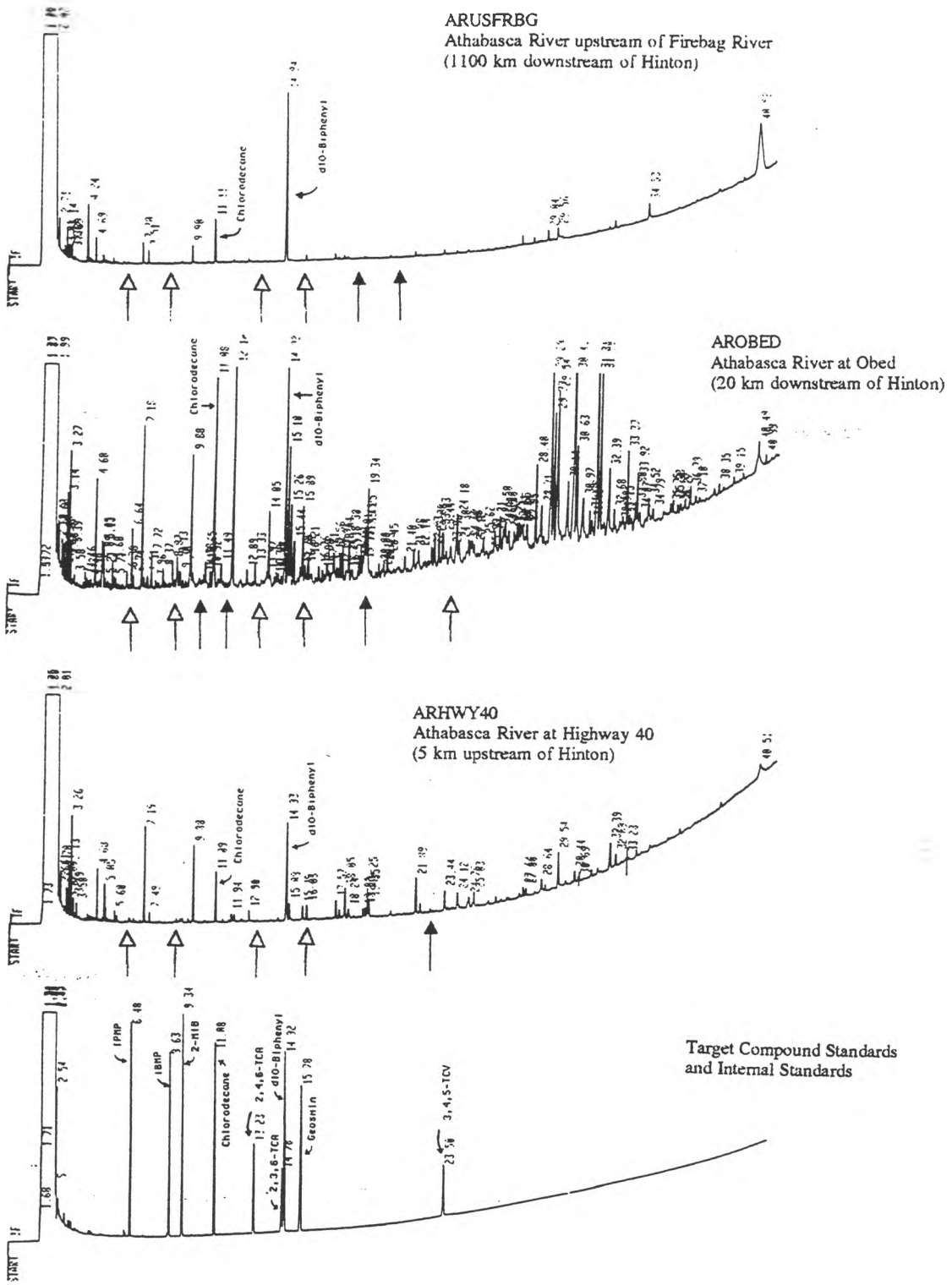

Figure 8. FID CHROMATOGRAMS OF ARUSFRBG, AROBED, AND ARHWY40 CLSA EXTRACTS WITH FID CHROMATOGRAPH OF STANDARDS FOR COMPARATIVE PURPOSES.

(" ▲ " marks odour peaks detected by OGC)

(" △ " marks target odour compound detected by OGC)

3.2 OLFACTORY GAS CHROMATOGRAPHY

Results of the OGC analyses are summarized in Table 5. Samples were run by two analysts and there is general agreement between the two, however one analyst showed greater sensitivity toward the pyrazines, IPMP and IBMP, and the other analyst showed greater sensitivity toward the musty cork odours such as 2,4,6-TCA. As well, each analyst experienced some day-to-day variation in sensitivity over the six-week period when samples were analyzed. The odour peaks detected by OGC are also marked by the large arrows underneath each FID sample chromatogram shown in Figures 6 and 7. These markers demonstrate that not all odour peaks detected by OGC are detected using an instrumental flame ionization detector, verifying the greater sensitivity of the human nose.

Results obtained for OGC of CLSA blanks indicate that there may be some carry-over of IPMP, IBMP and 2,4,6-TCA after running standard calibration solutions. Adequate purging and washing of the CLSA apparatus between samples is essential.

Mainstem Athabasca samples, including the site upstream from Hinton at Highway 40, showed moderate to strong levels of 2,4,6-TCA all the way to the Firebag River. The two unidentified musty cork odour peaks at 19.3 and 22.4 retention times showed up quite consistently at weak to moderate intensities. 3,4,5-TCV showed up occasionally at trace to weak intensities. The SULF and CRDOIL odour peaks were strongest in the upper reaches and diminished to non detectable levels by Fort McMurray. These or similar peaks also appeared in some of the effluent and tributary samples. Geosmin occurred quite consistently at trace to moderate levels. There were a variety of miscellaneous odour peaks at trace to weak intensity in the mainstem samples. These are not reported nor discussed at present because they occurred at low frequency and showed no consistent pattern. Tributary samples (Berland, McLeod, Pembina, Lesser Slave, Clearwater) had fewer odour peaks than mainstem samples and intensities of reported peaks were generally lower than mainstem samples.

The predominant odours in the Hinton Combined Effluent (bleached kraft pulp mill and municipal) were the pyrazine (IPMP and IBMP), sulfurous and musty cork odours. One analyst also detected woody, sewage and spicy odours. Geosmin was not detected by either analyst. The Chemi-thermomechanical pulp (CTMP) mill effluents (MWEF and SLPEF) were generally low in odour. Distinctive odours were woody, cardboard and sulfurous. The municipal waste water treatment plant effluents (WCSTPEF, ATHSTPEF, and FMCMSTP) were also low in the odour peaks we have reported here, but had a variety of distinctive odour peaks not found in the other samples such as waxy, flowery, soapy, woolly, spicy and cucumber. The odour profiles for river, raw and finished water samples at Fort McMurray were all similar, but the agreement between analysts was poor for these samples. Lake Athabasca and Fort Chipewyan finished water samples had very few odour peaks and intensities were low for peaks observed.

3.3 FLAVOUR PROFILE ANALYSES

The two flavour profile panels for this project worked completely independently of one another. The results for the experienced panel (E.L. Smith) are presented in Table 6 and those of the newly trained panel (U. of A.) are presented in Table 7. The experienced panel generally used a higher range of intensities for all samples than those assigned by the inexperienced panel. These differences likely reflect the wider range of samples that the experienced panel has tested, providing them with greater confidence in assigning maximum ratings (3). Although the main objective of this study was to evaluate spatial variability of odour characteristics of the Athabasca River, a secondary concern was the subjective nature of sensory evaluations. Combining the results of a trained panel with those of a less experienced panel made up of members of the general population allowed for a qualitative evaluation of

Table 5: OLFACTORY GAS CHROMATOGRAPHY RESULTS

Site	Odour Descriptor and Retention Time (minutes)								
	IPMP 6.6	IBMP 8.8	SULF 10.3	CRDOIL 11.9	246TCA 13.4	Geosmin 16.0	MCX 19.3	MCY 22.4	345TCV 23.9
ARHWY40	■ ❑	■ ❑❑			■■ ❑	■		■	
HCEEF#	❑❑❑❑	❑❑	■ ❑❑❑❑	■■	■■■■ ❑❑❑❑❑		■ ❑❑ (?)		
AROBED	■■■■ ❑❑❑❑	❑❑	■ ❑❑❑	■■	■■■■ ❑❑❑	■■	■■		■
ARUSBERL	■ ❑❑		■ ❑❑	■■	■■■■■ ❑❑❑	■■	■■	■	■■
BERLAND†	■ ❑❑	❑	■					■	
ARWFALL	❑	❑	■■	■■ ❑	■■■■■ ❑❑❑❑❑	❑	■■		
MCLEOD†	■ ❑	❑			❑❑	❑		■	
ANCEF#	■■■ ❑			■	■■ ❑	■■			
MWEF#	■■■ ❑ (?)	■	❑		■ ❑	■■■■ ❑			
WCSTPEF#	■■ ❑❑	■ ❑❑	❑❑	■■■	■	■			

IPMP - isopropylmethoxypyrazine
IBMP - isobutylmethoxypyrazine
SULF at 10.33 minutes is a sulfury/septic odour
CRDOIL at 11.92 minutes is a sulfury/mercaptan/crude oil odour
246TCA - 2,4,6-trichloroanisole
MCX at 19.33 minutes is an unidentified musty cork odour
MCY at 22.42 minutes is an unidentified musty cork odour
345TCV - 3,4,5-trichloroveratrole

Effluent sample
† Tributary sample
‡ Water Supply sample

Intensities are on a six-point scale from ■ (very weak) to ■■■■■■ (very strong)
"■" and "❑" are intensities recorded by the two panelists
(?) after an intensity indicates odour descriptor did not match well

continued...

Site	Odour Descriptor and Retention Time (minutes)								
	IPMP	IBMP	SULF	CRDOIL	246TCA	Geosmin	MCX	MCY	345TCV
	6.6	8.8	10.3	11.9	13.4	16.0	19.3	22.4	23.9
ARBLUER	■■■	■			■■■	■■■	■	■	
	❑	❑			❑❑❑	❑	❑❑		
ARUSPEMB			■		■■■	■	■■	■	■
	❑❑❑		❑❑		❑❑❑				
PEMBINA†	■■				■■	■■	■	■■	
	❑❑❑	❑			❑❑				
ARUSSMTH	■■	■			■■■	■	■■		■
	❑❑❑	❑❑	❑❑		❑❑				
LESSERSL†	■	■		■	■	■			
	❑	❑	❑		❑				
SLPEF#	■■				■	■■			
	❑❑❑	❑				❑❑			
ARATHA	■■■	■■ (?)		■	■■■■	■■	■	■	
	❑❑❑ (?)		❑		❑				
ATHSTPEF#	■■■■	■■			■	■			
	❑	❑			❑				
ARUSLAB	■■■	■			■■■■	■■■	■■	■	■
	❑	❑	❑		❑				
ARUSGR	■■■	■			■■■■	■■	■■		
	❑	❑			❑				
ARDSGR	■■		■		■■■■	■■■	■■■	■	■
	❑	❑	❑		❑	❑	❑		

IPMP - isopropylmethoxypyrazine
IBMP - isobutylmethoxypyrazine
SULF at 10.33 minutes is a sulfury/septic odour
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345TCV - 3,4,5-trichloroveratrole

Effluent sample
† Tributary sample
‡ Water Supply sample

Intensities are on a six-point scale from ■ (very weak) to ■■■■■■ (very strong)
"■" and "❑" are intensities recorded by the two panelists
(?) after an intensity indicates odour descriptor did not match well

continued...

Table 5. Concluded.

Site	Odour Descriptor and Retention Time (minutes)								
	IPMP 6.6	IBMP 8.8	SULF 10.3	CRDOIL 11.9	246TCA 13.4	Geosmin 16.0	MCX 19.3	MCY 22.4	345TCV 23.9
ARUSFMCM	■■■				■■■■■	■■	■■■	■	■
					❑				
FMCMRAW‡	■■■				■■■■	■	■■	■	
					❑				
FMCMFIN‡	■■■	■■			■■■■	■■■	■■■	■	■
	❑❑	❑			❑				
FMCMSTP#	■■■				■■	■			
	❑❑	❑							
CLEARWAT†	■■■	■			■■	■■■■	■	■	
	❑				❑				
SUNCOREF#	■■	■■			■■■	■			
	❑❑	❑❑	❑		❑❑				
ARUSFRBG	■■■				■■■■	■■	■■	■	
	❑❑❑❑❑	❑❑❑❑❑			❑❑❑❑	❑❑❑			
LAKATHFC	■■				■				
	❑❑				❑				
FCHIPFIN‡	■■				■				
	❑				❑❑❑				

IPMP - isopropylmethoxypyrazine
IBMP - isobutylmethoxypyrazine
SULF at 10.33 minutes is a sulfury/septic odour
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Effluent sample
† Tributary sample
‡ Water Supply sample

Intensities are on a six-point scale from ■ (very weak) to ■■■■■■ (very strong)
"■" and "❑" are intensities recorded by the two panelists
(?) after an intensity indicates odour descriptor did not match well

Table 6. E.L. SMITH FLAVOUR PROFILE PANEL RESULTS

Sample Site	Panelist 1 Descriptor	Intensity	Panelist 2 Descriptor	Intensity	Panelist 3 Descriptor	Intensity
ARHWY40	chemical	0.50	musty	0.50	woody musty	0.50
HCEEF	muni ww decay	3.00	rancid fish	3.00	rancid oil	2.50
AROBED	decay	3.00	rancid fish	3.00	rancid oily	2.50
ARUSBERL	muni ww	2.50	rancid fish	3.00	sulphur oily	3.00
BERLAND	chemical	0.50	musty	0.50	trace	0.25
ARWFALL	resin rotting septic	3.00	decay veg septic	3.00	decay potato bin	3.00
MCLEOD	light chemical	0.50	sweet	0.50	wood shavings	0.50
ANCEF	musty ascorbic	0.50	sweet ascorbic	0.50	moldy ascorbic	0.50
MWEF	geosmin earthy	0.50	musty earthy	1.00	geosmin	1.00
WCSTPEF	pepper spicy	0.75	sour	0.50	chemical spicy	0.50
ARBLUER	resin rotting septic	3.00	decay veg septic	3.00	decay potato bin	2.50
ARUSPEMB	rotting	2.00	-		-	
PEMBINA	musty moldy	1.00	-		-	
ARUSSMTH	resin swampy	1.00	-		-	
LESSERSL	musty	0.50	-		-	
SLPEF	chemical organic	1.00	-		-	
ARATHA	resin woody	2.50	-		-	
ATHSTPEF	rotting pepper	1.50	-		-	
ARUSLAB	rotting sewage	2.50	decay	1.50	-	
ARUSGR	org decay sewage	2.00	decay	1.50	-	
ARDSGR	org decay sewage	2.50	decay	1.50	-	
ARUSFMCM**	dichloromethane	3.00	sweet org solvent	3.00	-	
FMCMRAW	org decay sewage	2.00	decay	1.50	-	
FMCMFIN	sweet org chem	2.00	plast chemical	1.00	-	
FMCMSTP	rotting	1.00	sewage	1.50	-	
CLEARWAT	glue plastic	0.75	trace	0.25	-	
SUNCOREF	heavy hydro oil	3.00	decay burnt wood	2.00	-	
ARUSFRBG	woody musty	0.50	chemical pesticide	1.50	moldy musty	1.00
LAKATHFC	musty	0.50	trace	0.25	trace	0.25
FCHIPFIN	chlorine	1.00	chlorine	1.50	chlorine	1.50

chem - chemical
veg - vegetation
plast - plastic
org - organic
muni ww - municipal wastewater
hydro - hydrocarbon
** - contaminated sample container

continued...

Table 6. Concluded.

Sample Site	Panelist 4 Descriptor	Intensity	Panelist 5 Descriptor	Intensity	MEAN	Standard Deviation
ARHWY40	woody geranium	1.00	woody	1.00	0.70	{0.27}
HCEEF	woody rancid	1.50	woody rancid	3.00	2.60	{0.65}
AROBED	litter box rancid	3.00	woody rancid	2.00	2.70	{0.44}
ARUSBERL	geraniums	2.50	woody rancid	2.50	2.70	{0.27}
BERLAND	woody	1.00	woody	1.00	0.65	{0.33}
ARWFALL	fishy	3.00	sweet plastic	2.50	2.90	{0.22}
MCLEOD	wood shavings	1.50	sweet plastic	0.75	0.75	{0.43}
ANCEF	trace	0.25	flowery sweet	0.75	0.50	{0.18}
MWEF	trace	0.25	trace	0.25	0.60	{0.38}
WCSTPEF	trace	0.25	pepper	1.00	0.60	{0.29}
ARBLUER	fishy woody	3.00	sweet plastic	1.50	2.60	{0.65}
ARUSPEMB	musty peat fragrant	2.50	plastic weedy	2.50	2.33	{0.29}
PEMBINA	musty	0.50	plastic	1.00	0.83	{0.29}
ARUSSMTH	fishy musty	0.50	burnt weedy	1.00	0.83	{0.29}
LESSERSL	trace	0.25	plastic weedy	2.00	0.92	{0.95}
SLPEF	musty oily	3.00	rubber plastic	1.00	1.67	{1.15}
ARATHA	sweet clover peat	1.50	damp musty	1.00	1.67	{0.76}
ATHSTPEF	decay musty	1.00	damp musty	0.75	1.08	{0.38}
ARUSLAB	chemical decay	1.50	musty plastic	1.00	1.63	{0.63}
ARUSGR	sweet chem decay	2.00	musty plastic	1.00	1.63	{0.48}
ARDSGR	sweet chem decay	2.00	musty plastic	1.00	1.75	{0.65}
ARUSFMCM**	chem acetone	2.00	medicinal	3.00	2.75	{0.50}
FMCMRAW	musty decay	2.00	musty vegetation	1.00	1.63	{0.48}
FMCMFIN	sweet chem	1.50	musty plastic	0.75	1.31	{0.55}
FMCMSTP	musty decay	0.50	musty veg	1.00	1.00	{0.41}
CLEARWAT	trace	0.25	trace	0.25	0.38	{0.25}
SUNCOREF	sweet chem decay	2.00	moldy vegetation	3.00	2.50	{0.58}
ARUSFRBG	woody	1.00	sweet barn like	2.00	1.20	{0.57}
LAKATHFC	rancid fishy	0.50	-	0.00	0.30	{0.21}
FCHIPFIN	chlorine	1.50	sweet	0.50	1.20	{0.45}

chem - chemical
veg - vegetation
plast - plastic
org - organic
muni ww - municipal wastewater
hydro - hydrocarbon
** - contaminated sample container

Table 7. UNIVERSITY OF ALBERTA FLAVOUR PROFILE PANEL RESULTS

Sample Site	Panelist 1 Descriptor	Intensity	Panelist 2 Descriptor	Intensity	Panelist 3 Descriptor	Intensity
ARHWY40	fruity	1	grassy	0.5	slight camphor	0.5
HCEEF	swampy	2	p&p/sewage	2	earthy/damp soil	1
AROBED	fruity	1	hydro/sewage	1	earthy/potato	1
ARUSBERL	swampy	1	p&p/chlor	1	must/mold	1
BERLAND	almost nothing	0.25	odorless	0	odorless	0
ARWFALL	earthy	1	septic/sulphur	0.5	-	-
MCLEOD	almost odorless	0.25	earthy	t	-	-
ANCEF	fragrant	0.75	earthy	t	-	-
MWEF	?	0.5	earthy	0.5	-	-
WCSTPEF	weak	t	odorless	0	-	-
ARBLUER	earthy	2	septic/swampy	1.5	-	-
ARUSPEMB	marshy/swampy	2	swampy (p&p)	2	earthy/musty	1
PEMBINA	marshy/ swampy	2	earthy/grassy	0.5	menthol	0.5
ARUSSMTH	marshy/swampy	1	swampy/p&p	1.5	-	-
LESSERSL	faint chlor	1	earthy/chalk/grass	t	-	-
SLPEF	chem/ medicinal	0.5	grassy/ woody	0.5	-	-
ARATHA	-	-	-	-	-	-
ATHSTPEF	-	-	-	-	-	-
ARUSLAB	-	-	-	-	musty	2
ARUSGR	septic	1.5	swampy/p&p	2.5	musty, p&p	1.5
ARDSGR	septic	0.75	swampy/p&p	2	p&p/slight chlor	1.5
ARUSFMCM	swampy	1.75	earthy camphor	2	p&p/camphor	1.5
FMCMRAW	woody	2	earthy/grassy p&p	1.5	musty/camphor	2
FMCMFIN	chem	0.5	grassy/ chlor	0.5	musty/ chlor	1
FMCMSTP	almost odorless	0.25	earthy/sewage	t	citrus	0.75
CLEARWAT	faint	0.25	grassy	0.5	odorless	0
SUNCOREF	med/phenolic	2	musty root cellar	1.5	-	-
ARUSFRBG	marshy	1.25	earth/grass/swamp	1.25	-	-
LAKATHFC	musty/earthy	0.75	grassy, earthy	0.5	-	-
FCHIPFIN	chlor	2	chlor	2.5	-	-

p&p - pulp & paper
chem - chemical
veg - vegetation
org - organic
hydro - hydrocarbon
med - medicinal
chlor - chlorine

continued...

Sample Site	Panelist 4 Descriptor	Intensity	Panelist 5 Descriptor	Intensity	Panelist 6 Descriptor	Intensity
ARHWY40	?	0.5	-	-	odorless	0
HCEEF	p&p	3	-	-	woody/septic/musty	2.5
AROBED	sewage, rotten	1.75	-	-	woody	1.5
ARUSBERL	musty	1	-	-	septic/ p&p	1.5
BERLAND	odorless	0	-	-	odorless	0
ARWFALL	swampy/sewage	1.5	damp cellar/woody	1	woody/ sulphur	1.5
MCLEOD	musty	0.5	?potato	t	odorless	0
ANCEF	musty	t	dry dirt	0.5	woody	t
MWEF	acidic??	0.5	turnip/ earthy	1.5	sharp musty	t
WCSTPEF	odorless	0	odorless	0	odorless	0
ARBLUER	p&p	2	earthy/woody	1	woody/ sulphur	1.5
ARUSPEMB	marshy/p&p	2.5	woody/earthy	2	wood/sulphur/p&p	1.75
PEMBINA	grassy	0.75	grassy	t	earthy/woody	0.5
ARUSSMTH	-	-	earthy	1	p&p/sulphur	1.5
LESSERSL	-	-	marshy	1	earthy/camphor	0.5
SLPEF	-	-	musty/chalky	2	woody/camphor	0.5
ARATHA	-	-	-	-	p&p/sulphur	1.5
ATHSTPEF	-	-	-	-	sewage/fecal	1.5
ARUSLAB	swampy/septic	1.5	-	-	septic/p&p	2
ARUSGR	p&p	2	earthy/ p&p	2	sharp/woody	1.5
ARDSGR	swampy/p&p	2.5	earthy/p&p/chlor	1.5	p&p	0.5
ARUSFMCM	septic/p&p	2	grassy	1	p&p	1
FMCMRAW	p&p	1	earthy/p&p	1.5	sharp/woody	t
FMCMFIN	chlor	1	chlor/ chem	1.5	benzaldehyde	1
FMCMSTP	musty/sharp	0.5	septic/sewage	1.5	septic/sewage	0.5
CLEARWAT	veg/fragrant	1.5	fishy?	t	flowery	t
SUNCOREF	-	-	gasoline/chem	1	grassy/hydro	1.5
ARUSFRBG	-	-	earthy	2	woody/sulphur	0.5
LAKATHFC	-	-	earthy	0.5	woody/septic	t
FCHIPFIN	-	-	chlor (grassy?)	1.5	chlor/aldehyde	1.5

p&p - pulp & paper
chem - chemical
veg - vegetation
org - organic
hydro - hydrocarbon
med - medicinal
chlor - chlorine

continued...

Table 7. Concluded.

Sample Site	Panelist 7 Descriptor	Intensity	Panelist 8 Descriptor	Intensity	MEAN	Standard Deviation
ARHWY40	weak acid	0.5	odorless	0	0.43	{0.35}
HCEEF	distinct sewage	3	sewage/swampy/p&p	1.5	2.1	{0.75}
AROBED	weak sewage	1.5	p&p	2	1.39	{0.40}
ARUSBERL	sewage natural gas	1	sewage/swampy	1	1.07	{0.19}
BERLAND	? hint chlor	t	?	t	0.06	{0.09}
ARWFALL	-	-	p&p	1	1.08	{0.38}
MCLEOD	-	-	odorless	0	0.16	{0.19}
ANCEF	-	-	chalky (clay)	1	0.43	{0.40}
MWEF	-	-	earthy	0.5	0.6	{0.47}
WCSTPEF	-	-	flowery	t	0.03	{0.05}
ARBLUER	-	-	p&p	1.25	1.54	{0.40}
ARUSPEMB	-	-	p&p	1.5	1.82	{0.47}
PEMBINA	veg/grass?	0.5	earthy	t	0.62	{0.60}
ARUSSMTH	p&p/sewage	1	septic/p&p	0.5	1.08	{0.38}
LESSERSL	dry grass	0.5	clay	0.5	0.6	{0.35}
SLPEF	dry hay (musty)	0.5	woody	0.75	0.79	{0.60}
ARATHA	septic	1	p&p	1	1.17	{0.29}
ATHSTPEF	medicinal	0.5	muddy/septic	0.5	0.83	{0.58}
ARUSLAB	septic/musty	1	-	-	1.63	{0.48}
ARUSGR	p&p	1	-	-	1.71	{0.49}
ARDSGR	p&p, septic	0.75	-	-	1.36	{0.73}
ARUSFMCM	p&p/septic	0.5	-	-	1.39	{0.57}
FMCMRAW	p&p/septic	2	-	-	1.44	{0.70}
FMCMFIN	musty/chlor	0.5	-	-	0.86	{0.38}
FMCMSTP	muddy/musty	0.5	-	-	0.59	{0.45}
CLEARWAT	grassy	0.5	-	-	0.42	{0.51}
SUNCOREF	veg/ grassy	2	-	-	1.6	{0.42}
ARUSFRBG	p&p	1	-	-	1.2	{0.54}
LAKATHFC	weak p&p	0.5	-	-	0.47	{0.23}
FCHIPFIN	strong chlor	3	-	-	2.1	{0.65}

p&p - pulp & paper
chem - chemical
veg - vegetation
org - organic
hydro - hydrocarbon
med - medicinal
chlor - chlorine

this subjectivity. The results are summarized in Figures 9 to 14, which show the average intensity ratings for each panel, along with the descriptors used. The results obtained by the University panel were significantly correlated to the average intensities obtained by the E.L. Smith panel (Figure 15, r=0.708, P=0.01, n=30). Although the University of Alberta panel was established as an indicator of the odours detected by the general population, the results demonstrate that even with limited training the new panel reported intensities similar to those of the water treatment industry panel.

Both panels were able to detect a strong increase in odour for Athabasca River samples downstream of Hinton, particularly for the Obed, upstream of Berland, Windfall, Blue Ridge and upstream of Smith samples. Furthermore panelists used descriptors similar to those used for the Hinton combined effluent and some panelists recognized these samples as reminiscent of pulp mill odour. There little indication of any reduced intensity of odour until the sample upstream of Smith. In this stretch the odours of the tributaries (Berland River, McLeod River, and Pembina River were minor and likely contributed little to the odour of the mainstem Athabasca River. The effluent samples from Alberta Newsprint, Millar Western and Whitecourt sewage treatment plant were only slightly more odorous than the tributaries and all were much less odorous than the Hinton combined effluent. For this reach of the Athabasca River to upstream of Smith, the flavour panel results indicate that the Hinton combined effluent is likely the dominant source of odour in the mainstem river.

The mainstem Athabasca River sample near Smith showed a substantial drop in odour intensity compared with the upstream samples, although the odour descriptors used remained consistent with a Hinton combined effluent source. Beyond this point, the Lesser Slave River was about as odorous as the Pembina River and was also not likely a major contributor to the mainstem Athabasca River odour. The Slave Lake Pulp effluent and the Athabasca sewage treatment plant effluents were somewhat more odorous than the upstream effluents (except for the Hinton combined effluent). The mainstem Athabasca River odour increased in intensity at Athabasca and maintained the higher intensity at sites upstream of the LaBiche River, upstream and downstream of Grand Rapids, upstream of Ft. McMurray and in Ft. McMurray raw water. For these samples, the University of Alberta panel often used descriptors implicating pulp and paper, while the City of Edmonton panel used more generic descriptors associated with decaying organic matter. Both panels found that odour in the Ft. McMurray water supply decreased substantially from raw to treated and descriptors that had been primarily associated with the Athabasca River samples downstream of Hinton were not used. These findings suggested that any odour contribution that upstream effluents may have made to odour of Ft. McMurray finished drinking water would not likely be recognized as being associated with pulp mill effluents.

Downstream of Ft. McMurray, the contributions from the Ft. McMurray sewage treatment plant and the Clearwater River to the mainstem Athabasca River odour were likely small. The Suncor effluent was recognized as very distinctive and relatively strong, but with the possible exception of a sulphur descriptor it is difficult to recognized any impact of the Suncor effluent on the Athabasca River sample upstream of the Firebag River. Several panelists described this sample as woody and one used pulp and paper as a descriptor, indicating the possibility of some pulp mill influence remaining apparent this far downstream on the Athabasca River. The Lake Athabasca sample received some descriptors that were consistent with those used on the upstream Athabasca River, but the intensity of perceived odour was as low as that for any sample obtained anywhere in the entire watershed and could be considered background. The treated water at Ft. Chipeweyan had a very strong chlorine odour that likely masked any other subtle odours that may have been present. Future samples should be split into two aliquots with one analyzed by FPA as is and one analyzed after being dechlorinated so that other odours can be detected

These findings are consistent with the work Kovacs and Voss (1986) wherein they found that biologically treated bleached kraft mill effluent could impair drinking water odour (as perceived by a flavour panel) at effluent dilutions in river water as high as 300 to 1000 fold. In the Athabasca River system during low winter flow much lower dilution levels were available to the Hinton combined

effluent and the other less odourous effluent contributions to the Athabasca River. Apparently, the low temperatures and ice cover allow the odour to be transported long distances, as had been observed by field staff during earlier surveys (Brownlee et al. 1992). Except for the drop in odour observed for the Athabasca River upstream of Smith, the flavour panel results suggest that the Hinton combined effluent is currently the major influence upon odour in the Athabasca River, possibly as far downstream as the Firebag River, downstream of Ft. McMurray.

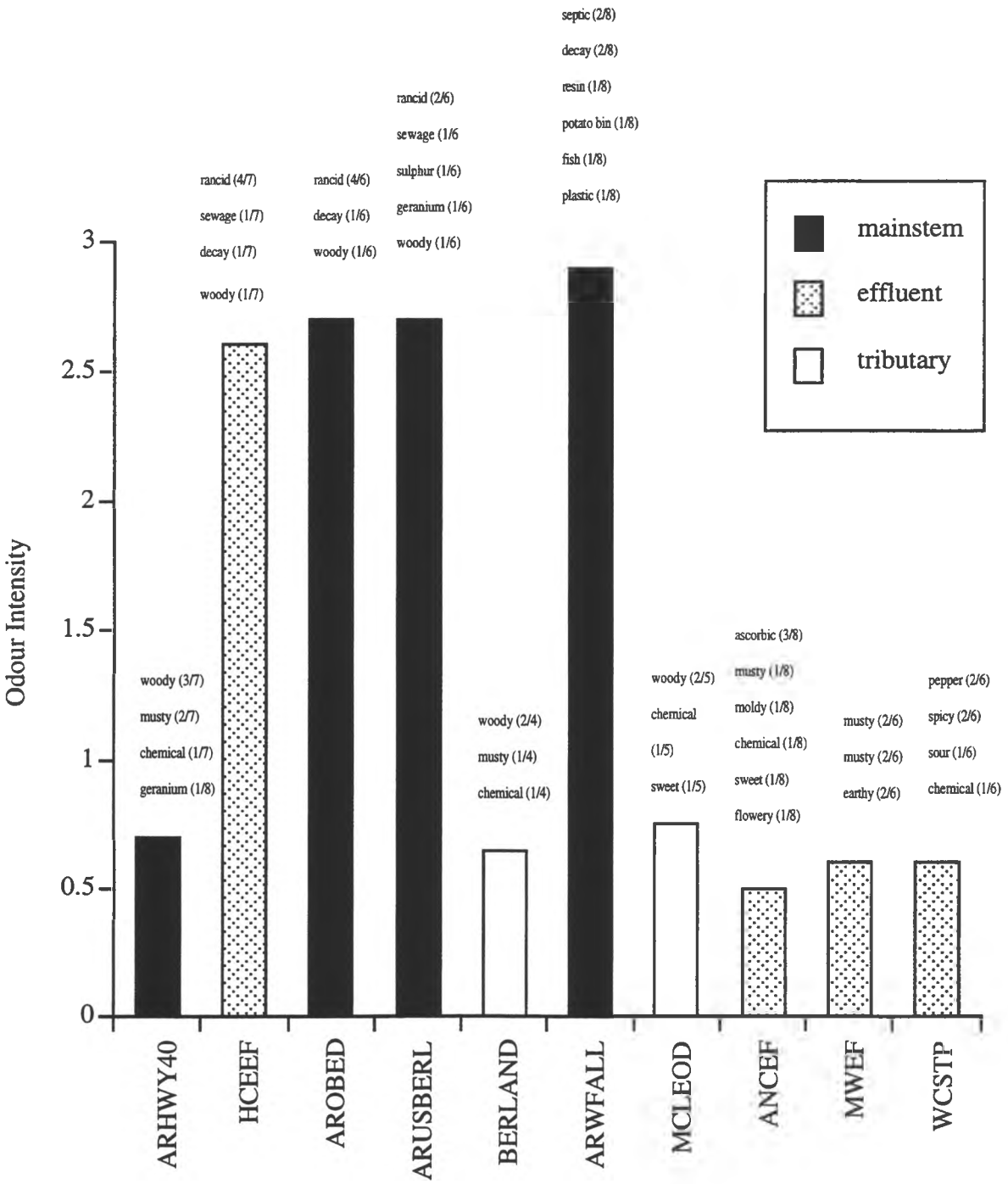

Figure 9. E.L. SMITH FLAVOUR PROFILE ANALYSES SUMMARY FOR FIRST 10 SAMPLING SITES

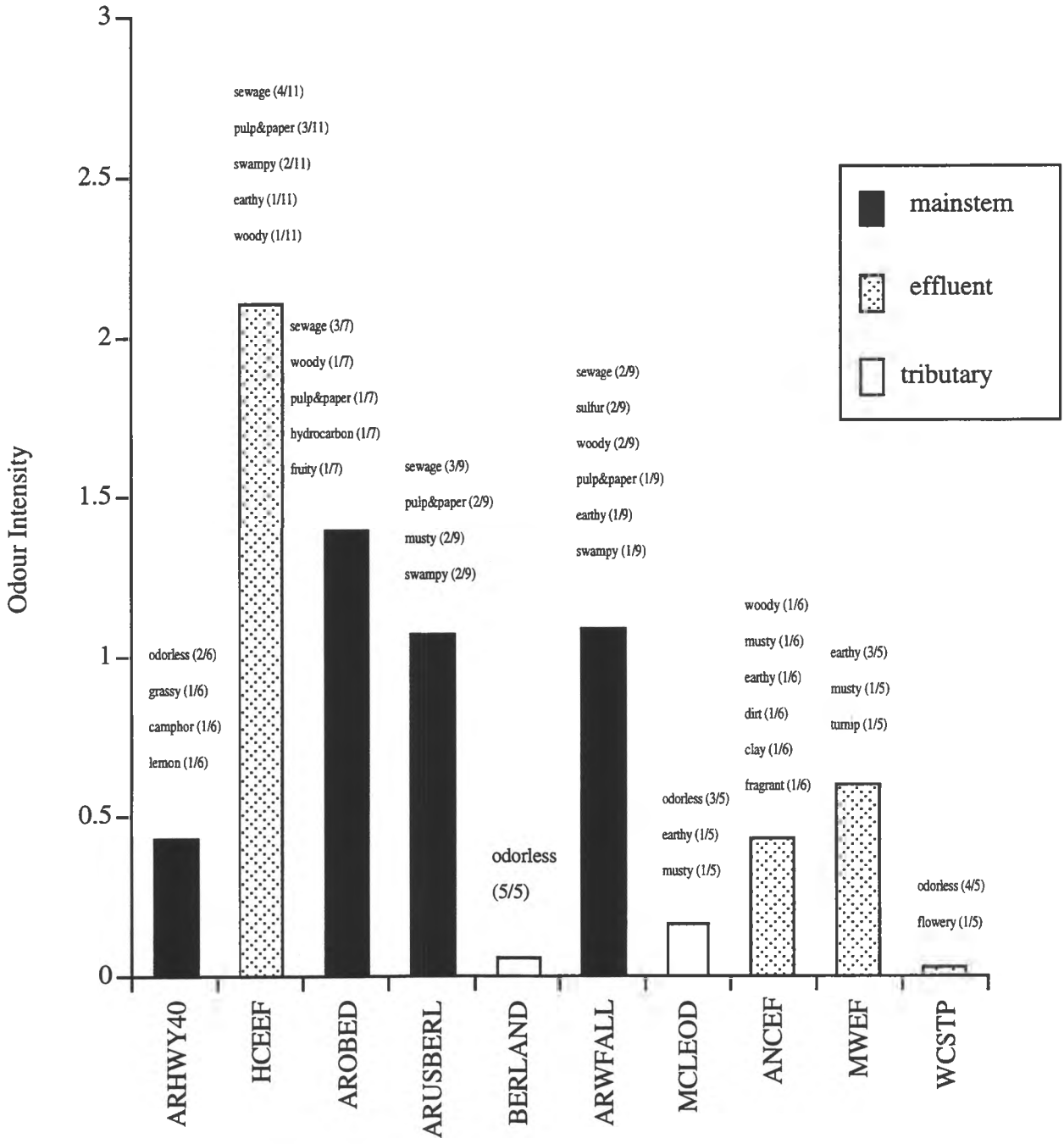

Figure 10. U of A FLAVOUR PROFILE ANALYSES SUMMARY FOR FIRST 10 SAMPLING SITES

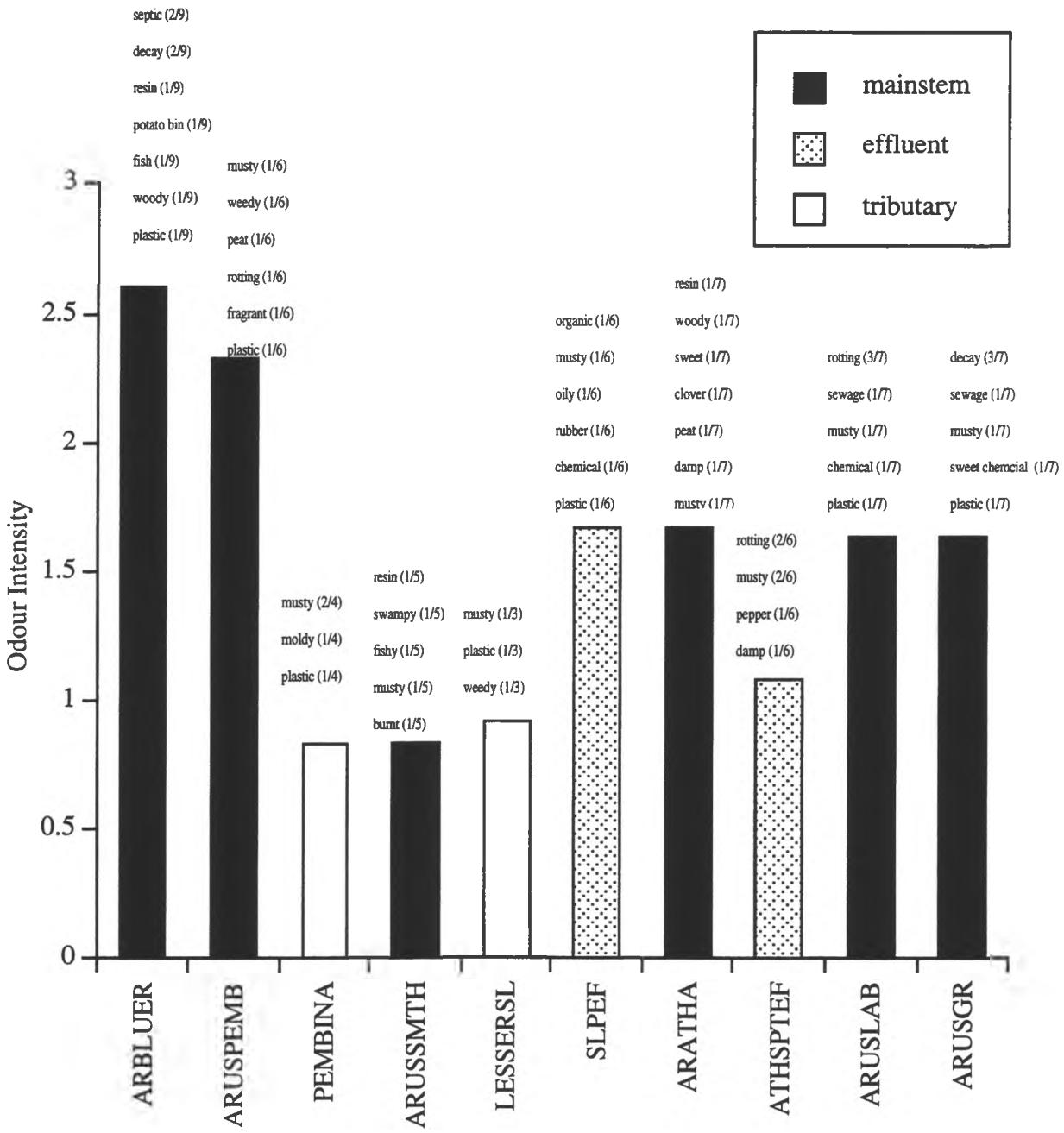

Figure 11. E.L.SMITH FLAVOUR PROFILE ANALYSES SUMMARY FOR SECOND 10 SAMPLING SITES

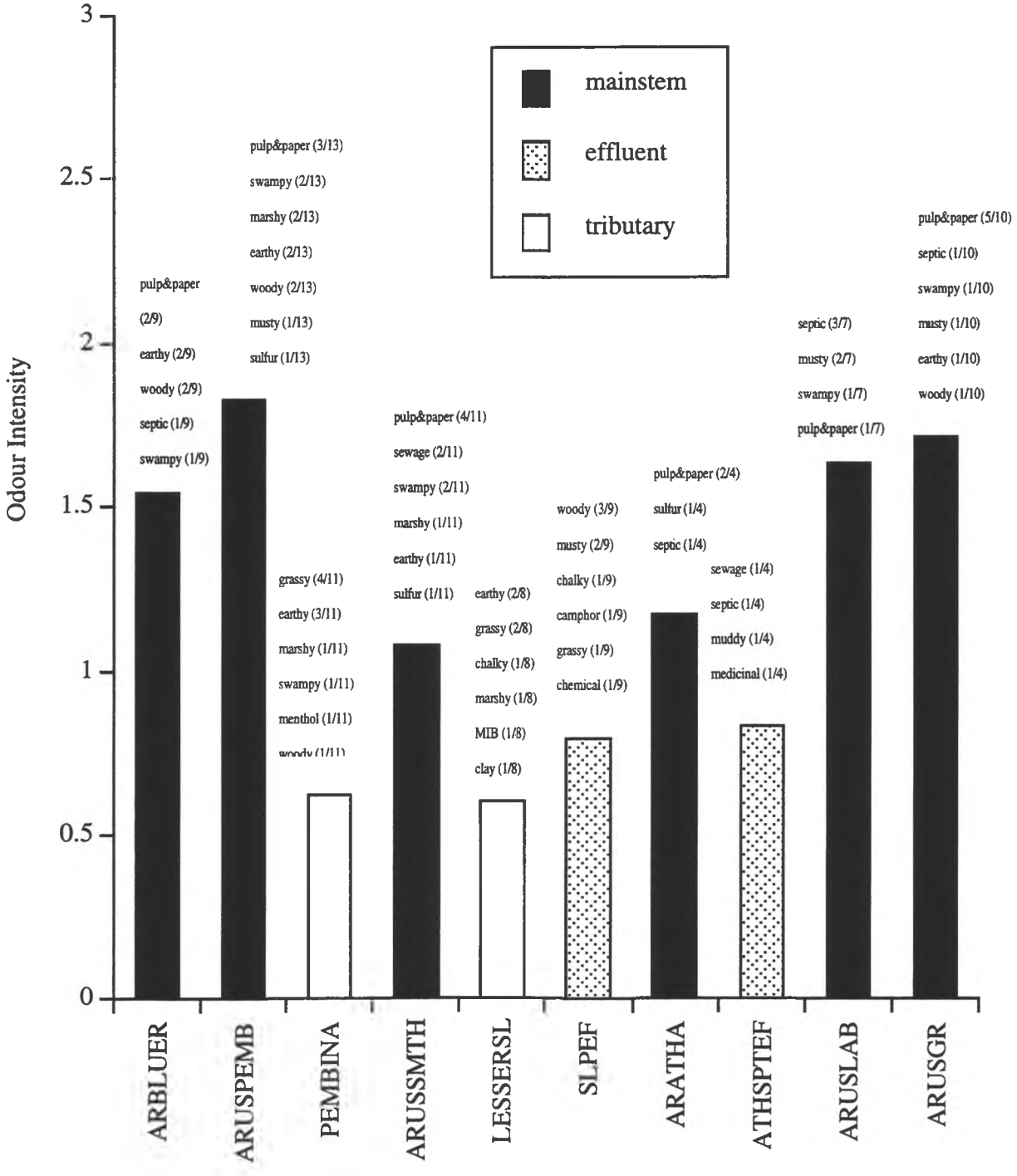

Figure 12. U of A FLAVOUR PROFILE ANALYSES SUMMARY FOR SECOND 10 SAMPLING SITES

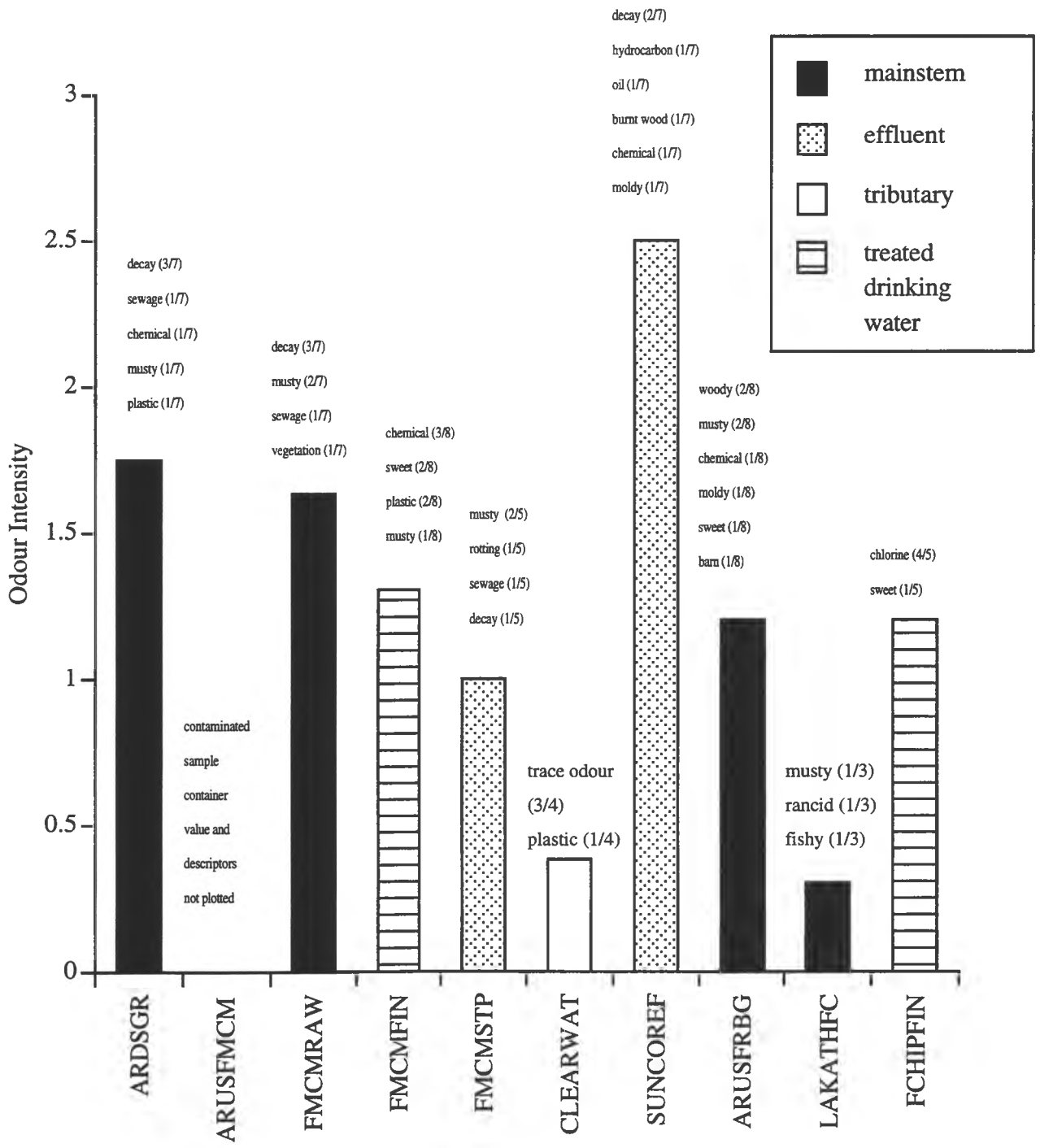

Figure 13. E.L. SMITH FLAVOUR PROFILE ANALYSES SUMMARY FOR THIRD 10 SAMPLING SITES

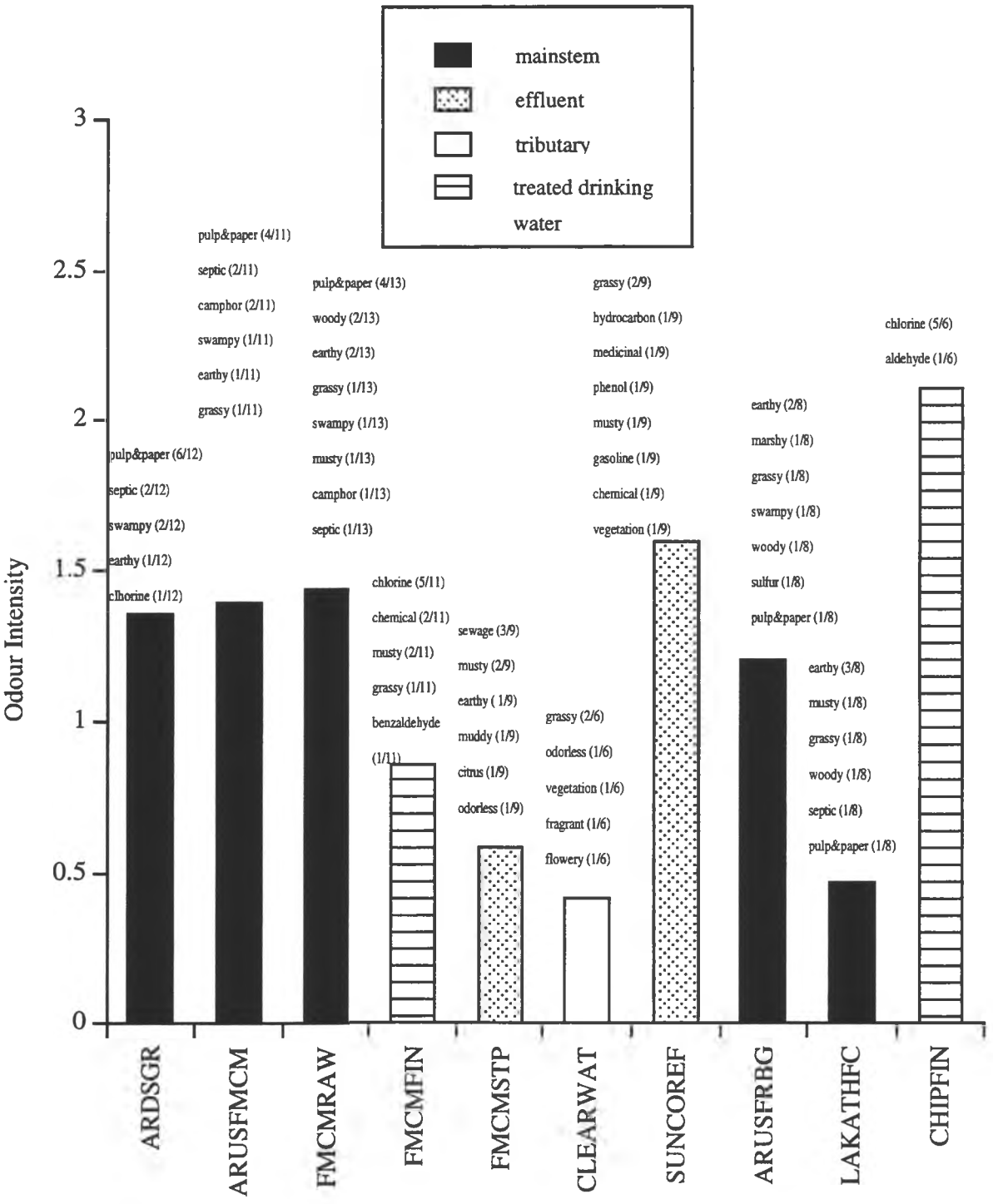

Figure 14. U of A FLAVOUR PROFILE ANALYSES SUMMARY FOR THIRD 10 SAMPLING SITES

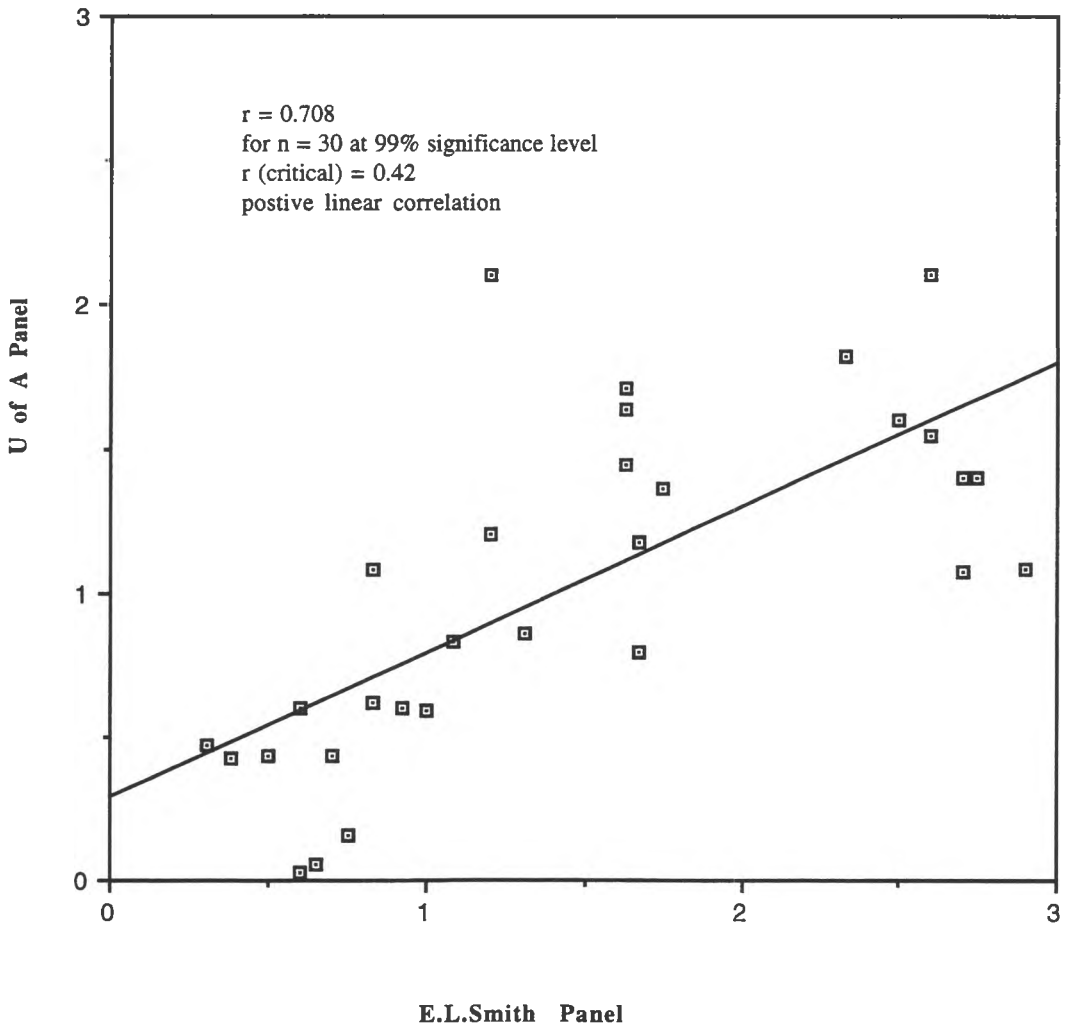

Figure 15. COMPARISON OF FLAVOUR PANEL DATA FOR TWO PANELS

4.0 CONCLUSIONS

This study combined three different analytical methods commonly used in monitoring for the presence of odorous compounds in water supplies. Two flavour profile panels were used to characterize the odour of river samples, two trained analysts evaluated the samples using olfactory GC and all samples were quantitatively analyzed for the presence of target odour compounds using GC/MS. These three techniques all provide quite different information and all have certain limitations.

The flavour profile panel method is most appropriate when monitoring for the presence of compounds that will lead to public complaints. However it is not yet fully standardized, it relies on varying sensitivities to certain odours, and it does not easily allow for reporting the presence of specific compounds. Consistency and specificity of the flavour profile panel results requires rigorous training in the recognition of target compounds and assignment of appropriate intensities. The short lead time available for implementing this study precluded any intensive training of the University of Alberta odour panel, but this panel did provide a general indication of public reactions to odour contributions. In addition, the use of two panels allowed a comparison of FPA results to be documented and validated the new panel's capabilities for future studies.

The olfactory GC technique is useful when there are a number of odorous compounds present in a sample. The GC accomplishes the separation of each of the odour compounds and still allows for olfactory detection. The sensitivity of this method is limited by the dilution of odours by the inert carrier gas as well as the small volumes of sample that can be injected for capillary gas chromatography. The extraction of the samples using CLSA offers a ten thousand fold concentration of the sample so that these sensitivity problems are partially offset.

The analyses by gas chromatography with mass selective detection was the most quantitative analytical method, but also the least sensitive. In order to increase sensitivity of the instrument a selected ion monitoring program was set up to monitor the abundance of certain ions that are known to be present in the mass spectra of the target compounds. Sensitivity is increased because rather than slowly scanning for all possible ions, the detector scans many more times and much more rapidly for the small group of selected ions. However, the analysis is then limited to monitoring for the chosen target compounds. Any odorous non-target compounds, which may significantly contribute to the odour of a sample, will not be reported. The quality control / quality assurance procedures highlighted possible problems related to CLSA artifacts when running samples with very low levels of the target compound levels after a sample with high levels.

The flavour profile panel work, involving two independent panels, clearly shows an impact of Hinton combined effluent on the odour of the Athabasca River for substantial distances downstream. The odour contributions to the Athabasca River from tributaries are minor. The odour contributions from other effluent sources (sewage treatment plants and chemi-thermomechanical pulp mills) are less distinctive than the Hinton combined effluent and their role in affecting downstream odour is not as clear. Notwithstanding these observations, the observed impacts on raw water odour could not be identified for the treated drinking water at Ft. McMurray, possibly because of removal of odorous compounds in treatment and / or masking of the raw water odour with chlorinous odours. The raw water supply at Ft. Chipeweyan was not particularly odorous and the finished water exhibited a very strong chlorine odour that would have masked any subtle odours present. Future samples should have an aliquot dechlorinated prior to FPA analysis so that other odours can be identified.

The CLSA-GC/MS for target compounds also suggests that there was limited contribution of odour compounds from the tributaries. However, with possible exception of geosmin, none of the effluent samples, including the Hinton combined effluent, contributed substantial concentrations of the target odour compounds to the Athabasca River. There were other odorous compounds detected by OGC

immediately downstream of the Hinton combined effluent at AROBED but these were not any of our target odour compounds. Notwithstanding these findings, there was a very distinctive rise in 3,4,5-trichloroveratrole and a measurable, but less distinctive rise in 2,4,6-trichloroanisole in the Athabasca River downstream of Hinton. Because neither of these compounds were present in substantial concentration in the Hinton combined effluent, and their concentrations rise downstream of Hinton, there is not a simple explanation for a possible role of this effluent source in the observed Athabasca River system concentrations for these compounds. In any case, none of the target odour compounds, by themselves can explain the odour character that was perceived by the flavour panel in the Hinton combined effluent and affected downstream samples. However, Figure 8 clearly indicates that the AROBED samples contains many non-target compounds not found upstream of Hinton. The OGC should have provided a separate approach to account for non-target odorous compounds that might explain the odours perceived by the flavour panel. There were very few extra odour peaks detected by OGC (Table 5), with perhaps only the sulfury / septic odour at 10.3 min and the sulfury/mercaptan/crude oil odour at 11.9 min likely to have contributed in any substantial way to the pulp mill odour character. Identifying these compounds would likely assist the odour characterization process, but there are likely other contributing compounds that have not yet been detected by the methods employed in this survey. This possibility suggests the need for a better characterization of the compounds that are primarily responsible for creating the odour of pulp mill effluents.

This report summarizes olfactory GC, CLSA/GC/MS and FPA results, but the three methods are difficult to correlate because:

a) CLSA/SIM is somewhat selective and determines target compound presence only. Unfortunately the current literature base was not sufficient to develop a complete list of compounds responsible for the characteristic "pulp mill" odour.
b) CLSA/OGC detects non-target odour compounds but does not give the overall odour of a sample. However, the variety and intensities of odour peaks gives a semi-quantitative statement about spatial distribution of odours.
c) Flavour panel results give an overall odour intensity for each sample and probably yield the most useful information, but these FPA results can not be compared with chromatographic results.

Unless the target compound list is thoroughly researched and adequately expanded, the three methods are difficult to link and they will remain difficult to correlate.

5.0 RECOMMENDATIONS

The tributary samples collected during this sampling did not contribute significantly to odour compound levels as detected by OGC, FPA, or GC/MS. In order to obtain the most informative data in the most efficient manner we recommend that the post-AlPac study be directed to only the mainstem Athabasca River and effluent samples. Monitoring only these samples for odorous compounds will also allow adequate time for purging of the CLSA between each sample. We also recommend that in the future, finished water samples are to be split into two portions with one portion being dechlorinated to unmask other odour compounds which cannot be detected in the presence of chlorine.

Finally we would recommend that a detailed study of the Hinton combined effluent be undertaken, to isolate and identify the compounds responsible for this odour, so that the set of target odour compounds can be expanded. Large volume extracts that have been stored at National Water Research Institute at the Canada Centre for Inland Waters could be analyzed for the presence of the expanded set of compounds to document pre-AlPac levels of these compounds. This would also allow for quantitative monitoring for the presence of these compounds upstream and downstream from the Alberta Pacific mill after the mill comes on stream in 1993. Furthermore, if successful in identifying appropriate effluent target compounds, these may be used to train the flavour panel to refine their discrimination and intensity rating for these odours.

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APPENDIX A: TERMS OF REFERENCE

NORTHERN RIVER BASINS STUDY

SCHEDULE A - TERMS OF REFERENCE

Project 4411-B1: Water Taste and Odour Study (Athabasca River)

I. Objective

The purpose of this project is to conduct chemical (gas chromatography mass spectometry, gas chromatography electron capture detector and gas chromatography sniff analyses) and flavour panel analyses on water and effluent samples collected from the Athabasca River in February and March 1993 to give a preliminary indication of the extent of water taste and odour problems along the river as well as the types of compounds being discharged into the river that may cause water taste and odour problems.

II. Requirements

1) Undertake a literature review to document the current knowledge about the role of pulp mills and other effluent sources found in the northern river basins in causing off-flavours in water supplies and fish. The literature search will also include a review of analytical methods used to investigate taste and odour problems. While no formal reporting of the literature review is required at this time, the consultant may be asked to prepare an annotated bibliography and synthesis report on water and fish taste and odour problems under a separate contract. It is therefore assumed that information gained during the literature review for this project would serve as important background information for subsequent preparation of an annotated bibliography and/or synthesis report.

2) Conduct chemical evaluations of water and effluent samples from the Athabasca River using large volume solvent extractions and closed loop stripping extraction combined with gas chromatography mass spectometry, gas chromatography electron capture detector and gas chromatography sniff analyses. Water samples for these analyses will be collected from the Athabasca River at the time-of-travel of the river in February and March 1993. The water and effluent samples will be collected by Alberta Environmental Protection on behalf of the Northern River Basins Study. Water and effluent sampling from the Athabasca River will be carried out in a manner specified by the consultant to Alberta Environmental Protection/Northern River Basins Study.

3) Conduct a flavour profile analysis of water and effluent samples collected from the Athabasca River in February 1993. The flavour profile analysis is to be conducted by a pre-established, trained sensory panel.

III. Reporting Requirements

1) Prepare a draft report outlining the results of the chemical and flavour profile analyses of water and effluent samples. The report should include a detailed description of the methodology employed, quality assurance/quality control measures and a comprehensive discussion of the results of the chemical and flavour profile analyses. Ten copies of the draft report are to be submitted to the Project Liaison Officer (Greg Wagner, Office of the Science Director, Northern River Basins Study, phone (403) 427-1742, fax (403) 422-3055) by March 22, 1993.

2) Three weeks after the receipt of review comments, submit ten cerlox bound copies and two unbound, camera-ready originals of the final report to the Project Liaison Officer. The final report is to include an executive summary, table of contents, list of tables (if appropriate), list of figures (if appropriate) and an appendix which includes the Terms of Reference for this project. An electronic copy of the report, in Word Perfect 5.1 format, is to be submitted to the Project Liaison Officer at the same time as the final report. Data presented in tables, figures, appendices, etc. in the final report are also to be compiled in an electronic database (dBase IV preferred) and submitted to the Project Liaison Officer. All sampling locations presented in the report and in electronic form are to be geo-referenced (lat./long. preferred).