8

UNDESIRABLE ODOR AND TASTE EVENTS IN BOVILLA DRINKING WATER, RELATION TO LAKE LIMNOLOGY AND PRELIMINARY ANALYSIS

DUKURIA E ERËS DHE SHIJES SË PAPËLQYESHME NË UJIN E PIJSHËM TË BOVILLËS, MBËSHTETUR NË LIMNOLOGJINË E LIQENIT DHE ANALIZAT KIMIKE PARAPRAKE

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Përmbledhje

Çdo vit duke filluar nga viti 2001 në periudhën vjeshtë deri pranverë në ujin e pijshëm të Bovillës ka pasur shqetësime në lidhje me erën dhe shijen e papëlqyeshme, në disa raste mjaft e theksuar. Për të mënjanuar këtë dukuri, Impianti i Trajtimit të Ujit, Kodra e Kuqe, Tiranë, është detyruar të përdorë trajtimin e ujit me qymyr aktiv, në këtë mënyrë, duke rritur mjaft koston e prodhimit.

Shkaqet e këtij problemi janë objekt i një debati të gjerë në literaturën shkencore. Sot pranohet se shkaqet kryesore mund të jenë produktet e dekompozimit të bimësisë në liqen dhe në pellgun ujëmbledhës si dhe disa metabolite të llojeve të veçanta të fitoplanktonit ose aktinobaktereve aerobe fijezore (Streptomyces).

Ka shumë lëndë organike flurore (VOC) që shkaktojnë probleme të erës dhe shijes në ujin e pijshëm; ndër më të zakonshmet janë dy terpene, Geosmina dhe 2-MIB. Organizmat që kanë lidhje me këto lëndë janë disa baktere të tokës (*Actinomycetes*) dhe disa lloje të algave blu- të blerta (*Cyanobacteria*).

Në kuadrin e projektit të përbashkët kërkimor SCOPES 2005-2008 (nr. IB7320-111032), për studimin e limnologjisë së liqenit të Bovillës, ne u ndalëm në këtë problem duke pasur si qëllim:

(i) Studimin e lidhjeve të mundshme ndërmjet dukurisë së erës dhe shijes dhe parametrave limnologjikë të liqenit, të cilët mund të shërbejnë për parashikimin e kësaj dukurie.

(ii) Hetimin paraprak të përmbajtjes së lëndëve flurore në ujin e Bovillës për të arritur, nëpërmjet një studimi të mëtejshëm, në identifikimin e lëndëve që shkaktojnë këtë dukuri.

Nga studimi limniologjik i liqenit kemi arritur në përfundimin se dukuria e erës dhe shijes ndodh në periudhën e prishjes së shtresëzimit të liqenit (Fig. 8-1). Gjithashtu, janë gjetur korrelime të rëndësishme ndërmjet zhvillimit të kësaj dukurie dhe ndryshimeve të disa parametrave kimikë, si turbullisë së ujit, sasisë së reshjeve, pH-it, përmbajtjes së fosfateve dhe përqendrimit të hekurit, korrelime që shihen qartë në figurat 2, 3, 4 dhe 5. Shfaqja e erës dhe shijes në periudhën e temperaturave relativisht të ulëta (dhe me reshje të shumta) dhe përmbajtja shumë e ulët e

biomasës së fitoplanktonit gjatë kësaj periudhe tregojnë se ka shumë mundësi që burimi i lëndëve organike flurore (VOC) të jenë kryesisht bakteret e tokës (actinomicetes).

Për të pasur informacion të përgjithshëm mbi përmbajtjen e lëndëve VOC në ujin e Bovillës, u krye analiza kimike e dy mostrave kompozite uji, që përfaqësonin thellësinë 0-10 m, të marra në janar 2008, kur liqeni ishte në gjendje të përzierjes së plotë (*holomixis*), dhe uji paraqiste erë dhe shije karakteristike. Ekstraktimi me diklormetan i lëndëve VOC u bë në Laboratorin e Kimisë Analitike, Universiteti i Tiranës, kurse analiza kimike e ekstrakteve me gaz-kromatograf / masë-spektrometër (GC-MS), u krye në Stacionin Limnologjik, Universiteti i Zyrihut (Zvicër), nga Prof. F. Juttner. Si mund të shihet edhe në figurën 8-6, në mostër përmbahej një numër shumë i madh i lëndëve flurore. Nga identifikimi i disa prej tyre u vu re se mostra përmbante mjaft alkoole, aldehide dhe terpene, si p.sh. borneol, kamfor, karvon, norpinin, pinokarvon, propilcikloheksanon, α-terpinol, verbenon, butil-4-metilciklopenten etj. Ka mundësi që karvoni të ishte nga përbërësit kryesorë që shkaktonte erën dhe shijen (Fig. 8-7).

Rezultatet e këtij studimi duhet të konsiderohen paraprake, meqenëse kërkohen studime të mëtejshme më të plota për këtë dukuri.

Keywords: Bovilla reservoir (Albania), drinking water, VOCs

8.1. Introduction

Undesirable odors in drinking water are a global problem; they have been observed worldwide in many countries. Seasonal unpleasant taste and smell problems are possibly the single greatest public relation issue many water utilities face in many countries. This is embarrassing for waterworks because consumers rely on the taste and odor of their drinking water as the primary indicator of its safety (Dietrich, 2006; Ortenberg & Telsch, 2003; Suffet *et al.,* 2004). Because the extremely low detection limit of men the earthy-musty odors in drinking water are a particular nuisance for consumers.

The causes of this problem have been debated broadly in the scientific literature. The decay of vegetation in and around drinking water reservoirs was discussed as a major contributor, as well as the production by specific phytoplankton organisms. From data collected over nearly a century, it seems that both the decay of plants remains as well as release of certain metabolites and storage products of living microbiota contribute to tastes and odors in water supplies (Ortenberg & Telsch, 2003; Sigworth, 1957; Wood *et al*., 2001).

Many studies have identified various biogenic taste and odor compounds in water. The majority of all biologically caused taste-and-odor outbreaks in drinking water are due to two volatile organic compounds (VOC) terpenes, geosmin (1*a*, 10*b*-dimethyl-9*a*-decanol) and 2-MIB (2-methylisoborneol) (Jüttner, 2007; Wood *et al.,* 2001). Both are earthy-muddy-smelling metabolites, which present an extremely low threshold of perception as humans are able to detect levels down to 4-10 ng/L (Taylor *et al*., 2006). Geosmin has an earthy odor, which can be described as dirt, corn silk, and beet (Omür-Ozbek & Dietrich, 2005), whereas 2-MIB has a typical musty-muddy and camphor-like taste and odor (Lanciotti *et al.,* 2003; Suffet *et al*., 2004). Geosmin and 2-MIB are tertiary alcohols, both exist as (+) and (-) enantiomers. Odor outbreaks are caused by the biological production of the naturally occurring (-) enantiomers, which are some 10 times more potent in odor than the (+) molecules (Jüttner, 2007).

Organisms most frequently linked to odor problems have been the *Actinomycetes* (*Streptomycetes*) and several genera of the blue-green algae (Cyanobacteria). The most frequently documented taste and odor producing blue-green algae include the genera *Oscillatoria* spp*., Aphanizomenon* spp*., Anabaena* spp*.,* and *Microcystis* spp*.* (Lange & Wittmeyer, 2005; Wood *et al.,* 2001). These cyanobacteria may cause taste and odor problems in two ways: Firstly, the organisms excrete these compounds or they are released upon lysis (Ozaki *et al*., 2008). Secondly, while decaying nitrogen is liberated which will support bacterial growth, especially *Actinomycetes* as the second group of organisms producing these taste and odor compounds (Schöller *et al.,* 2002; Schulz & Dickschat, 2007).

The Treatment Plant of Bovilla that supply Tirana city with drinking water, since 2001 they have been facing taste and odor problems usually during fall and winter time up to early spring, often quite severe. The nuisance could only be removed by using advanced and costly treatments through adsorption on activated carbon or with ozone (Srinivasan *et al*., 2008).

The aim of our work was:

1. To study the basic limnology of Bovilla reservoir to find possible correlations between taste and smell events and the water quality variables that potentially could predict the occurrence of such events.

2. To get preliminary information on volatile compounds in Bovilla water for a future project to identify the volatile compound(s) in the water responsible for bad smell and taste.

8.2. Water quality parameters correlated to the occurrence of noxious odor events

Because the high costs to remove noxious taste and odor compounds from the raw water, it is not appropriate to permanently treat the water with active carbon. Instead, predictive tools are needed that allow the water treatment operators to determine when taste and odor events are most likely to occur (Izydorczyk *et al.,* 2005; Dzialowski *et al.,* 2007). Because humans detect many taste and odor compounds at very low concentrations (5-10 ng/L; Taylor *et al.,* 2006), there is only a short time window in which a developing taste and odor event can be treated before customer complaints are received. One approach to manage taste and odor events would simply be to measure the concentrations of geosmin or other malodorous compounds on a regular

basis. However, these analyses require a great deal of technical training and equipment, and sending samples to a contract laboratory can be very expensive. Additionally, the results are often not received in a time that would allow for a decision to treat the raw water from the drinking water reservoir. Alternatively, there may be correlations between concentrations of malodorous compound(s) and water quality variables that are easier and more cost effective to control. Such potential predictor variables may include nutrient concentrations (particularly change in phosphate levels), water turbidity, water temperature, pH, content of chlorophylls and the food web structure. These predictor variable(s) could then be used to estimate odor compound concentrations and ultimately influence the decision when to start to treat the drinking water (Taylor, 2006).

Comparing our data obtained during the two years monitoring with periods of odor events in drinking water from Bovilla Lake (Çullaj *et al*., Nr. 2 *in this edition*), we found that there is an obvious relation between the unpleasant smell and taste periods and the stratification situation: smell starts when stratification begins to weaken, and it ends after the overturn when stratification is again establishing. Hence, the periods of a treatment of water with active carbon for the two monitoring years were: 6/10/2006 to 24/04/2007 and 17/09/2007 to 30/04/2008, both phases coincide with the overturn periods in Bovilla Lake. It is clearly seen in the changes of water temperature in three layers 1-5 m, 10-15 m, and 15 m-bottom during two year period (Fig. 8-1).

Maximum values of turbidity and rainfall (Fig. 8-2), a gradual increase of the pH (Fig. 8-3) and maximum values of phosphate (Fig. 8-4) and iron concentrations (Fig. 8-5) have been observed during the smell and taste periods. Similar relations have been realized also in former years, although no detailed study was carried out.

Furthermore, odor periods in the water of Bovilla Lake occur in the fall and winter period, when the temperature of the water is much lower than during the rest of the year. We observed that there was a very low phytoplankton biomass during this period. However, from May to September 2007 a peak of phytoplankton biomass was observed dominated by diatoms, mainly by *Cyclotella* sp., and by dinoflagellates (Koni *et. al*, Nr. 3 *in this volume*).

As mentioned in the literature (Lanciotti *et al.,* 2003), the presence of odor during the winter, contradicts that cyanobacteria are the only source of odors in freshwaters and indicates that actinomycetes, possibly in association with microalgae, were the major odor producers.

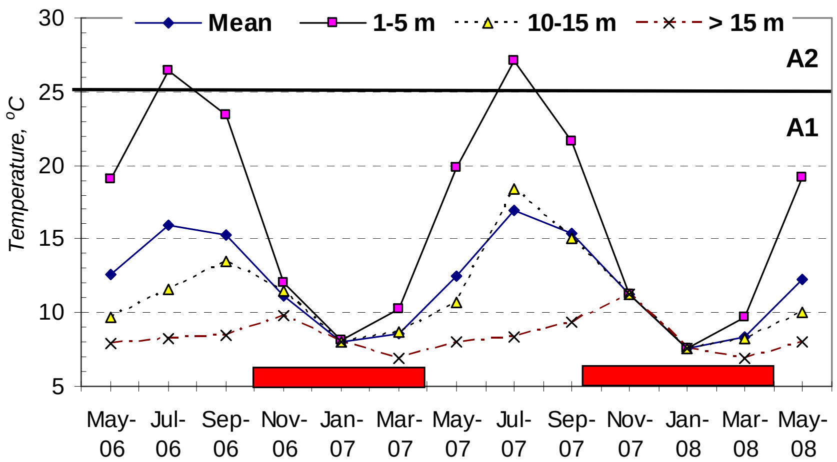

Figure 8-1. Time course of water temperature in the three layers: epilimnion (1 – 5 m), thermocline (10 – 15 m), hypolimnion (15 m to bottom) in Bovilla Lake (station S1, close to the dam), May 2006 to May 2008, and the periods of noxious smell and taste (bars below winter periods). / Ecuria e temperaturës së ujërave në tre shresat: epilimn (1-5 m), termoklin (10-15 m) dhe hipolimn (15 m deri në fund) në liqenin e Bovillës (stacioni S1, afër digës), Maj 2006-Maj 2008, dhe periudhat me erë dhe shije të papëlqyeshme (shënuar poshtë muajve të dimrit); A_1, A_2, kategoritë e cilësisë sipas 75/440.

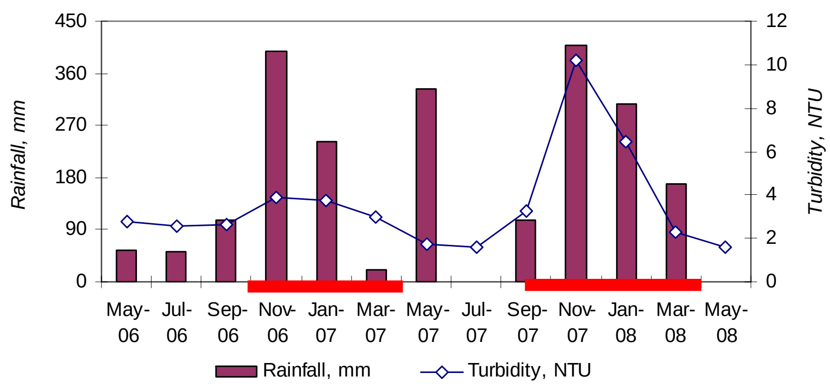

Figure 8-2. Time course of mean turbidity (NTU; 1-40 m) in Bovilla Lake (station S1, close to the dam), and rainfall (mm) in the catchment (Zall Dajti station), during May 2006 - March 2008, and the periods of noxious smell and taste (bars below during winter months). / Ecuria e turbullisë mesatare (NTU; 1-40 m) në liqenin e Bovillës (stacioni S1, pranë digës), dhe e reshjeve (mm) në pellg (nga stacioni i Zall Dajtit), gjatë periudhës Maj 2006 - Mars 2008, dhe periudhat e erës dhe shijes së papëlqyeshme (shënuar poshtë muajve të dimrit).

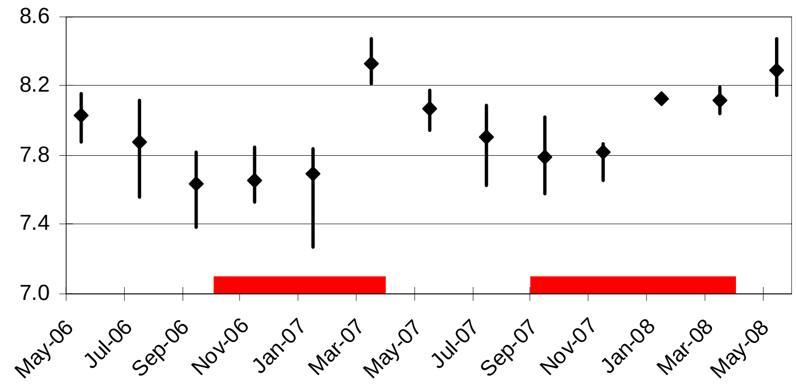

Figure 8-3. Time course of mean, minimum and maximum pH (1-40 m) in Bovilla Lake from May 2006 to May 2008, and the periods of noxious smell and taste (bars below winter months). / Ecuria e pH (vlerës mesatare, minimale dhe maksimale) në kolonën e ujit (1-40 m) në Bovillë, gjatë periudhës Maj 2006-Maj 2008, dhe periudhat e erës dhe shijes së papëlqyeshme të (shënuar nga poshtë gjatë muajve të dimrit).

Additionally, changes in temperature (ranging on an average from 22.4°C to 10.3°C) are more favorable for actinomycetes and unfavorable to algae and cyanobacteria. Actinomycetes, mainly soil microbes, may enter the water through the wash-in of spores and during the vegetative phase; they can also be hosted inside of other microorganisms which may induce the excretion of volatile odor-causing metabolites other than geosmin and MIB (Cross, 1981).

The presence of odor compounds is closely related to the turbidity of the lake water. During the odor period in Lake Bovilla there was an increase in turbidity as a consequence of heavy rainfall (Fig. 8-2). The washing of the soil from the lake shore will bring in detritus, mud, plants and rotting vegetable remains which could be colonized by autochthonous actinomycetes. Fluctuating water levels, common in a water reservoir, also increase the input of soil materials. For algae and cyanobacteria, the increase in turbidity is a stress factor as it reduces the penetration of light and as a consequence the thickness of the primary production layer. The neutral and alkaline pH support also the development of actinomycetes (Cross, 1981); the gradual increase of pH values during the smell period would favor them (Fig. 8-3).

An increase of phosphorus concentration is observed during the odor period in Bovilla Lake (Fig. 8-4). Regardless of the mechanisms, it is important to note that phosphate was suggested as important predictor of rising VOC concentrations in various models (Dzialowski *et al.,* 2007) and changes in phosphate concentrations may play an important role in early warning systems in drinking water reservoirs. We found also a relationship between odor period and the concentration of dissolved iron in water (Fig. 8-5).

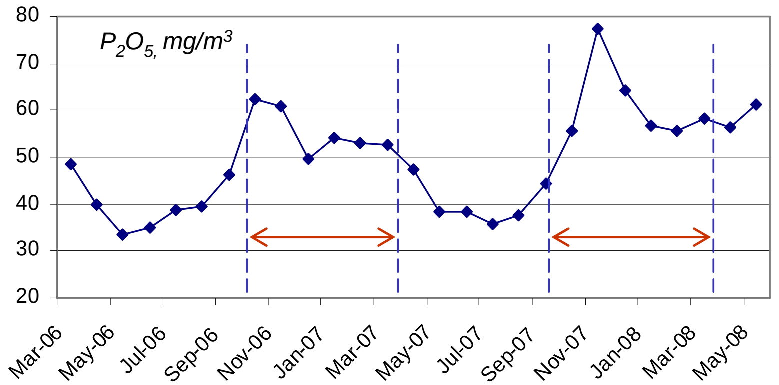

Figure 8-4. Time course of mean phosphate concentrations (as P_2O_5, mg/m^3) in water column (1-40 m) in Bovilla Lake (S1), during May 2006 - May 2008, and the periods of noxious smell and taste (double arrows in winter months). / Ecuria e vlerës mesatare të fosfateve (si P_2O_5, mg/m^3) në gjithë kolonën e ujit (1-40 m) në liqenin e Bovillës (S1), gjatë periudhës Maj 2006 - Maj 2008, dhe periudha e papëlqyeshme e erës dhe shijes gjatë dimrit (treguar me shigjetë në muajt e dimrit).

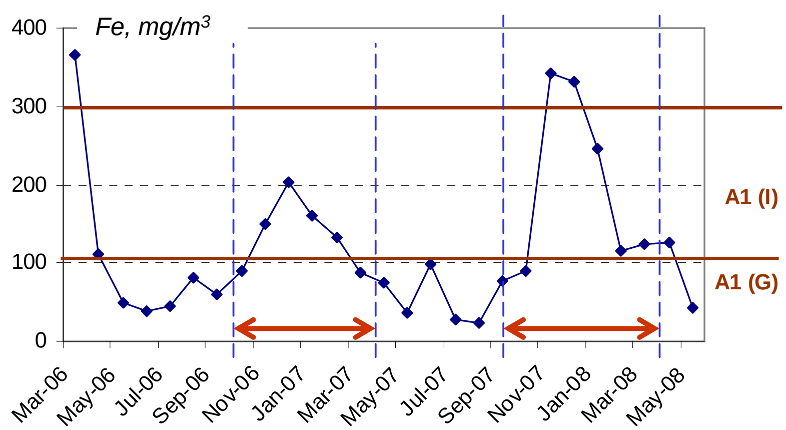

Figure 8-5. Time course of mean iron concentrations (1-40 m) in Bovilla Lake from May 2006 to May2008, and the periods of noxious smell and taste (double arrows in winter months). / Ecuria e përqëndrimit mesatar të hekurit (mg/m^3) në kolonën e ujit (1-40 m) në Bovillë gjatë periudhës Maj 2006 – Maj 2008, dhe periudhat me shije dhe erë të papëlqyeshme (shigjetat në muajt e dimrit). A_1 (I & G) quality categories after 75/440.

Another point of view is to discuss the smell and test period relative to the overturn, as these periods overlap. The vertical circulation (overturn) could redistribute the volatile decomposition products which had been accumulated in the deep water layers and the sediment to the whole water column (Isodorow & Jdanova, 2002). Bovilla is a new and not yet stabilized water body and the process of decomposition of biota remained in the sediments is still active. Such a state is also suggested from the composition of its zooplankton community (Shumka & Nikleka, Nr. 4 *in this volume*). Possibly a combination of both, the decay of plants remained in the sediments as well as release of metabolites and storage products of living microbiota originating from watershed discharges, contribute mainly to tastes and odors in Bovilla Lake.

Our evaluation about this problem is preliminary, because proving correlations between environmental factors and biological processes, and constructing appropriate predictive models need a longer and more extensive monitoring study.

8.3. Preliminary experiments to identify the volatile organic compounds in Bovilla water

To get an overview on the compounds causing the smell and taste problems, two composite water samples integrating the depth from 0 to 10 m from Bovilla Lake were taken during full circulation (holomixis) indicated by isothermic conditions on 18 January 2008. During this period, the drinking water plant is forced to treat the water with active carbon to eliminate the unpleasant smell and taste.

For the liquid extraction of VOC by dichloromethane (DCM) the procedure of Brownlee *et al.* (2004) was followed. A washed and dried amber glass bottle (1 liter) was completely filled with lake water, 50 ml were removed and 40 ml DCM added. The bottle was shaken for one hour at 80 strokes per minute. After separation of the layers, about 800 ml of the upper water layer was discarded and the remainder transferred to a 250 ml separatory funnel.

After the separation, 30 to 35 ml of the lower DCM layer is transferred into a 25 ml calibrated glass cylinder with glass cap containing a layer of ca. 5 mm anhydrous sodium sulfate. The cylinder was shaken to provide a good contact of the liquid with the sodium sulfate. The DCM extract was reduced to volume to ca. 10 ml under a stream of argon and then transferred into a 15 ml centrifuge tube.

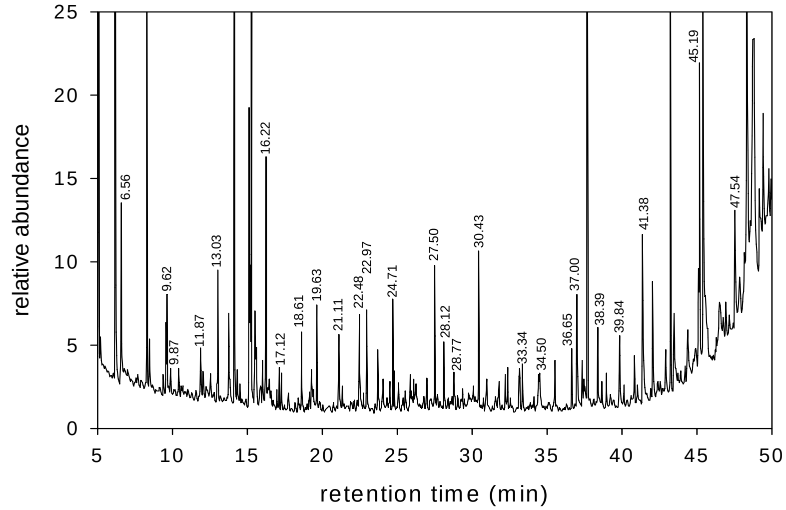

Figure 8-6. GC spectrum of the DCM-extract from Bovilla Lake, showing a sum of peaks characterized by the retention time (see text for more details). / Spektri GC i ekstraktit DCM nga liqeni i Bovillës, që tregon një shumicë pikesh që karakterizohen nga koha e mbajtjes (*shih* tekstin për më shumë hollësi).

The Gas-Chromatograph (GC) separation of the DCM extract of Bovilla water is presented in figure 8-6. It clearly demonstrates the high complexity of the different volatile compounds in the sample. Some of the compounds could be tentatively identified using the Wiley library of the software used.

Compounds possibly present in Bovilla water included lower alcohols and aldehydes, borneol, butyl-4-methylcyclopentene, camphor, carvone, norpinon, pinocarvone, propyl-cyclohexanone, alpha-terpineol, and verbenon.

It seems that neither geosmin nor 2-MIB were definitively present in the water at the sampling time, but an astonishing number of mono- and sesquiterpenes, among which carvone (Fig. 8-7) may be a dominating compound causing the smell. Carvone is produced by over 70 different plants (Burdock, 1995) and has been found in the bark of several trees (Eriksson *et al.,* 2008). Furthermore, some fungi produce carvone besides other volatiles (Mdaini *et al*., 2006; Morrish *et al.,* 2008). It must be kept in mind that because of many isomers the mass spectrum is not sufficient for an unequivocal

determination of such monoterpenes. A proper determination needs the necessary reference compounds, possibly to be synthesized.

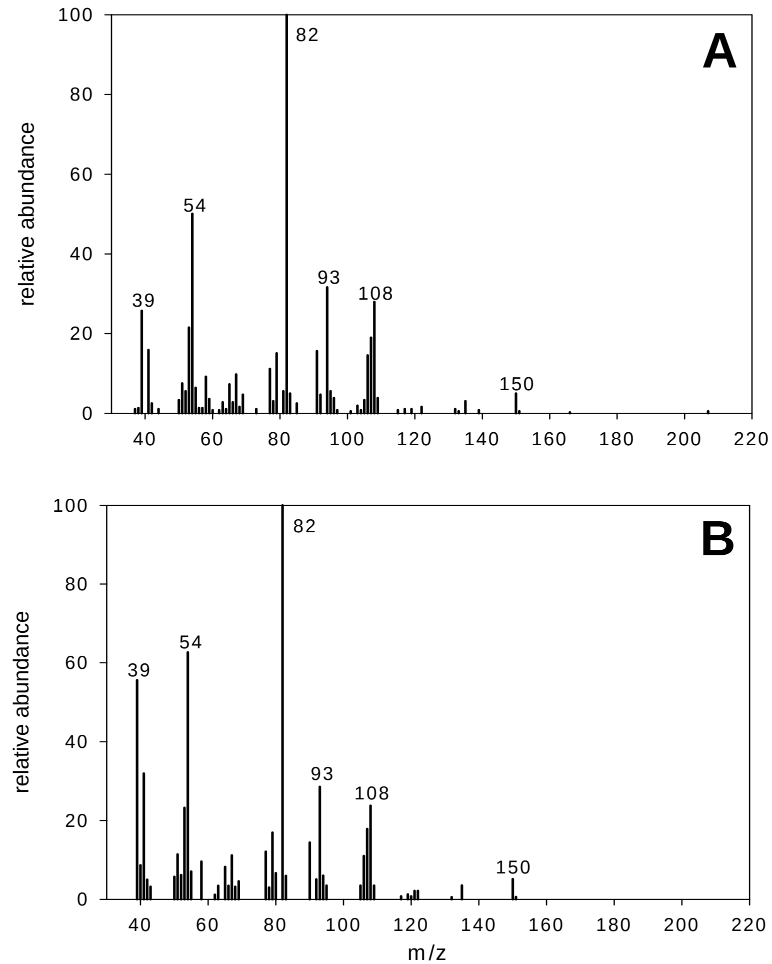

Figure 8-7. Mass-spectrum of the compound eluting at 27.5 min (*see* Fig. 8-6) from DCM extract of water of Bovilla Lake (A) and reference spectrum of carvone (B). / Spektri i masës së lëndëve të veçuara në 27.5 min (*shih* Fig. 8-6) nga ekstrakti DCM i ujit të Bovillës (A) dhe spektri krahasues i karvonit (B).

Conclusions

1) Unpleasant smell and taste periods in water of Bovilla Lake were observed during autumn and winter that correspond to the period of water column mixing.

2) Striking links are observed between smell and taste periods and some physico-chemical water parameters: drop of temperature, increase in turbidity, abundant rainfall, gradual increase of pH, maximum values of phosphorus and iron concentrations. These parameters (and perhaps others) could be used to predict in future the start and the end of odor problems.

3) It is assumed that the causes of the odor phenomena in water of Bovilla Lake are not the cyanobacteria growth, but rather actinomycetes, possibly in association with microalgae. Actinomycetes enter the water through the wash-in of spores and vegetative cells. This is supported by the links between physico-chemical parameters and odor periods in Bovilla Lake, but also by the low phytoplankton biomass in the water in winter and the data from zooplankton studies.

4) The problem of odor events is particular for every lake and reservoir and also for Bovilla Lake. There are two ways how to face it:

(i) using a treatment with active carbon, as at the moment best technology to remove the unpleasant smell and taste compounds in drinking water;

(ii) to take appropriate measures for the lake management. Control of non-point source pollution through reduction of erosion is expected to be beneficial to some extent. Long-term proactive management needs to address the source(s) of the problem, by identifying the compound(s) responsible and their producers, as well as the impact of environmental and biological agents on these organisms. Clearly, an ecologically sound watershed and source water remediation and management is essential for the long-term effectiveness and sustainability of a successful water supply program (Jüttner, 2007).

5) By GC-MS analysis of a single water sample from Bovilla Lake a complex mixture of odor VOC, mainly monoterpenes as carvone, campher, pinocarvone, borneol, alpha-terpineol, beta-cyclocitral, and verbenon was found, but neither Geosmin nor 2-MIB were detected.

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