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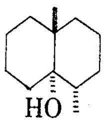
Odorous Compounds in Water Supplies. Identification of Geosmin from Water of Lake Inbanuma by Mass Spectrometry combined with Gas Chromatography[1]

In a previous paper[2] we described the gas chromatographic detection of geosmin (I)[3] from the raw water of Kyoto City Water Supplies, which was supplied from the southern basin of Lake Biwa and carried an earthy-musty taste and odor. Also during past four years a taste and odor of a distinct earthy-musty character developed frequently in the water of Lake Inbanuma, which is one of the water sources of Chiba Prefectural Water Supplies. The present paper concerns with the causative material of this objectionable taste and odor.



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The water of Lake Inbanuma (about 1400 tons in total amount), after pre-chlorination (1 ppm) and aluminum sulfate treatment (80 ppm) followed by filtration through a sand

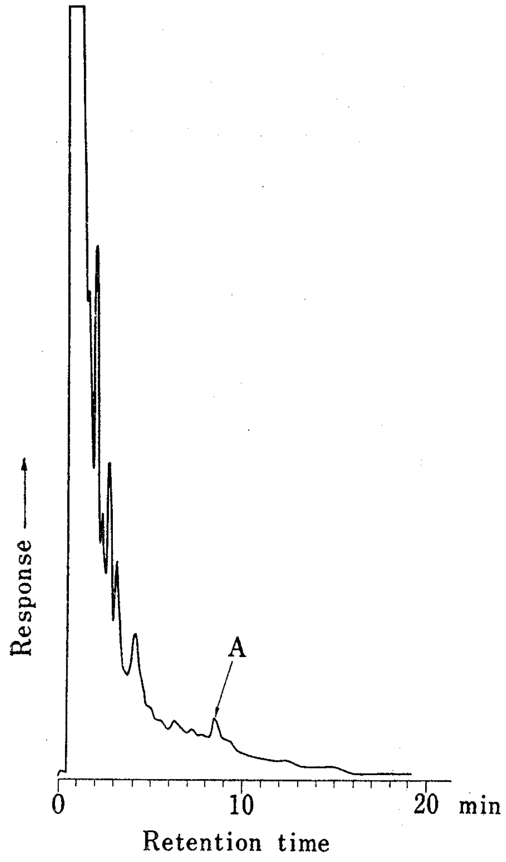

Fig. 1. Gas Chromatogram of the Odorous Compounds from the Water in Lake Inbanuma

condition: Hitachi Gas Chromatograph Model 063; column, 15% Reoplex 400 on chromosorb WNAW (2 m × 3 mm i.d. stainless steel); detector, FID; column temp., 150°; carrier gas, N_2 (30 ml/min)

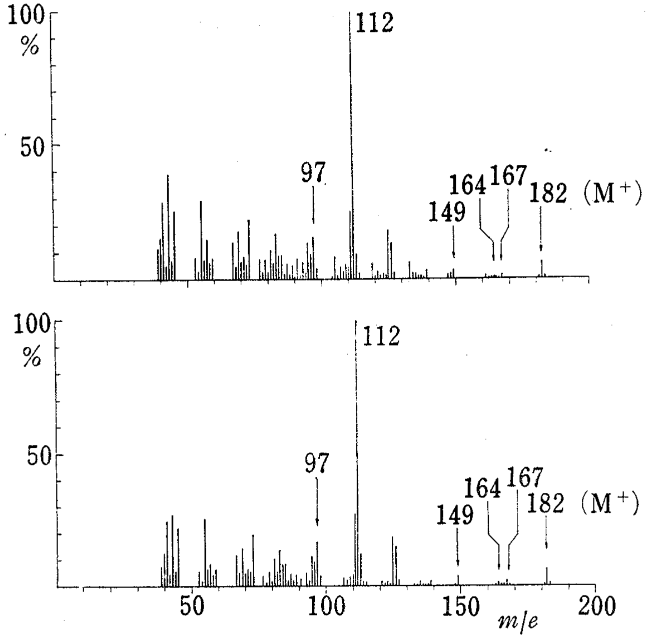

Fig. 2. a) Mass Spectrum of the Odorous Compound from the Water in Lake Inbanuma
b) Mass Spectrum of Authentic *dl*-Geosmin

condition: GC column, 25% PEG 20 M on chromosorb W (1.5 m × 2 mm i.d. glass tube); column temp., 115°; carrier gas, He (2 kg/cm²); MS ionization voltage, 70 eV; accelerator voltage, 1800 V; ion source temp., 220°; carrier gas separator, Watson-Biemann type molecular separator (Hitachi Mass Spectrometer RMU-6E)

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1) This work was supported in part by a Grant-in-Aid for Scientific Research from the Ministry of Education, which is gratefully acknowledged.
2) T. Kikuchi, T. Mimura, and Y. Moriwaki, *Yakugaku Zasshi*, **92**, 1441 (1972).
3) N.N. Gerber, *Tetrahedron Letters*, **1968**, 2971.

layer (80 cm in thickness), was passed through a column packed with granular activated charcoal[4] (20×200 cm) over a period from April to September, 1972 in order to adsorb the odorous compounds. An 800 g portion of this activated charcoal was extracted several times with methylene chloride to give a small amount of oily substance. To this substance was added purified water (50 ml) and the solution was slowly distilled. The distillate (10 ml) obtained was extracted with methylene chloride, dried (anhydrous $MgSO_4$), and the solvent was carefully removed by slow evaporation. The residual substance was then subjected to silica gel column chromatography (0.8×12 cm) using pentane and ether–pentane mixture as solvents, where elution with 1: 99 ether–pentane mixture afforded a small amount of oily substance having the earthy-musty odor.

Gas chromatographic examination of this substance using three kinds of columns (10% PEG 20M, 10% SE-30, and 15% Reoplex 400) indicated the presence of a compound which has the same retention time as authentic *dl*-geosmin (I). Fig. 1 reproduces the gas chromatogram taken on 15% Reoplex 400 column, where the peak A corresponds to geosmin (I).

In order to confirm this finding we then examined the mass spectra obtained by mass spectrometry combined with gas chromatography (GC-MS). As seen in Fig. 2, the mass spectrum of peak A was quite identical with that of authentic *dl*-geosmin (I) measured under the same condition.

On the ground of above data, one of the causative materials of earthy-musty odor and taste occurred in Lake Inbanuma was fully proved to be geosmin (I). This is the first example of the complete identification of geosmin (I) in the raw water of public water supplies by the GC-MS method.

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[4] Originally, this activated charcoal column was used for the removal test of odorous materials from the raw water at the Inbanuma Water Intake Plant, Chiba Prefectural Water Works Bureau.