

**ANALYSIS OF TASTE AND ODOUR COMPOUNDS  
IN THE EDMONTON WATER SUPPLY, MARCH, 1986**

by

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## MANAGEMENT PERSPECTIVE

The North Saskatchewan river is the water supply for the City of Edmonton. The City has two water treatment plants which serve a total population of 700,000. Episodes of objectionable taste and odour occur regularly in the spring. Until recently, the approach has been to treat the symptom rather than understand the cause. Partly in response to public concern over the quality of their drinking water, the City commissioned Prof. Steve Hrudey at the University of Alberta to design a detailed study of drinking water quality, including taste and odour.

In early March, 1986, during a period of unusually warm weather, the city water supply developed an odour caused by upstream runoff. The University of Alberta provided several samples to analyze for geosmin and 2-methylisoborneol. We have been studying taste and odour problems caused by these two naturally-occurring substances, and have specialized in analyzing and identifying them. This report contains the results of our analysis of the Edmonton samples. Geosmin was tentatively identified in several of the samples near the threshold odour concentration. The presence of 2-methylisoborneol could not be confirmed or ruled out.

## PERSPECTIVES GESTION

La ville d'Edmonton s'approvisionne en eau dans la rivière Saskatchewan-Nord. La Ville possède deux usines de traitement des eaux, qui desservent une population totale de 700 000 habitants. Au printemps, il arrive régulièrement que l'eau prenne une saveur et une odeur désagréables. Jusqu'à récemment, on cherchait surtout à traiter les symptômes plutôt qu'à en comprendre la cause. C'est en partie à la suite des inquiétudes exprimées par le public au sujet de la qualité de l'eau potable que la Ville a chargé le professeur Steve Hrudey (Université de l'Alberta) d'élaborer une étude détaillée de la qualité de l'eau potable, y compris de sa saveur et de son odeur.

Au début du mois de mars 1986, pendant une période de chaleur inhabituelle, l'eau a pris une odeur particulière, due à des eaux de ruissellement se déversant en amont de la prise d'eau. L'Université de l'Alberta nous a fourni des échantillons en nous demandant d'y rechercher la géosmine et le 2-méthylisobornéol. Nous étudions depuis un certain temps les problèmes de saveur et d'odeur causés par ces deux substances naturelles et nous nous spécialisons dans leur dosage et leur identification. Le présent rapport renferme les résultats de l'analyse des échantillons de l'eau d'Edmonton. La géosmine a été provisoirement identifiée dans plusieurs des échantillons à une concentration voisine du seuil de perception de l'odeur. Il n'a pas été possible de confirmer ou d'infirmer la présence du 2-méthylisobornéol.

## Abstract

During early March, 1986, the Edmonton water supply developed an objectionable odour due to upstream runoff. This odour was subjectively described as "grassy" or "camphor-like," suggesting that it was caused by geosmin or 2-methylisoborneol. The University of Alberta provided us with samples to analyze for these two compounds. Geosmin was tentatively identified in several of these samples. Samples of raw and finished water collected March 7 at the E. L. Smith and Rosedale treatment plants contained 0.01-0.03  $\mu\text{g/L}$  of geosmin, similar to the threshold odour concentration of 0.02  $\mu\text{g/L}$  for geosmin. The presence of 2-methylisoborneol was suspected on the basis of the "camphor-like" odour reported in the water and observed in the hexane extracts. However, we were unable to confirm that 2-methylisoborneol was in the samples. The hexane extracts had complex odours, suggesting that a variety of odiferous compounds was present in the water. Attempts to identify these substances by gas chromatography-mass spectrometry were unsuccessful.

## Résumé

Au début du mois de mars 1986, l'eau d'approvisionnement de la ville d'Edmonton a pris une odeur désagréable, due à des eaux de ruissellement se déversant en amont de la prise d'eau. Cette odeur a été subjectivement décrite comme une odeur "d'herbe" ou de "camphre", ce qui a donné à penser qu'elle était due à la présence de géosmine ou de 2-méthylisobornéol. L'Université de l'Alberta nous a fourni des échantillons de cette eau en nous demandant d'y rechercher ces deux composés. La géosmine a été provisoirement identifiée dans plusieurs de ces échantillons. Les échantillons d'eau brute et d'eau traitée prélevés le 7 mars aux usines de traitement E.L. Smith et Rosedale présentaient une concentration de géosmine de 0,01-0,03  $\mu\text{g/L}$ , analogue au seuil de perception de l'odeur qui est de 0,02  $\mu\text{g/L}$  pour la géosmine. On avait soupçonné la présence de 2-méthylisobornéol à cause de l'odeur "camphrée" qui avait été signalée et qu'on a aussi décelée dans des extraits à l'hexane. Nous avons toutefois été incapables de confirmer la présence du 2-méthylisobornéol dans les échantillons fournis. Les extraits à l'hexane dégageaient des odeurs complexes qui semblaient indiquer la présence de divers composés odorants dans l'eau. Les tentatives visant à identifier ces substances par chromatographie en phase gazeuse couplée à la spectrométrie de masse se sont révélées infructueuses.

## Introduction

Taste and odour episodes in the Edmonton water supply are a common problem in the spring (Walker et al 1986). A comprehensive study of this problem is being carried out for the City of Edmonton by Prof. Steve Hrudey at the University of Alberta. During March, 1986, the city water supply had a strong odour subjectively described as "camphor-like" or "grassy." This suggested the presence of 2-methylisoborneol (MIB) and/or geosmin in the water. These are naturally-occurring, moderately volatile, organic compounds with threshold odour concentrations (TOC) of 0.01-0.02  $\mu\text{g/L}$ . We have been studying the occurrence of these compounds in Canadian water supplies and the taste and odour problems which result from their presence. The situation in Edmonton is of special interest because of the large population affected and because the problem occurs in late winter/early spring. Geosmin production is usually associated with mid-summer blooms of blue-green algae in lakes. Because of our interest in this subject, we welcomed the opportunity to work with Prof. Hrudey on this problem.

Raw and finished water samples (2 L) collected March 7 and 8 at the E. L. Smith and Rosedale water treatment plants were sent to us for analysis. The results indicated low levels of geosmin in these samples, but the concentrations were too low to attempt confirmation by gas chromatography-mass spectrometry (gc-ms).

In addition, the odours were complex indicating that a variety of odour-causing compounds was present in the water. For further work on identification of compounds, a larger sample (16 L) of Rosssdale raw water (March 4) was processed.

### Methods

Samples were shipped from Edmonton by air and kept cool until processing. Just before extraction, they were warmed to room temperature and the odour noted (Table 1). The 2 L samples were extracted by stirring with hexane in a volumetric flask (Brownlee et al. 1984). The extracts were dried, an internal standard (octadecane) added, and the extracts concentrated to 0.1-0.2 mL under a stream of argon using isooctane as the "keeper" solvent. The extracts were then analyzed by gas chromatography on a 30 m DB-17 fused silica capillary column programmed from 80-260 C at 4 deg/min in a Hewlett-Packard 5710 chromatograph using a flame ionization detector. Peak areas were measured with a Hewlett-Packard 3392 integrator. The odour of the hexane-extractable material was determined by evaporating a 1-2  $\mu$ L aliquot on a tissue. The odour descriptions and quantitative results are in Table 1.

The large volume sample was extracted in 2 L portions by the same method and the extracts were combined. These were used for

qualitative work only. The retention time of geosmin was compared with authentic material on three different columns (Table 2) by co-injecting sample and standard. This sample was also analyzed by gc-ms in the Water Quality National Laboratory, Burlington.

### Results

The subjective odour evaluations for the March 7 and 8 samples from both water treatment plants are given in Table 1. The water samples did not have strong or distinctive odours. However, the hexane extracts had strong and complex odours such as swampy, camphor, mildew, or hay. Gas chromatography of these extracts showed numerous small peaks with a few larger ones (Fig. 1). Several chromatograms contained peaks with retention times corresponding to geosmin. The detection limit was about 0.01 µg/L. Measurable concentrations of 0.01-0.03 µg/L were found in the March 7 samples and one March 8 sample (Table 1). These concentrations are near the threshold odour concentration of about 0.02 µg/L, so at least some of the odour could have been due to geosmin.

Several extracts had a camphor odour and the chromatograms had a peak with retention time very similar to, but not identical with, that of MIB. On the basis of this, we can only say that MIB might have been present in the samples. If this peak was due to

MIB, then the concentrations were similar to those found for geosmin.

After these results were obtained, it was clear that a larger sample was needed for qualitative analysis for two reasons: 1) to (attempt to) confirm the identification of geosmin and MIB, and 2) to try to identify other compounds responsible for some of the other odours. The large sample of Rosedale raw water from March 4 was analyzed by gc-ms in the Water Quality National Laboratory. Both full-scan and multiple ion detection (MID) methods were used. The results were not conclusive. MIB could not be confirmed by either method. MID showed ions for the base peak ( $m/z$  112) and molecular ion ( $m/z$  182) with the correct retention time for geosmin. Full scan runs failed to produce a clean spectrum for geosmin due to the complex background in the sample. However, a spectrum was obtained at the expected retention time which had ions at  $m/z$  112 and 182.

Because we were unable to obtain a clean spectrum for geosmin, we resorted to retention time comparison by gc using columns of differing polarity. Three columns were used: non-polar (DB-1), moderately polar (DB-17), and polar (Supelcowax 10). The results are shown in Table 2 and Figs. 2 to 4. In all three cases, the peak assigned to geosmin was enhanced by co-injection with authentic standard. A peak with retention time corresponding to MIB was present in each case. These results and

the gc-ms results can be explained in two ways: 1) MIB is present, but the concentrations are too low to be detected by gc-ms or it is masked by closely eluting compounds; 2) a compound with properties very similar to MIB is present.

### Summary

Geosmin has been tentatively, but not positively, identified in raw and finished water samples from the E. L. Smith and Rosssdale water treatment plants. Concentrations on March 7, 1986, were near the threshold odour concentration. From the complex odours observed in hexane extracts of these water samples, it is apparent that the odour is not due to geosmin alone, but to a variety of substances. The presence of 2-methylisoborneol in these samples could neither be confirmed nor ruled out.

### Acknowledgements

I would like to thank John Carron of the Water Quality National Laboratory for doing the gas chromatography-mass spectrometry on these samples. The geosmin standard was provided by the Drinking Water Research Division of the United States Environmental Protection Agency, Cincinnati, Ohio.

### References

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Table 1. Results for E. L. Smith (ELS) and Rosssdale (RD) raw water (RW) and finished water (FW) collected March 7 and 8, 1986.

Sample	Water Odour	Hexane Extract Odour	Geosmin Conc. µg/L.
Mar. 7			
ELS-RW	faint earthy	swampy camphor	0.01
ELS-FW	nearly odourless	camphor	0.02
RD-RW	nearly odourless	mildew, hay camphor	0.03
RD-FW	chlorinous	complex	0.03
Mar. 8			
ELS-RW	sulfurous	swampy	ND
ELS-FW	chlorinous	mildew	ND
RD-RW	faint earthy	swampy mildew	ND
RD-FW	weak chlorinous	swampy camphor geosmin	0.01

ND Not detected; below the detection limit of about 0.01 µg/L.

Gas chromatography conditions: 30 m by 0.25 mm id DB-17 column programmed from 80-260 C at 4 deg/min; He carrier at 0.8 mL/min; injector 250 C; flame ionization detector 300 C.

Table 2. Retention time comparisons on three columns for the hexane extract of March 4 Rosedale raw water against MIB and geosmin standards.

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Column	"MIB" Ret. Time, min		"Geosmin" Ret. Time, min	
	Sample	Sample+Std	Sample	Sample+Std
DB-1	9.27	9.28	15.82	15.80
DB-17	10.72	10.76	18.48	18.49
Supelcowax 10	12.90	12.88 *	19.30	19.30

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Sample+Std runs were co-injections of sample and standard.

\* This retention time is for the standard only.

Gas chromatography conditions: columns were fused silica capillary columns 30 m by 0.25 mm id; He carrier flow 0.8 mL/min; initial temperature 80 C; final temperature 260 C for DB-1 and DB-17, 240 C for Supelcowax 10; temperature program rate 4 deg/min; injector temperature 250 C; flame ionization detector temperature 300 C for DB-1 and DB-17, 250 C for Supelcowax 10; instrument Hewlett-Packard 5710 chromatograph with 3392 integrator.

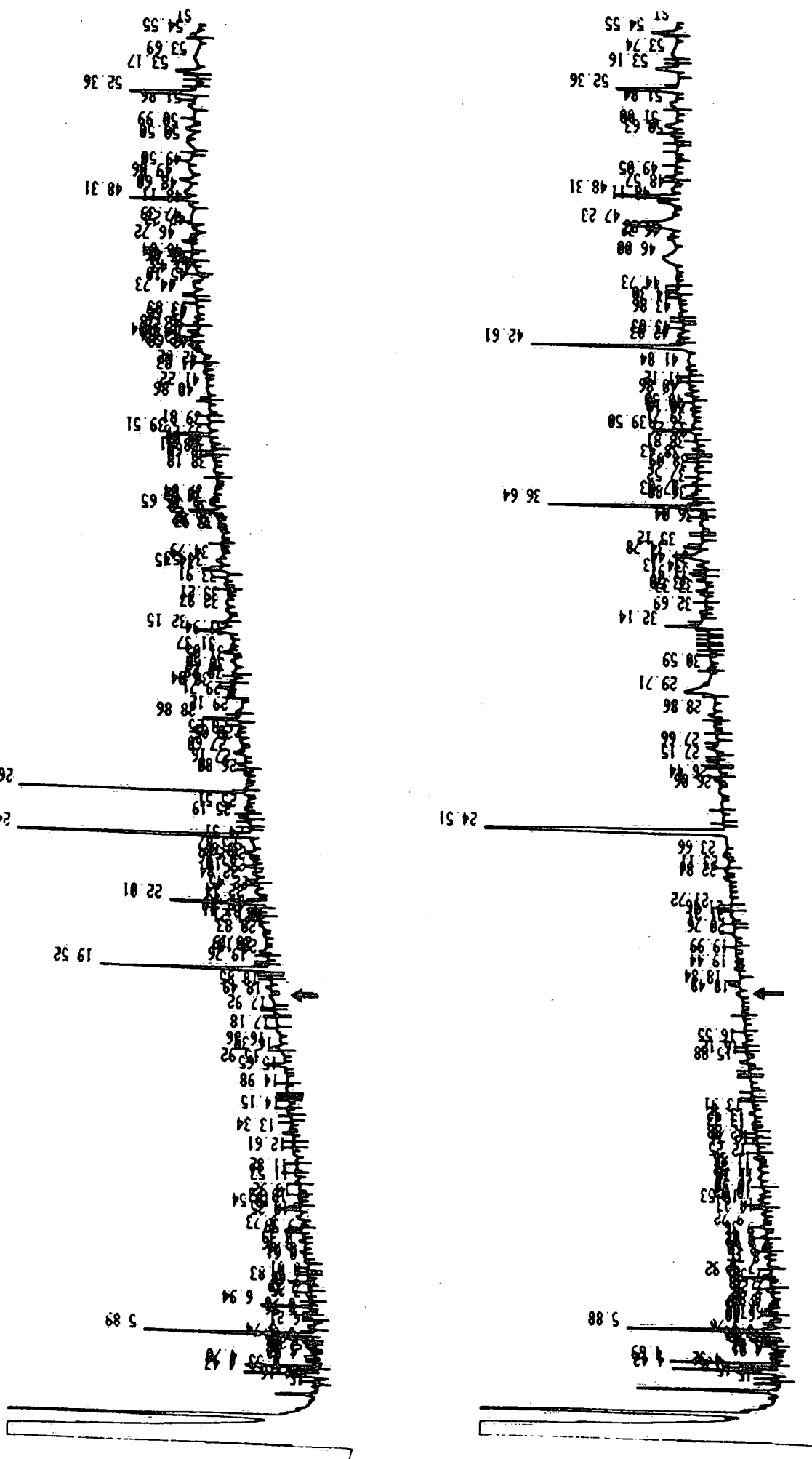


Fig. 1. Gas chromatograms of hexane extracts of E. L. Smith finished water (top) and Rossdale finished water (bottom) collected March 7, 1986, and run on a 30 m DB-17 column. The geosmin peaks are noted with an arrow (↑), and noise spikes with a star (★).

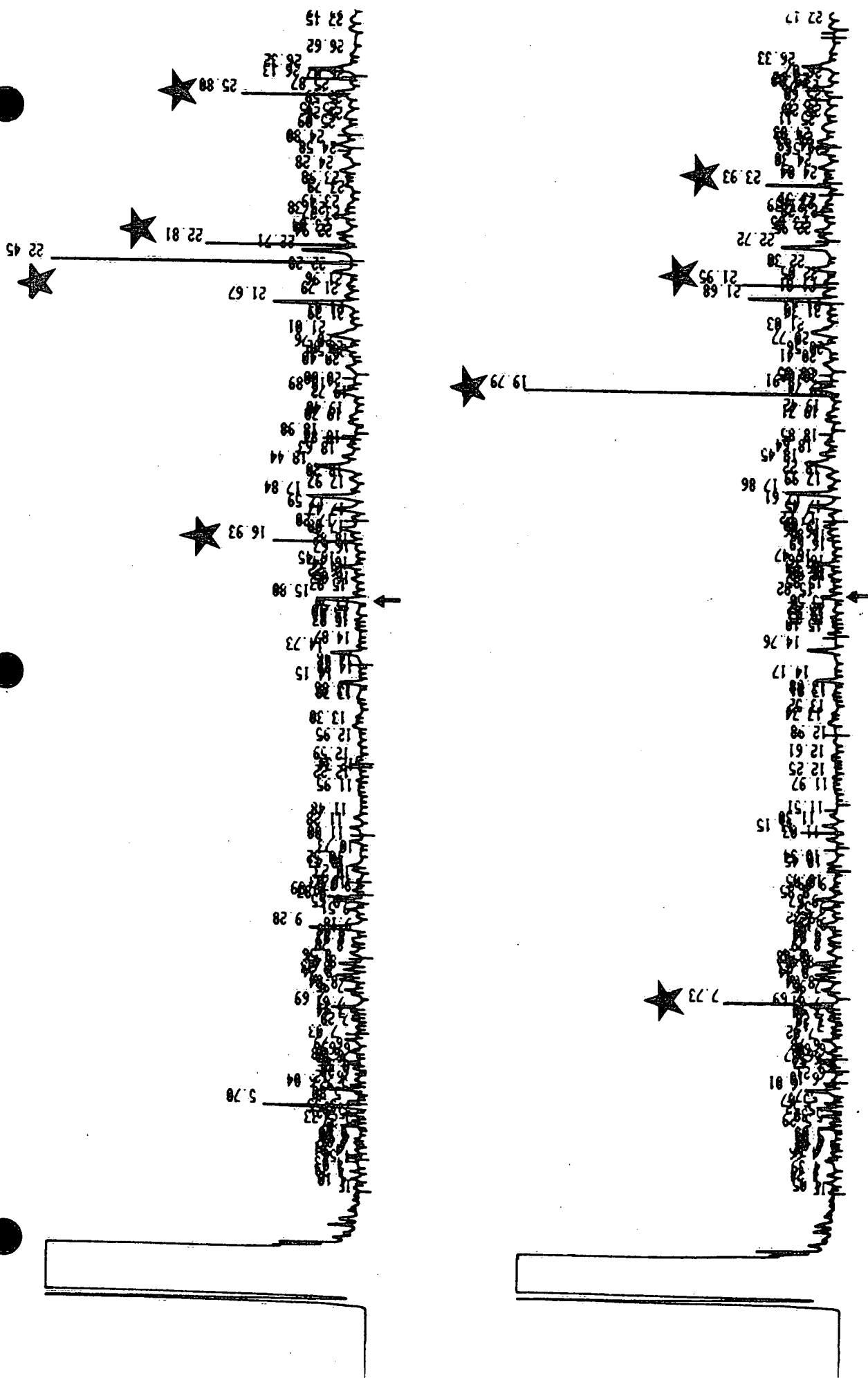


Fig. 2. Gas chromatograms of the hexane extract of Rossdale raw water collected March 4, 1986, with (top) and without (bottom) co-injection with geosmin standard on a 30 m DB-1 column. The geosmin peaks are noted with an arrow (↑), and noise spikes with a star (★).

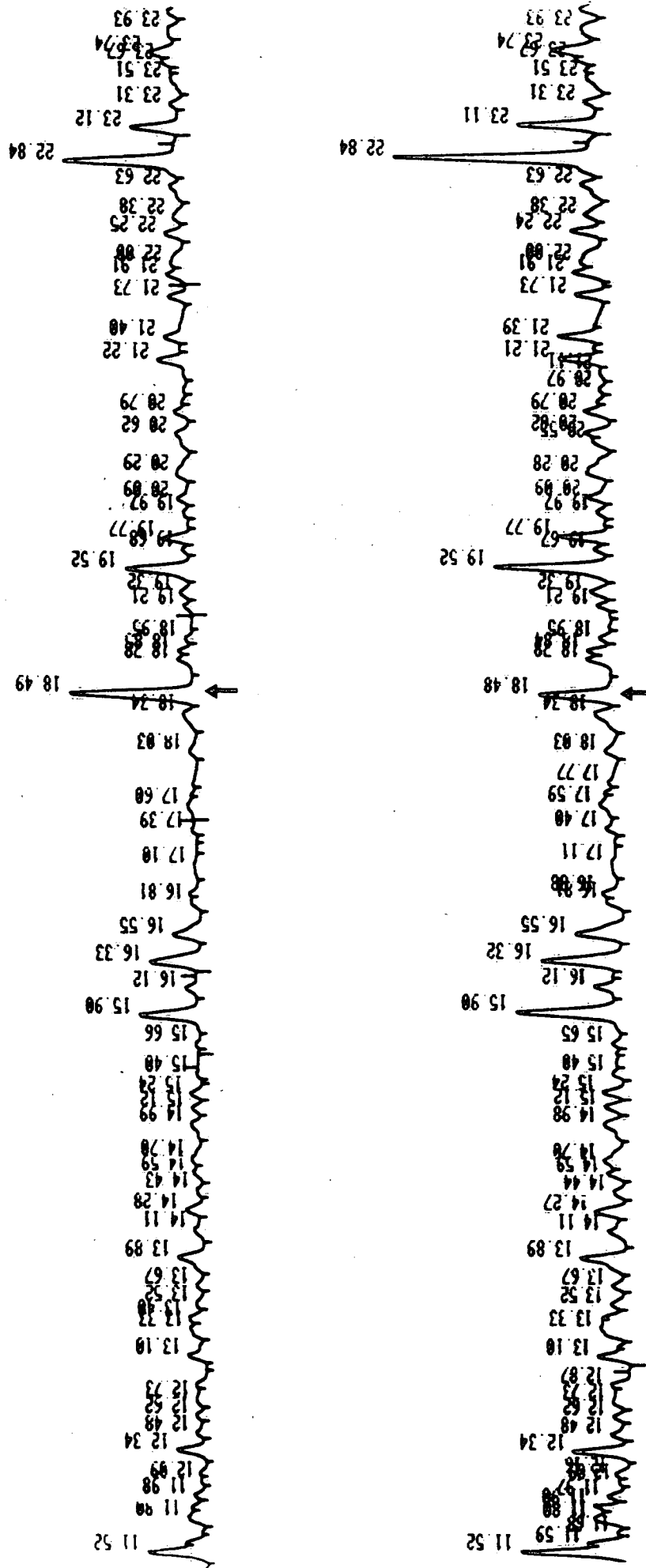


Fig. 3. Gas chromatograms of the hexane extract of Rossdale raw water collected March 4, 1986, with (top) and without (bottom) co-injection with geosmin standard on a 30 m DB-17 column. Geosmin peaks are noted with an arrow (↑).

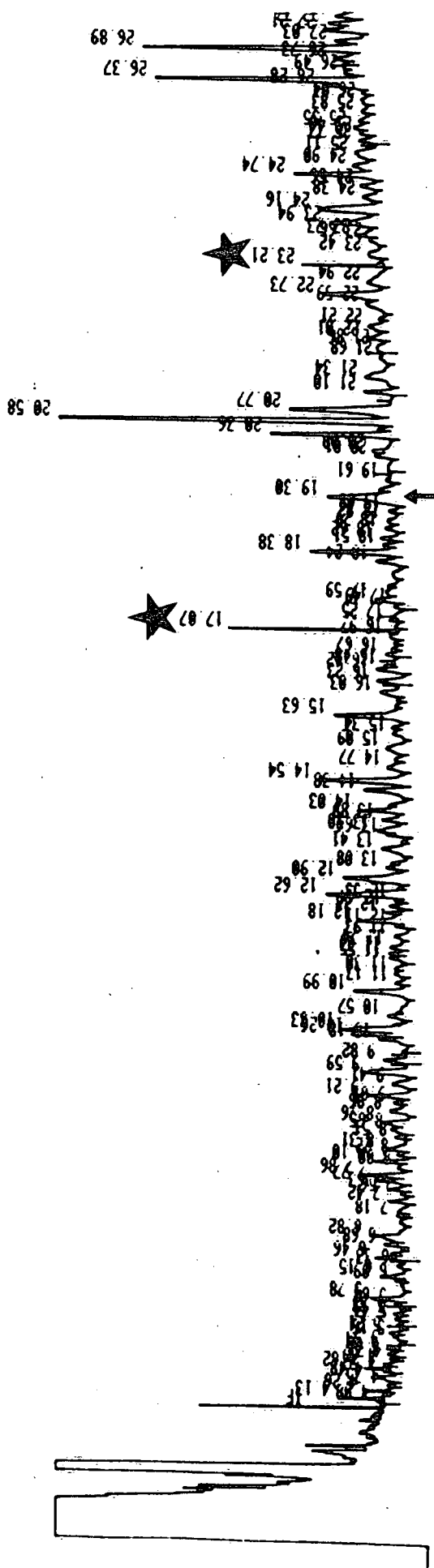
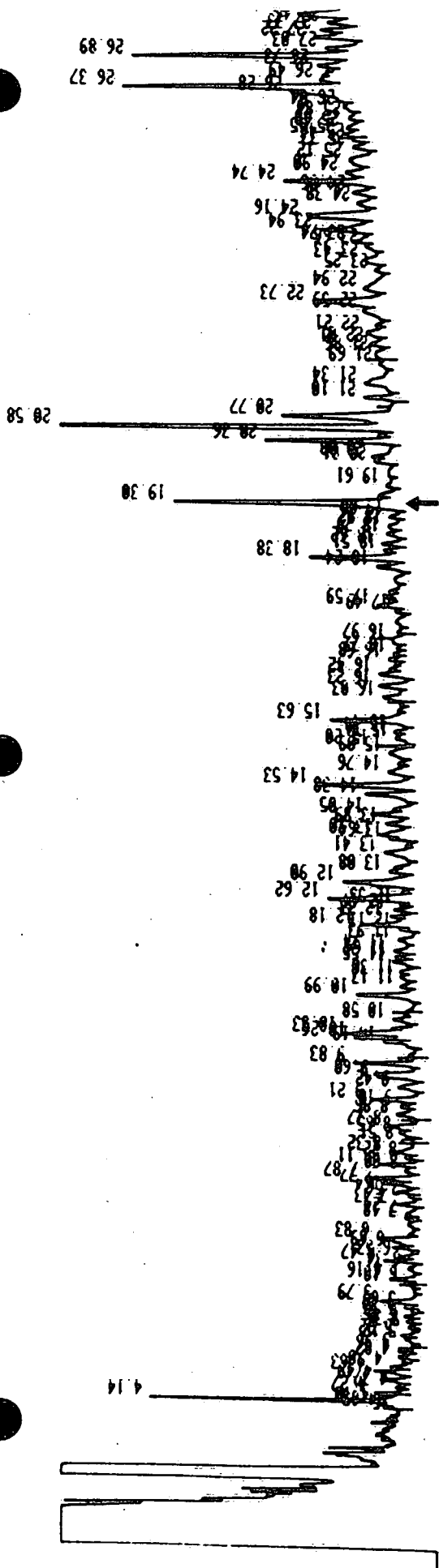
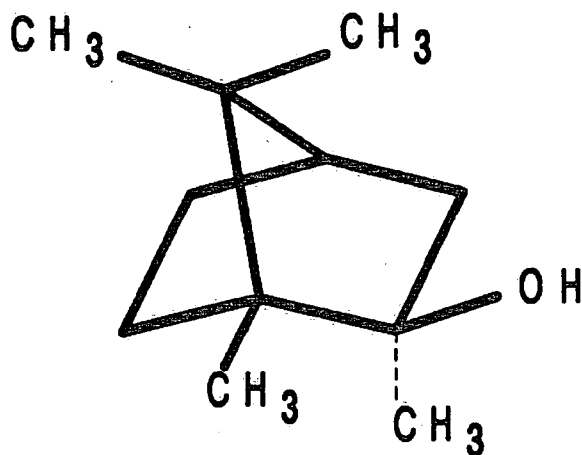


Fig. 4. Gas chromatograms of the hexane extract of Rosedale raw water collected March 4, 1986, with (top) and without (bottom) co-injection with geosmin standard on a 30 m Supelcowax 10 column. Geosmin peaks are noted with an arrow (↑), and noise spikes with a star (★).

## Appendix

The systematic names, and molecular and structural formulae for MIB and geosmin.

MIB is 1,2,7,7-tetramethyl-exo-bicyclo[2.2.1]heptan-2-ol,  $C_{11}H_{20}O$ , mol wt 168.



Geosmin is trans,trans-1,10-dimethyl-9-decalol,  $C_{12}H_{22}O$ , mol wt 182.

