

ISOLEUCINE EPIMERIZATION IN QUATERNARY BENTHONIC FORAMINIFERA FROM THE NORWEGIAN CONTINENTAL SHELF: A PILOT STUDY

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ABSTRACT

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The extent of epimerization of the protein amino acid L-isoleucine has been measured in Late Quaternary benthonic foraminifera found in cores from two areas of the Norwegian Continental Shelf in order to evaluate the potential of this reaction as a geochronological tool. Measurements were restricted to monospecific assemblages of *Cibicides lobatulus* and *Elphidium excavatum* from ¹⁴C-dated and seismostratigraphic units representing a range of ages. By comparing with data on the same species from last-interglacial and late-glacial sites in western Norway (Miller et al., 1983) and with the assumption of similar temperature history for the shelf and coastal areas, the absolute ages of the samples are discussed. Of the nine samples analysed one was of Holocene, seven of Late to Middle Weichselian and one sample of pre-Weichselian age.

From this pilot study we conclude that isoleucine epimerization ratios in benthonic foraminifera provide a reliable index of relative age that can be used to correlate sedimentary units that contain in situ faunas and have similar thermal histories. Absolute ages of the enclosing sediment can be approximated if suitable well-studied calibration sites are available.

INTRODUCTION

Thick sequences of Quaternary sediments cover large areas of the Norwegian continental shelf. An average thickness of about 100 m has been found off both north and mid-Norway, but with local variation from a patchy cover to more than 300 m (Rokoengen et al., 1979a; Bugge, 1980). Both the shelf morphology and the sediments strongly reflect the glacial impact through the Quaternary. The surface sediments and the stratigraphy found in gravity cores have been thoroughly studied during the last decade (Holtedahl and Bjørkli, 1975, 1982; Vorren et al., 1978; Bjørklund et al., 1979; Rokoengen et al., 1979a, b; Bugge, 1980; Sejrup et al., 1980, and others). Gravity cores

usually recover only sediments of the last deglaciation and the Holocene (ca. 13,000 yrs B.P. to Present). This is well within the range of the ^{14}C method and many such datings have been performed on carbonate fossils from the Norwegian continental shelf.

In recent years material for investigations of the older Quaternary deposits has been collected in two different ways: gravity- or vibro-coring in areas where shallow seismic data show that older strata are close to the sea bed, and drilling from ships or platforms. Many such drillings have been performed on the Norwegian continental shelf in connection with geotechnical investigations related to foundation of platforms for petroleum production.

The studies of these older sediments have revealed an urgent need for methods of correlation and age determination. Large portions of the sediments are of glaciomarine origin, and arctic faunas seem to have been repeated through different periods of glaciation without much evolution. As carbonate microfossils (mostly benthonic foraminifera) are common in these sediments, a pilot study for evaluating amino acid diagenesis in benthonic foraminifera as a help in correlation and, if possible, rough age determination was initiated.

MATERIAL

This study is based on material collected by gravity- and vibro-coring from two areas on the Norwegian continental shelf (Fig.1). The samples from the northern area have previously been described by Rokoengen et al. (1979b), and the samples in the southern area by Bugge (1980; B77-139), Rokoengen (1980; Profile II and B78-121) and Rokoengen et al. (1982; A79-146). In addition, material was analysed from two ^{14}C -dated late glacial marine sections on land in the Ålesund area.

Northern area

The material from the northern area was sampled along a SW–NE profile. Based on the seismic record, units of different ages were sampled (Fig.2). Core C76-113 was located on the outer glacial unit which was believed to represent a terminal moraine deposited after $13,310 \pm 110$ yrs B.P. (T-2326) (Rokoengen et al., 1979b). In this core normal consolidated glaciomarine sediments were found below a 45-cm thick layer of Holocene normal marine sediments (30–40 cm radiocarbon dated to 9360 ± 120 yrs B.P.; T-2879). The core did not reach overconsolidated sediments. A sample of arctic benthonic foraminifera between 70–80 cm depth in the core was chosen for amino acid analyses. A ^{14}C -date performed on molluscs from this level gave $11,770 \pm 170$ yrs B.P. (T-2528).

Core C76-112 (Fig.2) consisted of an overconsolidated clay, with a foraminifera fauna containing both boreal and arctic species. The seismic record indicated that this unit represented older Quaternary sediments (Fugløybanken drift). A sample of foraminifera from 10–20 cm depth in the core was chosen for amino acid analysis. A large number of thermophilous species in this sample was believed to have been derived from older interglacial sediments.

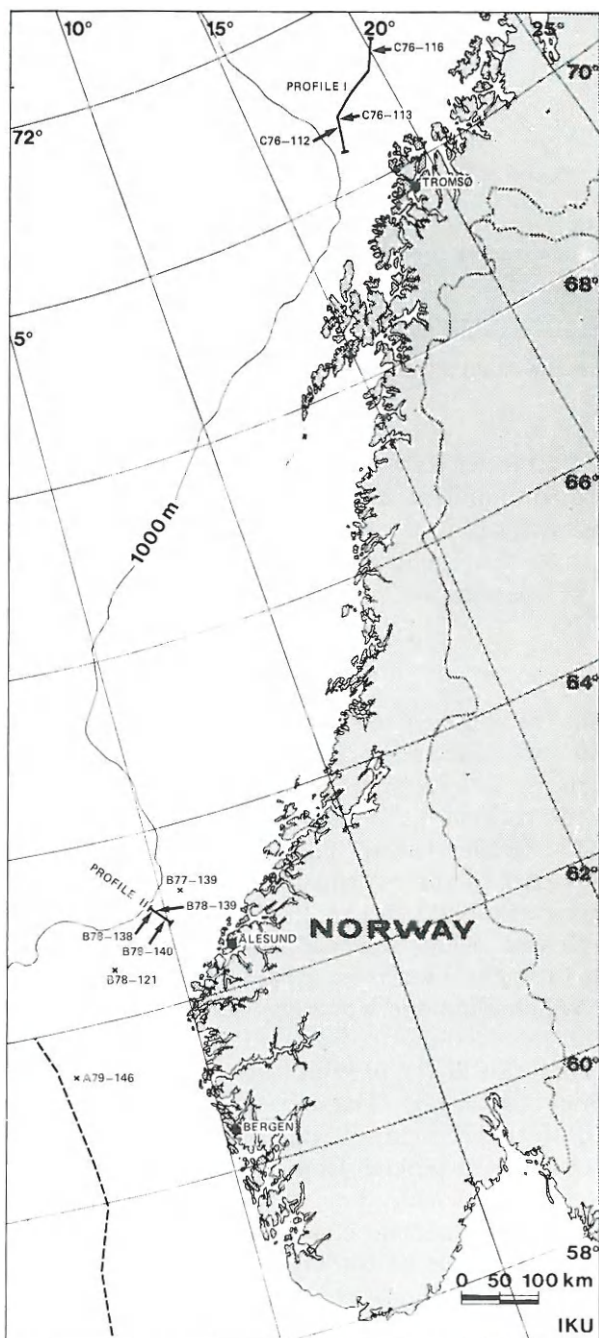


Fig.1. Location map. For core locations along the profiles, see also Figs.2 and 3.

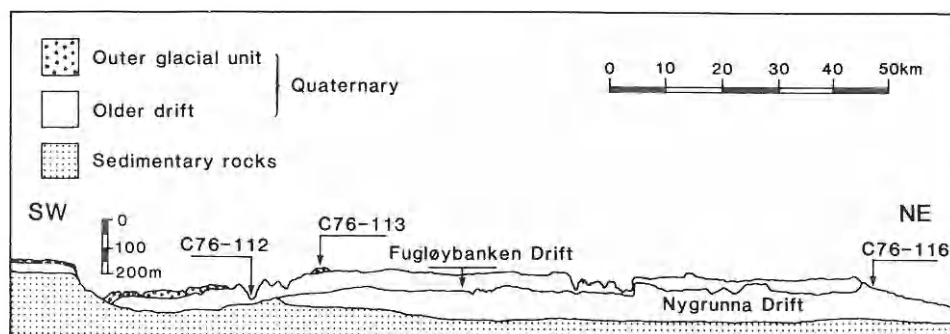


Fig. 2. Profile I, schematic seismic profile based on sparker records from the northern area. For location see Fig. 1.

Core C76-116 (raised from 240 m of water depth) also consisted of over-consolidated sediment, but the foraminifera indicated a high-arctic environment. Based on the seismostratigraphy it was supposed that this unit (Nygrunna drift) was older than the Fugløybanken drift. A foraminifera sample from 30–40 cm depth in the core was chosen for amino acid analysis.

Southern area

Six cores were collected from the southern area (Fig. 1). Core B77-139 was raised from 158 m water depth and a sample from 270–280 cm depth in the core, representing a glaciomarine unit with an arctic foraminifera fauna, was analysed. A radiocarbon date on marine molluscs from the same level gave $12,930 \pm 240$ yrs B.P. (T-2647; Bugge, 1980). Three samples were raised along profile II from the outer part of the continental shelf and the upper slope where large-scale sliding has exposed older sediments (Fig. 3). A sample from 3–5 cm in core B78-140 just below a gravelly top sand contained a low-diversity high-arctic fauna in a glaciomarine sediment. Based on correlation with other cores, a Late Weichselian age was suggested for this sample (Rokoengen, 1980).

Core B78-139 was taken from the lower part of the same seismic unit, interpreted to be ca. 40 m thick till (Fig. 3). The core was 25 cm long and contained an overconsolidated silty clay below 2 cm of gravelly sand. Foraminifera for amino acid analysis were picked from an arctic fauna from 5–10 cm in core.

In an attempt to obtain material from a lower stratigraphic unit, core B78-138 was raised from the steep upper part of the continental slope (Fig. 3). This core was 10 cm long and the upper 5 cm was a gravelly sand. The lower 5 cm contained an overconsolidated sediment with a boreal foraminifera fauna.

Core B78-121 (Fig. 1) was also raised from the upper continental slope (426 m water depth) and was 20 cm long. Based on foraminifera and seismic data it was assumed to penetrate older sediments. A foraminifera sample from 5–10 cm depth in the core was analysed.

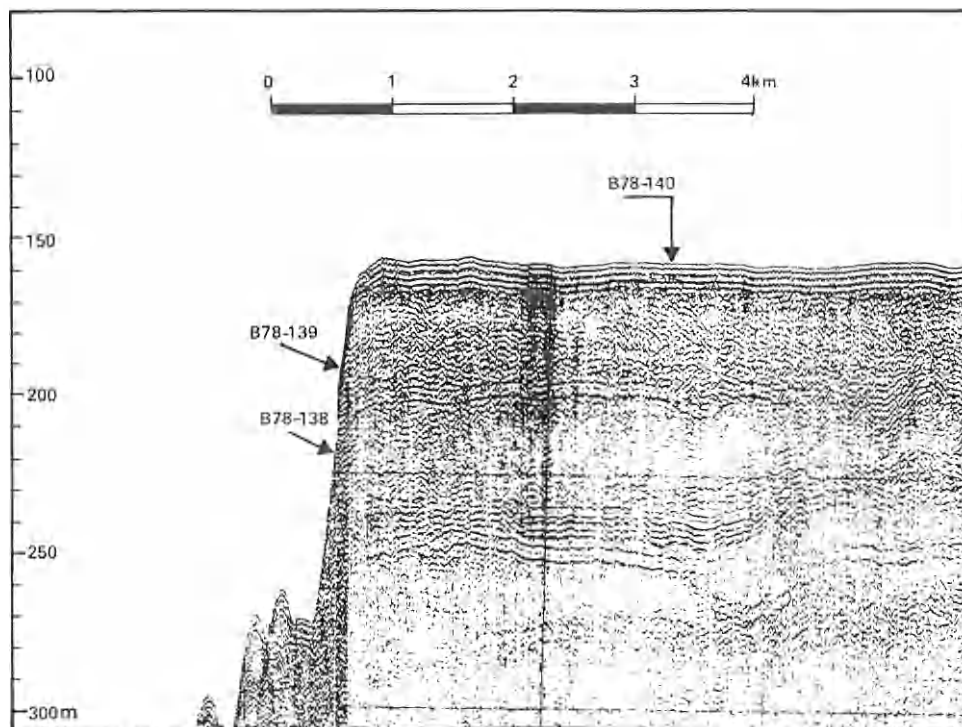


Fig.3. Profile II, sparker profile section across the shelf edge in the southern area. For location see Fig.1.

Samples B78-138 on profile II and B78-121 were both assumed to have been deposited in a period when this part of the shelf was not glaciated prior to the Holocene (Rokoengen, 1980).

Core A79-146 was raised from 144 m water depth in the northern part of the North Sea (Fig.1). Below Late Weichselian and Holocene sediments a very stiff unsorted clay was recorded (40–181 cm depth in core). This sedimentary unit is interpreted to represent a till, and mollusc fragments from it were ^{14}C -dated to $18,860 \pm 260$ yrs B.P. (T-3604). Below the till, an over-consolidated marine sediment with an arctic foraminifera fauna was found. Amino acid analysis was performed on a sample from this marine unit below the till (181–182 cm depth in core).

AMINO ACID GEOCHRONOLOGY

Since the first use of amino acid diagenesis in geochronology (Abelson, 1954, 1955) analyses of a wide range of material have been attempted (Williams and Smith, 1977). The racemization and epimerization reactions in amino acids from protein residues of carbonate fossils has received increasing attention over the last decade (Mitterer, 1974, 1975; Wehmiller and Belknap,

1978; Miller et al., 1979, 1983; Wehmiller, 1982). Most of these studies have dealt with fossil molluscs and rather few workers have focused on foraminifera. Analyses of foraminifera have mainly been performed on planktonic species found in deep-sea cores (King and Hare, 1972; Bada and Schroeder, 1972; King and Neville, 1977; King, 1980), rather few workers have given attention to benthonic species (Miller et al., 1979, 1983). The foraminiferal studies have relied mainly on the epimerization of L-isoleucine.

The reversible isoleucine epimerization reaction reaches equilibrium when the D-alloisoleucine/L-isoleucine ratio (alle/Ile) is close to 1.3 for foraminifera and molluscs (Miller et al., 1983). Living foraminifera have an Aile/Ile ratio close to zero and equilibrium values have only been found in samples older than 2 m.y. in this region (mollusc samples from Northwest Europe (Miller et al., 1983)).

Methods

The foraminifera samples used in this study have all been extracted after the method described by Feyling-Hanssen (1958). CCl_4 has been used for gravity separation and the 125–1000 μm fraction has been analysed.

Earlier investigations (Miller et al., 1983) have shown that the epimerization reaction in benthonic foraminifera (just as in many other organisms) is highly species dependent. Therefore we have analysed only monospecific samples. We have concentrated on the species *Cibicides lobatulus* (Walker and Jacob) and *Elphidium excavatum* (Terquem) f. *clavata* (Cushman) in this pilot study because of their relatively wide distribution, both in time and space along the continental shelves of Western Europe. Insufficient protein occurs in a single test for an amino acid analysis; each preparation consists of between 20 and 100 individuals dependent on their size. Thus the determination of the alle/Ile ratio is an unweighted average value for all tests, an important consideration when the possibility of mixed populations exists. The preparation of the samples follows that of Miller et al. (1983). All samples have been hydrolysed for 22 h at 110°C. The samples were then run on an automatic ion-exchange amino acid analyzer in the Amino Acid Geochronology Laboratory at INSTAAR, University of Colorado.

RESULTS AND DISCUSSION

Table I gives the alle/Ile ratios in the total (free plus peptide-bound) fraction recorded in the foraminifera samples from the continental shelf. Different laboratory numbers on the same sample refer to different pickings and preparations. Where enough material was present in one preparation, it was run twice. In Table II the alle/Ile ratios from the ^{14}C -dated localities in the Ålesund area are given.

TABLE I

The alle/Ile ratios recorded in samples from the Norwegian continental shelf

Profile	Sample No.	Depth in core (cm)	Water depth (m)	Lab. No.	alle/Ile (total)	Species	¹⁴ C-age on molluscs
II II II I I I	A79-146	181—182	144	AAL-1814	0.099	Cib.lob.	18,860 ± 260* (T-3604)
				AAL-1814	0.087	Cib.lob.	
				AAL-1815	0.085	Cib.lob.	
				AAL-1731	0.075	Cib.lob.	
	B77-139	270—280	158	AAL-1813	0.032	Elph.e.	12,930 ± 240 (T-2647)
	B78-140	3—5	158	AAL-1935	0.055	Elph.e.	
	B78-139	5—10	191	AAL-1811	0.041	Elph.e.	
	B78-139			AAL-1811	0.039	Elph.e.	
		5—10	218	AAL-1728	0.020	Cib.lob.	11,770 ± 170 (T-2528)
	B78-121	5—10	426	AAL-1800	0.080	Cib.lob.	
C76-113	70—80	198	AAL-1796	0.068	Cib.lob.		
			AAL-1797	0.064	Cib.lob.		
			AAL-2071	0.057	Cib.lob.		
			AAL-1933	0.036	Elph.e.		
C76-112	10—20	330	AAL-1798	0.341	Cib.lob.		
			AAL-1798	0.327	Cib.lob.		
			AAL-2069	0.230	Cib.lob.		
C76-116	30—40	240	AAL-1799	0.093	Cib.lob.		
			AAL-2070	0.042	Elph.e.		

*This dating was performed on molluscs from a till above the marine unit where the foraminifera samples was taken.

TABLE II

The Aile/Ile ratios in ¹⁴C-dated samples from the Ålesund area

Locality	Field No.	Lab. No.	aIle/Ile total
Hjørungavåg	1978-13	AAL 1842	0.044
	1978-16	AAL 2124	0.056
Vegsundet	1978-34	AAL 1854	0.035
			0.041
		AAL 1931	0.040

alle/Ile-ratios in *Cibicides lobatulus* from late glacial samples from the Ålesund area (Fig.1). ¹⁴C-datings which have been carried out on molluscs from these localities range from 10,380 ± 50 (T-3309) to 12,310 ± 130 (T-3125) (J. Mangerud, pers. commun., 1983) and we choose 11,000 as an age for our mean value of the Aile/Ile ratios in *Cibicides lobatulus* from these deposits.

Northern area

The present seasonal temperature variations in the northern area at depths between 200 and 300 m are between 4 and 5°C (Helland-Hansen and Nansen, 1909). The hydrographic regime in this area has probably remained relatively unchanged the last 10,000 yrs (Vorren et al., 1978; Rokoengen et al., 1979a). It is difficult to make any safe statements about the temperature conditions for the time prior to 10,000 yrs B.P. The more northerly position suggests, however, that the temperatures generally have been lower in this area than in the southern area. It should therefore be emphasized that the plotting of the alle/Ile ratios in *Cibicides lobatulus* from this area on the curve obtained from the coastal areas of western Norway (Miller et al., 1983; Fig.4) is just to visualize the data, and that the projected ages probably are too young.

The ^{14}C -date $11,770 \pm 170$ yrs B.P. (T-2528) in core C76-113 has been suggested to reflect an admixture of relatively old molluscs into the sediment (Rokoengen et al., 1979b). The relatively high alle/Ile ratio in *Cibicides lobatulus* (mean: 0.063) and low in *Elphidium excavatum* f. *clavata* (0.036) suggest that also the foraminifera in this sediment could be a mixture derived from sediments of different ages even if we consider the different rates of

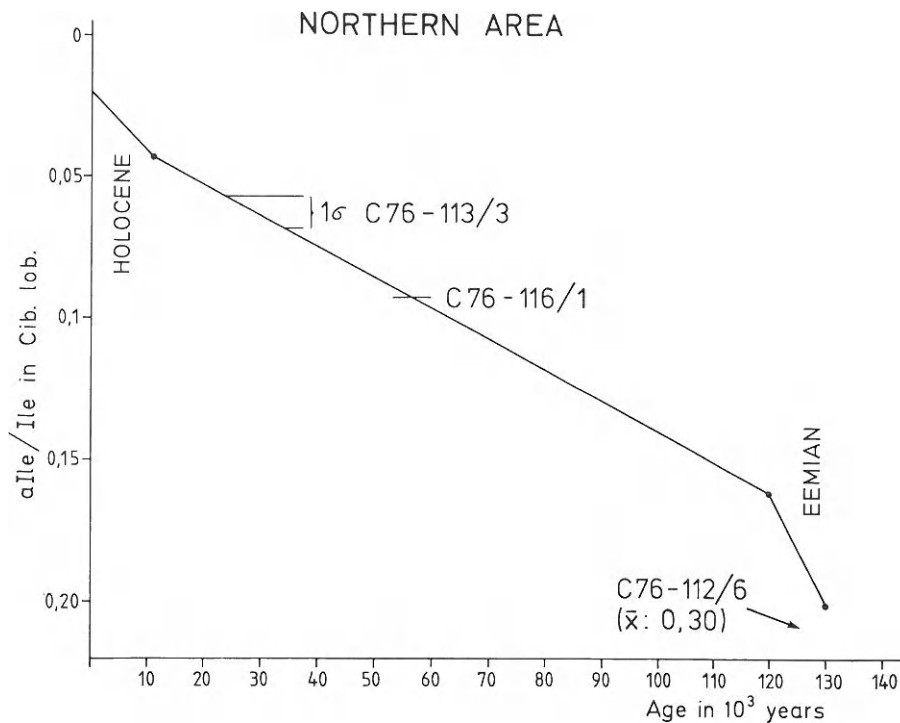


Fig.4. The alle/Ile ratios in *Cibicides lobatulus* in cores from the northern area plotted on a diagram based on analyses of this species from late glacial and last interglacial sediments in western Norway.

epimerization in these species (Miller et al., 1983). However, with this small number of analyses we cannot exclude analytical explanations for the differences.

Both core C76-116 and C76-112 were supposed to sample stratigraphic units older than the last deglaciation. The relatively high ratios in *Cibicides lobatulus* in the samples from these cores (Table I and Fig.4) suggest that this interpretation is correct. The reason for the higher ratio in the upper seismostratigraphic unit could be because both units represent tills which may contain a derived fauna of various ages. Another possibility is errors in the seismic interpretation. The ratios suggest that cores C76-113 and C76-116 contain fossils that must have lived on the continental shelf outside northern Norway in an ice-free period sometime in the Middle or Early Weichselian. The foraminifera analysed in core C76-112 indicate a much greater age. The relatively large difference between AAL-1798 and AAL-2069 (Table I) might result from mixed populations.

Southern area

The present temperature at the sampled localities in the southern area is presumed to characterize the last 10,000 yrs (Sejrup et al., 1980). The seasonal variation at these localities lies within 5–8°C (Helland-Hansen and Nansen, 1909) which is very close to the mean annual air temperature of the coastal areas of western Norway (7.5°C, Bruun, 1962). This is due to the fact that the climate of western Norway is governed by the hydrographic regime in the Norwegian Sea. However, for the time prior to 10,000 yrs B.P. it is difficult to reconstruct the thermal conditions. However, the fact that the Scandinavian ice sheet reached the shelf edge just prior to 13,000 yrs B.P. in this area (Bugge, 1980; Rokoengen, 1980) suggests, that also in earlier phases of glaciation both the shelf and coastal localities could have been covered by ice for about the same span of time. In these periods the shelf and coastal localities should have experienced the same temperature history with temperatures close to 0°C (the basis of the glacier at the pressure melting point). For the periods when the coastal localities and the shelf localities were ice-free, the coastal areas above sea level could have experienced much lower temperatures than the marine sites.

Calibration of the $aIle/Ile$ ratios in *Cibicides lobatulus* with sample age is outlined in Miller et al. (1983) and is based on ratios obtained from two marine last interglacial sites and ^{14}C -dated late glacial sites in coastal western Norway. This curve (plotted in Figs.4 and 5) can be used to provide a first approximation on the age of samples from the same area with $aIle/Ile$ ratios intermediate between the two calibration points. The $aIle/Ile$ ratios in *C. lobatulus* from the southern area have been plotted on the calibration curve in Fig.5.

It should be emphasized that the ages indicated for the older marine samples may be too old because of the possibility that the coastal sites experienced much lower temperatures than the marine localities. The $aIle/Ile$

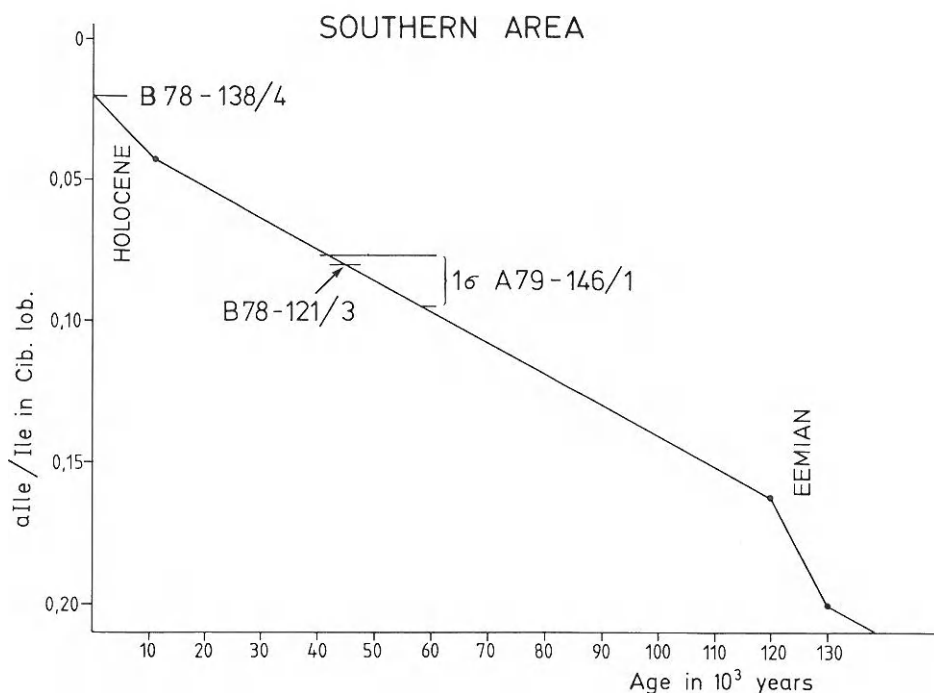


Fig. 5. The alle/Ile ratios in *Cibicides lobatulus* in cores from the southern area plotted on a diagram based on analyses of this species from late glacial and last interglacial sediments in western Norway.

ratios in *Cibicides lobatulus* from cores A79-146 and B78-121 (Table I) are intermediate between the ratios in the two calibration samples (Fig. 5) suggesting that these areas of the Norwegian continental shelf must have been ice-free at least one interval during the Middle-Early Weichselian. This agrees well with the reconstruction of ice-free periods through long parts of the Early and Middle Weichselian derived from studies of raised marine deposits in western Norway (Mangerud et al., 1981a, b; Andersen et al., 1981; Miller et al., 1983) and is supported by a ¹⁴C-date on molluscs found in the till above the sampled marine horizon in core A79-146. The similarity of alle/Ile ratios indicates that the two samples (A79-146 and B78-121) could be of about the same age, possibly 30,000–60,000 yrs old.

The samples from core B78-138 (Fig. 3) were believed to represent older Quaternary deposits. The measured alle/Ile ratio in *Cibicides lobatulus* was, however, only 0.020. The same level of epimerization is found in late Holocene and modern samples (Miller et al., 1983) and an Eemian age which was suggested by Rokoengen (1980) can be rejected. A Holocene age for this sample fits well with the fact that the foraminifera fauna resemble very closely the recent faunas in the area (Rokoengen, 1980; Sejrup et al., 1981). Sample B78-138 was only 10 cm, and infilling of animal burrows is a possible explanation for the occurrence of Holocene foraminifera in overconsolidated

sediment. The amino acid analyses show that the foraminifera are not indigenous to the older material and underlines the need to be very careful in using material from short cores to date seismic units.

The analyses of *Elphidium excavatum* f. *clavata* from the ^{14}C -dated Late Weichselian sample B77-139 gave an alle/Ile ratio of 0.032 (Table I). Late glacial samples of this species from the coast of Norway have not yet been analysed. Two analyses of *Elphidium excavatum* from sample B78-139 gave ratios of 0.041 and 0.039, only slightly higher than the ^{14}C -dated sample B77-139. *E. excavatum* from sample B78-140 gave an alle/Ile ratio of 0.055. For comparison it should be mentioned that this species from the Eemian/Early Weichselian boundary in western Norway yielded 0.13 (Miller et al., 1983). For all the three *Elphidium excavatum* samples analysed in the southern area (B77-139, B78-139 and B78-140) a Late Weichselian age seems reasonable from the amino acid data. On samples from the same supposed till unit, however, there are variations in the alle/Ile ratios from 0.032 to 0.055. Probably this is caused by mixing of two or more populations of foraminifera, as could be expected in till deposits.

CONCLUSIONS

Although few samples have been analysed (nine samples from the shelf and three from the Ålesund area) the following conclusions may be drawn from this pilot study:

(1) Even if the uncertainties in temperature history make conclusions on the absolute age difficult, the results from both the northern and southern areas provide evidence supporting the concept that ice-free periods with an arctic ocean existed in the Middle and/or Early Weichselian.

(2) Within a limited geographic area over which a similar temperature history can be assumed, amino acid analyses on benthonic foraminifera can be used to correlate sedimentary units deposited during periods when the shelf was not glaciated (sediments found in core A79-146 and B78-121).

(3) Where other evidence points to an interglacial age for a given sedimentary unit, the amino acid analyses can determine which of the two possibilities is most reasonable (e.g. B78-138) Eemian or Holocene?

(4) The amino acid method might be useful in correlating seismostratigraphical units from different areas.

(5) To achieve better age estimates, additional cores with a larger range in ^{14}C -dates on in situ molluscs from the shelf should be analysed. The alle/Ile ratio in foraminifera from a well-defined interglacial sequence on the shelf should also be determined to verify if our assumption of an equal temperature history through the Weichselian for the shelf and the coastal areas of western Norway is correct.

(6) Because of the frequent occurrence of benthonic foraminifera in shelf sediments, the rare occurrence of material dateable by other methods, and thick sequences of Quaternary sediments, the isoleucine epimerization reaction in benthonic foraminifera could be an important geochronological tool in future studies of the sedimentary history of shelf regions.

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