

ALKANES AND ALKANOIC ACIDS IN THE BEACON
SUPERGROUP SAMPLES FROM THE ALLAN
HILLS AND THE CARAPACE NUNATAK
IN ANTARCTICA

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Abstract: Alkanes and alkanolic acids in the Beacon Supergroup samples, sedimentary rocks, silicified wood and coal, from the Allan Hills and the Carapace Nunatak of southern Victoria Land in Antarctica were studied to elucidate the organic sources and sedimentary paleoenvironment. Normal alkanes ranging in carbon chain length from nC_{12} to nC_{30} , pristane and/or phytane were found in all the samples studied with concentrations ranging from 0.24 to 25 $\mu\text{g/g}$ of dry sample, along with near unity of carbon preference index (0.91-1.4). The major constituents were all short-chain n -alkanes ($<C_{20}$, nC_{16} , nC_{17} , nC_{18} , nC_{19}), pristane and/or phytane. Normal alkanolic acids of carbon chain lengths between nC_8 and nC_{28} were found in a sedimentary rock from the Carapace Nunatak and in silicified wood from the Allan Hills with low concentrations (2.3-25 $\mu\text{g/g}$), together with the dominance of even-carbon numbers (carbon preference index, 1.6-2.8). The higher abundances of long-chain ($\geq nC_{20}$) n -alkanes and n -alkanoic acids in the sedimentary rock from the Carapace Nunatak imply that the contribution of land plants to the sedimentary paleoenvironment of this area was greater than that in the Allan Hills. The pristane/phytane ratios suggest that the sedimentary paleoenvironment of the Allan Hills was oxic compared with that of the Carapace Nunatak.

1. Introduction

The Beacon Supergroup, composed of sandstone, siltstone, conglomerate and carbonaceous beds, including various fossils, constitutes the major formation of the Transantarctic Mountains, which are the largest mountain chains in Antarctica, extending 4000 km with the highest peak of 4528 m. The Beacon Supergroup, ranging in age from early or pre-Devonian to Jurassic, was formed in lacustrine and shallow marine environments of Gondwanaland. ELLIOTT and EVANS (1963) reported alkanes ranging in carbon chain lengths from C_5 to C_{10} generated by pyrolysis of digested Beacon sandstone collected from Cape Royds of Ross Island, Antarctica. MATSUMOTO *et al.* (1985) reported normal, branched and unsaturated fatty acids in the Beacon Supergroup samples from the McMurdo Sound region of southern Victoria Land in Antarctica.

The fatty acids found in the Beacon Supergroup samples are thought to have been derived not only from ancient organisms but also from Recent organic matter and/or living microorganisms, including bacteria, fungi and algae. Here we report the features of *n*-alkanes, pristane and phytane, and *n*-alkanoic acids in the Beacon Supergroup samples from the Allan Hills and the Carapace Nunatak of southern Victoria Land in Antarctica and discuss their sources and sedimentary paleoenvironments.

2. Materials and Methods

2.1. Sampling sites and samples

The Beacon Supergroup of the Allan Hills and the Carapace Nunatak regions is a sedimentary sequence of pale yellow quartz sandstone and siltstone, including coal seams and plant fossils. TOWNROW (1966) reported plant fossils characterizing *Glossopteris*, from the Permian and Triassic ages of the Allan Hills, and Jurassic plants from the Carapace Nunatak. According to synthesized geology of the Allan Hills by BALLANCE and WATTERS (1971), the Beacon Supergroup of the Allan Hills is covered partially by diamictite of Jurassic age and is intruded by basalt dykes of early Jurassic age. At the Carapace Nunatak, sandstone and fine conglomerates of Jurassic age referred to the Beacon Supergroup are conformably overlain by basalt with pillow.

During the 1978–79 austral summer, the sedimentary rock, silicified wood and coal samples were collected from the outcrops of the Allan Hills and the Carapace Nunatak (Fig. 1). At the Allan Hills there is no dyke intrusions within 50m from the sampling sites. The sample at the Carapace Nunatak was collected from 3 to 5m below the boundary of the basalt flow of 300m in thickness.

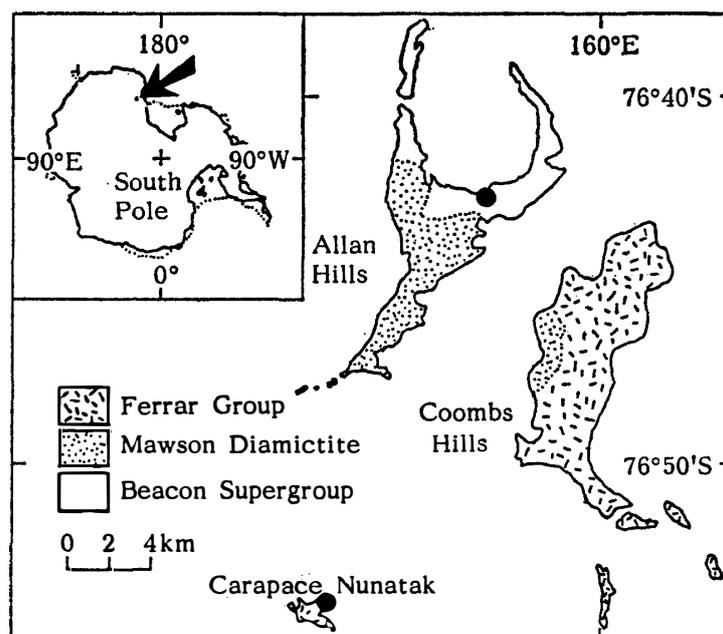


Fig. 1. The geology of the Allan Hills and the Carapace Nunatak of southern Victoria Land in Antarctica (BALLANCE and WATTERS, 1971). ●: Sampling site.

2.2. Analyses

The surfaces of sedimentary rock, silicified wood and coal samples were scraped off to avoid contamination and powdered under 200 mesh using a Retschmuhle with Elektro-Kleinmotoren, Altenhagen G. M. B. & Co. (West Germany). Total organic carbon (TOC) and total nitrogen (TN) were determined using a Yanako MT2 CHN Corder after treatment of samples with 6 M hydrochloric acid to remove inorganic carbon. The extraction methods were described elsewhere (MATSUMOTO *et al.*, 1985). The sample powders (20–30 g) were saponified with 0.5 M potassium hydroxide methanol solution under reflux (2 h). The saponified samples were centrifuged (1.0×10^3 g). The supernatants and the residues were acidified separately with concentrated hydrochloric acid and extracted three times with ethyl acetate. The ethyl acetate extracts were evaporated to dryness, redissolved with 50 μ l benzene:ethyl acetate (1 : 1) and chromatographed through a silica gel column (180 \times 5 mm i.d., 100 mesh, 5% water). Hydrocarbons were eluted with 2 column volumes of hexane. Normal and isoprenoid alkanes were determined using a Shimadzu GC-8A gas chromatograph (FID) fitted with a fused silica capillary column (Hewlett-Packard ultra performance No. 2, 25 m \times 0.2 mm i.d.). Gas chromatograms were monitored with a Shimadzu Chromatopac CR-3A. The column temperatures were programmed from 80 to 300°C at 10°C/min. The temperatures of injection block and detector were maintained at 330°C. The identification of alkanes was also made by the use of a Hewlett-Packard 5985 gas chromatograph-mass spectrometer equipped with a fused silica capillary column (Hewlett-Packard ultra performance No. 2, 25 m \times 0.31 mm i.d.). The splitless mode was used. The column oven temperature was programmed from 50 to 80°C at 30°C/min and then to 300°C at 10°C/min. The temperatures of molecular separator and ion source were maintained at 300 and 200°C, respectively. The mass spectra were obtained at an ionization energy of 70 eV. The scan speed was 50–450 m/z per s. Alkanes and alkanoic acids were identified by comparison of their retention times and mass spectra with those of authentic standards.

3. Results and Discussion

3.1. TOC and TN

The TOC values of the sedimentary rocks from the Allan Hills ranging from 15.9 to 44.9 mgC/g of dry sample were somewhat smaller than that of a sedimentary rock sample from the Carapace Nunatak (55.1 mgC/g), while those of the TN results for the Allan Hills were similar to that of the Carapace Nunatak (Table 1). These TOC values were generally considerably greater than those of sedimentary rocks from Mount Fleming and Plane Table of the McMurdo Sound region (MATSUMOTO *et al.*, 1985). These results can be explained by the thermal history of both the areas after the formation of the Beacon Supergroup. According to FUNAKI (1983, 1984), paleomagnetic investigations showed that some parts of the Beacon Supergroup at Mount Fleming may have been heated by dykes of the Ferrar dolerite and partially remagnetized parallel to the Jurassic geomagnetic field. The Beacon Supergroup in the Asgard Range, involving Plane Table may have been heated by the Ferrar dolerite intrusion (FUNAKI, unpublished results). In the case of the Allan Hills and the Carapace Nunatak, paleo-

Table 1. TOC and TN results for the Beacon Supergroup samples from the Allan Hills and the Carapace Nunatak of southern Victoria Land in Antarctica.

Sample	TOC (mgC/g)	TN (mgN/g)	Atomic ratio C/N	Color
Allan Hills				
Sedimentary rock*				
Sed-1	38.2	1.89	17.3	Gray
Sed-2	21.4	1.58	11.6	Olive gray
Sed-3	44.9	5.88	6.55	Olive black
Sed-4	18.5	5.59	2.84	Gray
Sed-5	15.9	1.49	9.15	Olive gray
Sed-6	16.1	3.07	4.50	Olive gray
Silicified wood				
Sil-1	32.7	0.86	33	Olive black
Sil-2	35.1	0.72	42	Olive black
Sil-3	22.8	1.15	17.0	Gray
Coal				
Coal-1	682	18.9	30.9	Black
Coal-2	637	13.7	39.9	Black
Carapace Nunatak				
Sedimentary rock**				
Sed-7	55.1	2.83	16.7	Brownish black

* Sand-siltstone containing plant fossils.

** Sand-siltstone containing ostracod fossils.

magnetic studies indicated that the surfaces of these regions were probably not heated higher than the magnetite Curie point (580°C). These estimations are also supported by the existence of volatile matter in coal samples from the Allan Hills (FUNAKI, 1983, 1984, unpublished results). Our TOC results are consistent with those of the above results.

The TOC content of silicified wood was similar to that of the sedimentary rocks, indicating that most of organic carbon was replaced with mineral constituents, whereas that of coal samples was much higher (682 and 637 mgC/g). The atomic C/N ratios of the sedimentary rocks varied largely in the samples between 4.50 and 17.3, which were considerably smaller than those of the silicified wood and coal samples, suggesting different source materials and/or subsequent alteration.

3.2. Alkanes

Normal alkanes ranging in carbon chain lengths from nC_{12} to nC_{27} , pristane and phytane were found in the capillary gas chromatogram of the sedimentary rock from the Carapace Nunatak of Antarctica, in addition to unresolved complex mixture (UCM) of hydrocarbons (Fig. 2). The compositions of n -alkanes, pristane and phytane are summarized in Table 2. The major alkanes (>10%) in the sediment samples from the Allan Hills and the Carapace Nunatak were all short-chain n -alkanes (< nC_{20}), pristane and/or phytane, which were similar to those of the silicified wood and coal samples. No long-chain n -alkanes as the major constituents were found in all the Beacon Supergroup samples studied so far, suggesting that the contribution of the waxes of higher plants was generally small, because long-chain n -alkanes are abundant in

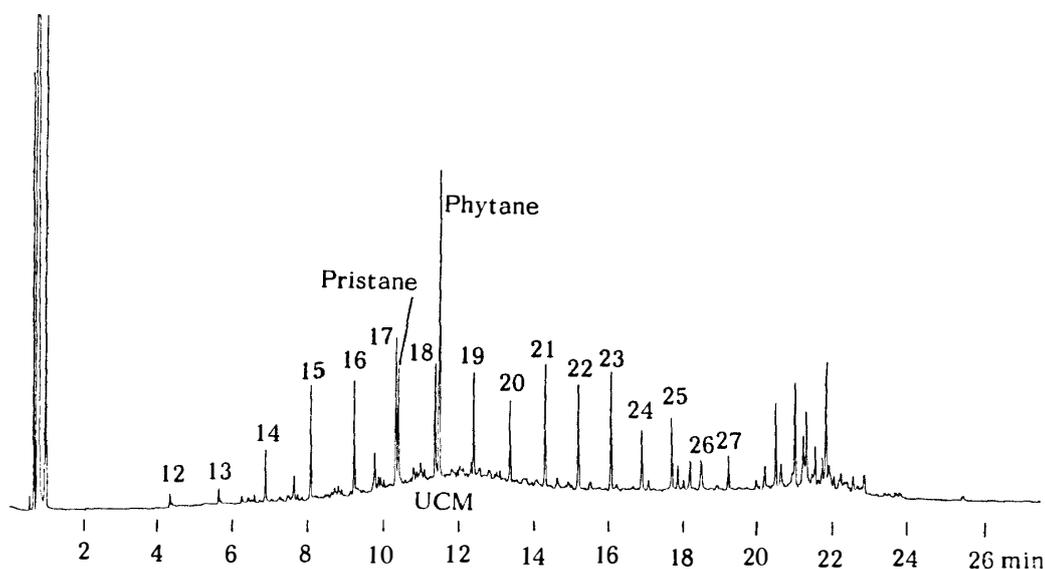


Fig. 2. Capillary gas chromatogram of the hydrocarbon fraction from the sedimentary rock of the Carapace Nunatak in Antarctica. Arabic figures on the peaks indicate the carbon chain length of *n*-alkanes. UCM: Unresolved complex mixture of hydrocarbons. Peaks with no figures were not identified.

Table 2. Alkane composition of the Beacon Supergroup samples from the Allan Hills and the Carapace Nunatak of southern Victoria Land in Antarctica (%).

Carbon chain length	Sedimentary rock							Silicified wood			Coal	
	Sed-1	Sed-2	Sed-3	Sed-4	Sed-5	Sed-6	Sed-7	Sil-1	Sil-2	Sil-3	Coal-1	Coal-2
<i>n</i> -Alkane												
12	3.1	0.4	—	—	0.6	0.9	0.5	0.7	0.2	0.4	2.6	—
13	1.6	0.6	—	—	2.1	3.4	0.7	3.2	1.6	1.0	3.5	1.8
14	5.0	3.8	4.0	1.8	6.1	11.5	2.8	12.1	6.4	2.7	11.1	9.0
15	8.6	8.5	8.3	5.5	8.1	16.3	5.9	18.0	12.5	3.6	13.7	15.1
16	9.1	10.9	10.2	6.7	12.2	17.1	5.9	11.9	9.7	2.5	15.8	20.1
17	11.6	14.7	14.8	11.7	13.1	18.0	8.1	13.3	11.2	4.0	12.2	16.3
18	12.6	16.2	13.6	14.1	12.6	10.1	6.4	7.6	12.6	5.3	8.4	10.6
19	10.6	10.5	8.4	11.6	8.6	3.6	5.8	8.3	10.8	5.7	5.9	4.6
20	8.3	7.4	5.9	8.5	6.5	1.7	4.7	5.0	7.9	7.6	3.6	2.5
21	5.7	3.6	2.5	6.7	2.7	0.9	7.2	2.7	3.3	8.8	2.2	1.3
22	4.0	2.0	2.1	5.5	2.9	0.5	6.7	1.8	2.1	11.4	1.2	0.8
23	1.8	0.6	2.0	4.1	1.6	0.1	7.2	1.0	1.4	9.7	0.7	0.5
24	0.8	0.2	1.5	2.7	0.6	—	4.1	0.7	0.9	7.9	0.3	0.3
25	0.5	—	1.2	1.8	0.1	—	4.0	0.5	0.6	5.8	0.3	0.2
26	0.2	—	0.6	1.5	—	—	3.3	—	0.5	6.1	0.1	—
27	—	—	—	1.8	—	—	2.1	—	0.4	2.4	0.1	—
28	—	—	—	—	—	—	0.7	—	0.4	2.5	Trace	—
29	—	—	—	—	—	—	—	—	0.4	1.6	Trace	—
30	—	—	—	—	—	—	—	—	0.3	1.1	—	—
Pristane	7.1	9.2	12.4	6.9	11.9	8.9	6.6	7.9	8.3	4.0	10.5	10.6
Phytane	9.4	11.4	12.5	9.1	10.3	7.0	17.3	5.3	8.5	5.9	7.8	6.3

Trace: Less than 0.05%.

Table 3. Alkanes found in the Beacon Supergroup samples from the Allan Hills and the Carapace Nunatak of southern Victoria Land in Antarctica.

Sample	Range	Conc. ($\mu\text{g/g}$)	Composition (%)			B/A	CPI _H * ¹	Pr* ² / <i>n</i> C ₁₇	Pr* ² + Ph* ³ / <i>n</i> C ₁₇	Pr* ² / Ph* ³
			<i>n</i> C ₁₂ - <i>n</i> C ₁₉ (A)	<i>n</i> C ₂₀ - <i>n</i> C ₃₀ (B)	Iso- prenoids					
Sedimentary rock										
Sed-1	C ₁₂ -C ₂₆	4.9	62.2	21.3	16.5	0.34	1.0	0.61	1.4	0.76
Sed-2	C ₁₂ -C ₂₄	6.0	65.6	13.8	20.6	0.21	0.99	0.62	1.4	0.81
Sed-3	C ₁₄ -C ₂₆	0.68	59.3	15.8	24.9	0.27	1.0	0.83	1.7	1.0
Sed-4	C ₁₄ -C ₂₇	0.24	51.4	32.6	16.0	0.63	1.1	0.59	1.4	0.76
Sed-5	C ₁₂ -C ₂₅	5.9	63.4	14.4	22.2	0.23	0.91	0.90	1.7	1.2
Sed-6	C ₁₂ -C ₂₃	3.6	80.9	3.2	15.9	0.040	1.1	0.50	0.88	1.3
Sed-7	C ₁₂ -C ₂₇	7.5	36.1	40.0	23.9	1.1	1.2	0.80	3.0	0.38
Silicified wood										
Sil-1	C ₁₂ -C ₂₅	1.5	75.1	11.7	13.2	0.16	1.4	0.60	0.99	1.5
Sil-2	C ₁₂ -C ₃₀	0.62	64.9	18.2	16.8	0.28	1.1	0.74	1.5	0.98
Sil-3	C ₁₂ -C ₃₀	3.3	25.2	64.9	9.9	2.6	0.92	0.99	2.5	0.68
Coal										
Coal-1	C ₁₂ -C ₂₉	25	73.2	8.5	18.3	0.12	1.0	0.86	1.5	1.3
Coal-2	C ₁₃ -C ₂₅	4.8	77.5	5.6	16.9	0.072	1.0	0.65	1.0	1.7

*¹ Carbon preference index. *² Pristane. *³ Phytane.

the waxes of higher plants (HERBIN and ROBINS, 1968; TULLOCH, 1976; MATSUMOTO, 1982). The carbon chain length of *n*-alkanes in the sediment samples ranged from *n*C₁₂ to *n*C₂₈, similar to those of the silicified wood and coal samples (Table 3). The total concentration of the alkanes in the sediment samples ranged from 0.24 to 7.5 $\mu\text{g/g}$ of dry sample, varying considerably among the samples as in the case of the TOC results (Table 1). The values are similar to those of the silicified wood and coal samples.

The ratio of long (*n*C₂₀-*n*C₃₀) to short (*n*C₁₂-*n*C₁₉) chain *n*-alkanes varied largely from 0.040 to 2.6, reflecting presumably the differences of relative contribution of the waxes of higher plants (land plant) and aquatic biota including plankton. The high Long/Short ratios of the Sed-7 and Sil-3 samples are likely to be due to larger contribution of the waxes of land plants, whereas those of low values for the Sed-6 and Coal-2 samples suggest the contribution of aquatic biota.

The carbon preference index (CPI_H) of *n*-alkanes, indicating the ratio of odd- to even-carbon numbers, decreases with increasing age of sediment and attains near unity (COOPER and BRAY, 1963; KVENVOLDEN, 1966, 1970). The CPI_H values of the Beacon Supergroup samples were close to unity (0.91-1.4, Table 3). Thus these results revealed that the degradation of *n*-alkanes occurred considerably after sedimentation.

In non-marine depositional paleoenvironments of sediments, a higher contribution of land derived organic matter can be expected. Such environments may show lower amounts of *n*-heptadecane than those laid down under fully marine conditions (DIDYK *et al.*, 1978). LIJMBACH (1975) proposed that the pristane/*n*-heptadecane (Pr/*n*C₁₇) ratio of a crude oil may give an indication of its depositional origin, distinguishing open water conditions of sedimentation (values <0.5) from inland peat-swamp environments (values >1.0). In our case, the Pr/*n*C₁₇ values of the Beacon Supergroup samples

ranged from 0.50 to 0.99, suggesting that organic matter in the sedimentary paleo-environment was derived both from land plants and aquatic biota in various ratios. It is known, however, that the concentration of pristane originated from phytol side chain of chlorophyll changes in the sedimentary conditions as discussed below. Therefore, here we calculated the ratios of pristane+phytane/ nC_{17} (Pr+Ph/ nC_{17}) in addition to Pr/ nC_{17} . The high Pr+Ph/ nC_{17} values of the Sed-7 and Sil-3 samples are consistent with those of the Long/Short ratio (Table 3). In addition, the low Pr+Ph/ nC_{17} values of the Sed-6 and Coal-2 samples also reflect the low Long/Short ratio. Thus the Pr+Ph/ nC_{17} ratio may be an useful marker of organic matter sources.

Isoprenoid alkanes, pristane and phytane, are considered to be products of the diagenesis of the phytol side chain of chlorophyll, although other sources are possible (POWELL and MCKIRDY, 1973; DIDYK *et al.*, 1978). Pristane is formed from phytol by an oxidative pathway, for example via phytanic and/or phytenic acids and subsequent defunctionalization, while phytane is generated through various reductive paths. Thus the ratio of Pr/Ph in a reducing (anoxic) environment tends to give low values, whereas the ratio in an oxic environment is reverse (BROOKS *et al.*, 1969; POWELL and MCKIRDY, 1973; DIDYK *et al.*, 1978). The Pr/Ph ratio of the sedimentary rock samples from the Allan Hills ranged from 0.67 to 1.3, similar to that of the silicified wood and coal samples, but much higher than that of the Carapace Nunatak (0.38, Table 3). These results suggest that the sedimentary rocks in the Carapace Nunatak were formed in a reducing environment as compared with those in the Allan Hills.

3.3. Fatty acids

Normal alkanolic acids ranging in carbon chain length from nC_6 to nC_{28} were found in the capillary gas chromatogram of the sedimentary rock collected from the Carapace Nunatak (Fig. 3). Fatty acids found in the three samples analyzed were summarized

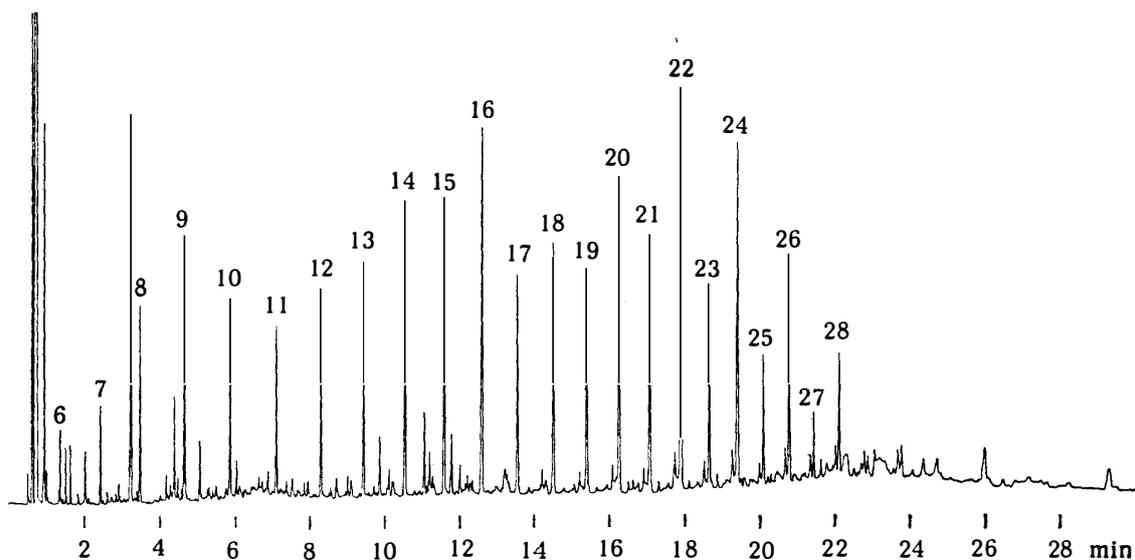


Fig. 3. Capillary gas chromatogram of the fatty acid fraction from the sedimentary rock of the Carapace Nunatak in Antarctica. Arabic figures on the peaks denote carbon chain length of n -alkanoic acids. Peaks with no figures were not identified.

Table 4. Normal alkanolic acids found in the Beacon Supergroup samples from the Allan Hills and the Carapace Nunatak of southern Victoria Land in Antarctica.

Carbon chain length	Sedimentary rock		Silicified wood	
	Sed-7	Sil-1	Sil-1	Sil-3
Composition (%)				
6	0.6	0.9		1.5
7	1.3	1.4		1.6
8	2.6	4.2		4.2
9	4.2	15.3		9.3
10	3.2	9.0		3.7
11	2.6	4.5		1.9
12	3.3	10.9		4.4
13	3.4	4.3		1.8
14	5.6	11.3		7.5
15	5.4	4.2		5.1
16	7.4	20.7		20.6
17	4.1	4.9		2.6
18	5.2	6.9		15.9
19	4.6	—		2.5
20	8.0	1.5		3.0
21	5.6	—		2.8
22	9.4	—		3.4
23	4.2	—		1.3
24	7.5	—		2.8
25	2.8	—		0.9
26	4.3	—		1.6
27	1.7	—		0.5
28	3.0	—		1.1
$nC_{20}-nC_{28}/nC_{10}-nC_{19}$	1.0	0.020		0.26
CPI _A *	1.6	2.6		2.8
Conc. ($\mu\text{g/g}$)	25	2.3		7.5

* Carbon preference index.

in Table 4. No unsaturated and branched acids were identified in our samples because of their small quantity. These results indicate that the contribution of Recent organic matter, including contamination is negligible. Besides, the effect of bacteria on our samples is also small, because bacterial lipids are characterized by the abundance of branched acids (LEO and PARKER, 1966; KANEDA, 1967). Very short-chain *n*-alkanoic acids ($<nC_{10}$) were found in considerable quantity in all the samples studied. These acids are unusual in common organisms, and suggest that the sedimentary environment had been heated considerably after sedimentation due to the intrusion of basalt dykes, although the temperature was lower than the magnetite Curie point. Fatty acid compositions of these samples are much different from those found in the sedimentary rocks from Plane Table of the Asgard Range and Mount Fleming in the McMurdo Sound region, in which branched and unsaturated fatty acids are abundant (MATSUMOTO *et al.*, 1985). Our *n*-alkanoic acids found in the Beacon Supergroup samples are also much different from those of soil in the McMurdo Sound region (MATSUMOTO *et al.*,

1979, 1981). Thus these results revealed again that the Beacon Supergroup is not important as a source of very long-chain *n*-alkanoic acids found in the soil. The concentrations of *n*-alkanoic acids in the sedimentary rock (Sed-7) and silicified wood (Sil-1 and Sil-3) were somewhat higher than those of alkanes in the same samples.

The Long (nC_{20} - nC_{28})/Short (nC_{10} - nC_{19}) ratio of the Sed-7 sample showed again the contribution of the waxes of higher plants to the sedimentary paleoenvironment, while that of the Sil-1 and Sil-3 samples suggests that most of waxes were lost after sedimentation.

In principle, the carbon preference index (CPI_A) for *n*-alkanoic acids, the ratio of even- to odd-carbon numbers, decreases with increasing age of sedimentary environments and reaches unity (COOPER and BRAY, 1963; KVENVOLDEN, 1966, 1970). The CPI_A value of Sed-7 sample was low (1.6), while those of silicified wood, Sil-1 and Sil-3, were considerably high (2.6 and 2.8), indicating the predominance of even-carbon numbers in the latter samples, but the reason is not clear (Table 4).

4. Conclusions

Normal alkanes ranging in carbon chain length from nC_{12} to nC_{30} , pristane and phytane were found in the Beacon Supergroup samples, sedimentary rocks, silicified wood and coal, from the Allan Hills and the Carapace Nunatak of southern Victoria Land in Antarctica, with short-chain *n*-alkanes, pristane and/or phytane as the major constituents ranging in concentrations from 0.24 to 25 $\mu\text{g/g}$ of dry sample. Normal alkanoic acids ranging in carbon chain length from nC_6 to nC_{28} were found in the samples of a sedimentary rock and silicified wood with concentrations between 2.3 and 25 $\mu\text{g/g}$. The Long/Short and Pr+Ph/ nC_{17} ratios for alkanes and the Long/Short values for *n*-alkanoic acids indicated that the contribution of land plants to the sedimentary paleoenvironment of the Carapace Nunatak may have been greater than that of the Allan Hills. The Pr/Ph values suggest that the sedimentary paleoenvironment of the Allan Hills was oxic compared with that of the Carapace Nunatak.

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