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Improving the Reproducibility of Stable Isotope Records from Planktonic and Benthic Foraminifera

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IMPROVING THE REPRODUCIBILITY OF STABLE ISOTOPES RECORDS FROM PLANKTIC AND BENTHIC FORAMINIFERA .

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Abstract

Foraminiferal oxygen and carbon stable isotope measurements have become an indispensable part of palaeoceanography. It is therefore important to understand and to improve the reproducibility of these measurements. We have estimated the reproducibility of oxygen and carbon stable isotopes of the planktic foraminifera, *Globigerina bulloides* and *Neogloboquadrina pachyderma (sinistral)* and the benthic foraminifera *Uvigerina peregrina.* To obtain stable isotope results from planktic foraminifera with a reproducibility of better than ±0.2‰ we suggest that: (1) tests should be picked from discrete size fractions of less than ±25μm standardised for each species and ocean, and (2) that at least 30 tests are measured per sample. Isotope measurement of individual tests of the benthic foraminifera *U. peregrina* heavier than 25 μg give good reproducibility. Below 25 μg both standards and measurements of individual tests show a deviation of up to 0.7‰ from the the 95% confidence limits of larger samples. This we believe is due to a memory effect in the mass spectrometer source. This effect can be reduced by allowing a longer pumping out time of at least 100 seconds between the standard and sample gas measurements. This, however, increases measurement time to 20 minutes per sample, and it can not guarantee reproducible $\delta^{13}C$ as there are vital effects associated with growth below 25 μg.

INTRODUCTION

Oxygen and carbon isotope analysis has become one of the most important tools in the study of palaeoceanography (e.g., Shackleton & Opdyke, 1973 and 1976; Shackleton, 1976; Shackleton & Pisias, 1985; Duplessy et al., 1988; Curry et al., 1988; Duplessy et al., 1993). Over the last decade there has been a significant improvement in mass-spectrometer technology measurement with reasonable machine accuracy of stable isotopes on single foraminifera tests.

There are, however, practical limits to the interpretation of isotopic measurements on small numbers of foraminifera because of the effects of ontogeny, seasonality, sampling and bioturbation. It is imperative that these limits are explored, as stable isotopes are now being employed beyond interglacial-glacial stratigraphy: For example oxygen isotopes, can be used to calculate local changes in sea surface salinity (Duplessy et al., 1991, 1992 and 1993; Maslin, et al., 1995a). For the North East Atlantic it has been shown that an analytical error of ± 0.1‰ in $\delta^{18}O$ represents a possible error of up to ±0.3‰ in the salinity estimate (Maslin, et al., 1995a). As the maximum salinity shift recorded in the North East Atlantic is 3‰ this represents an error of ±10%, similarly carbon isotopes, can be used to calculated the carbon shift from the oceans to the terrestrial biosphere since the Last Glacial Maximum (LGM). An error of ±0.1‰ in the $\delta^{13}C$ can cause an error of ±120 GtC, which represents an error of between ±10% and ±20% (Maslin et al., 1995b).

We present here experimental stable isotope results from both planktic and benthic foraminifera to determine: (1) the optimum test weight or size, and (2) the number of foraminifera tests, which should be measured to obtain reasonable reproducibility.

METHOD

For this study, material was used from a piston core, BOFS 5K (50°41.3'N, 21°51.9'W, water depth 3547 m) recovered from the East Thulean Rise in the North East Atlantic (McCave, 1989; Maslin, 1993; Manighetti, 1993). The core was sampled

in slices of 1 cm thickness. Alternate samples were disaggregated by soaking and gentle shaking in distilled water overnight, then washed through a 63μm sieve. After washing, the coarse fraction was oven dried at 60°C and weighed. Samples were then dried sieved at discrete size fractions and foraminifera picked out for isotope analysis. The planktic foraminifera species *Globigerina bulloides* (d'Orbigny) and *Neogloboquadrina pachyderma* (Ehrenberg) (left coiling = *s*) were picked from depths where their abundances were greatest to minimise the effect of bioturbation (Maslin, 1993): *G. bulloides* (30%) at 0-1 cm during the late Holocene, and *N. pachyderma (s)* (92%) at 82-83 cm during the LGM. The benthic foraminifera *Uvigerina peregrina* was picked from 187-188 cm (mid Marine Isotope Stage 3) where they were in abundance (Thomas et al., 1995). The samples were analysed in a VG PRISM mass-spectrometer using a VG ISOCARB automatic common acid bath containing 100% orthophosphoric acid at 90°C. The sample CO_2 and reference gases are admitted to the mass-spectrometer for analysis alternating between them 12 times. The average result is then corrected for instrumental effects and for the nature of the mass spectra, calibrated by Craig (1957). The results were then calibrated to PDB using the standard NBS 19 (U.S. National Bureau of Standards). See Figure 1 for summary of the method.

PLANKTIC FORAMINIFERA

Test Size Effect

Planktonic foraminifera display greater isotopic variation than benthics, due to the wider range of water mass that they can inhabit and because of vital effects caused by symbionts (Wefer, 1983; Wefer & Berger, 1991). The stable isotopic composition of planktic foraminifera changes with test size, due to alteration in habitat, with ontogeny and changes in metabolic activity (Berger et al., 1978; Fairbanks et al., 1980; Kahn & Williams, 1981; Hemleben et al., 1988; Erez & Honjo, 1991; Sautter & Thunell, 1991; Wefer & Berger, 1991).

In order to investigate the effects of size variations, samples were split into five size fractions and fifty specimens of *G. bulloides* (Holocene sample) and *N. pachyderma (s)* (LGM sample) were picked from each size fraction. Stable isotopes values were measured individually for each sample of fifty tests. Figure 2 shows that for both species $\delta^{18}O$ becomes heavier as test size increases. This is a predictable pattern caused by planktic foraminifera living deeper in the water column as they get older (Hemleben et al., 1988). The $\delta^{18}O$ range of the late Holocene *G. bulloides* is 0.65‰ (Fig. 2A) representing a 3°C temperature change (using the temperature equation of Shackleton, 1974). This is equivalent to a depth change of up to 75 m at the present site in the North East Atlantic (Pflaumann, et al., 1996), and is comparable to 80 m total depth range given for *G. bulloides* by Hemleben et al. (1988). The $\delta^{18}O$ range of *N. pachyderma (s)* is 0.90‰ (Fig 2B) representing a temperature change of 4°C. According to the SIMMAX modern analog technique for calculating sea surface temperatures (Pflaumann, et al., 1996) the closest modern analog to the LGM planktic foraminifera assemblages of BOFS 5K are in the mid-Greenland Sea. This temperature shift would represent a depth change much greater than 75 m. A more precise depth estimate is not possible because of the complexity of the analog area due to seasonal sea ice (Uwe Pflaumann, pers. comm.).

There was a more complicated, **S** shaped, relationship between test size and carbon isotopes (Fig. 3). Our results are comparable to the measurements of other planktic foraminifera species and the resultant theoretical model (Berger et al., 1978; Wefer & Berger, 1991). Wefer & Berger (1991) reiterated the suggestion that changes in the carbon isotopes associated with the growth of the foraminifera is due to changes in metabolic activity and not environment.

The wide range of isotopes results which can be obtained simply by varying the test size selected. suggests that isotopic measurements should be based on carefully size selected specimens. This is an important consideration when comparing isotopic records from different studies, and suggests standard sizes should be defined for each species. Standard size fractions now used at Cambridge, based on this study, for

samples from the North East Atlantic are for *G. bulloides* 300-350μm and for *N. pachyderma (s)* 250-300μm. These have been found to be a good compromise between availability of the species and the associated errors; This is especially noticeable with the carbon isotope results as the variation seems to plateau with the larger tests. These size range are used in the subsequent investigations in this study.

Sample Size

A determining factor in the reproducibility of the planktic foraminifera stable isotope signal is the number of tests measured. Individual foraminifera in a sample may have inhabited very different water masses in life. This is because a sample may represents at best a hundred years of sedimentation. There is, thus, a compound problem of combining the isotopic signal of foraminifera which lived during not only different seasons but also years. In the case of BOFS 5K each 1 cm slice represents a time-span of 90 to 170 years (Maslin, 1993).

Schiffelbein & Hills (1984) investigated this problem theoretically by calculating the analytical precision at different confidence limits for varying numbers of foraminifera test per sample, using the 'Jackknife' technique (see Miller, 1974, for full review of the technique). They found that obtaining reproducibility of 0.1‰ at 90% confidence would require the measurement of 417 specimens. A reproducibility of 0.15‰ it would require 55 specimens. They also showed that the 15 individuals benthic foraminifera measured by Shackleton & Opdyke (1976) in core V23-239 had a reproducibility of 0.25‰ (90% confidence limit).

To investigate the effect of sample size, sensitivity experiments were performed by measuring the stable isotopic composition of a distinct number of foraminifera five times to obtain a standard deviation. Both *G. bulloides* and *N. pachyderma (s)* results show a rapid decrease in the standard deviation of the sampling distribution (σ_N) with increasing numbers of specimens (Fig 4.A & B). These results are lower than those of Schiffelbein & Hills (1984) due to the lower population variability in our samples from the North East Atlantic, compared with those of Pacific. The most variable $\delta^{18}O$

measurements of *G. bulloides* were the samples containing one specimen, with an error of ±1.2‰. This is comparable with the glacial-interglacial shift in oceanic $\delta^{18}O$ due to changes in ice volume of over 1.2‰ (Labeyrie et al., 1987; Shackleton, 1987; Fairbanks, 1989). The carbon isotope errors are similar to those for the oxygen isotopes, but are larger relative to the down core interglacial-glacial $\delta^{13}C$ variation which has a maximum variation of 1.4‰, while $\delta^{18}O$ has a maximum variation of more than 3‰.

The standard deviation of the sampling distribution is normal even if the population distribution is not (Sage & Melsa, 1971). The standard deviation of the sampling distribution can thus be used to calculate the standard deviation of the population distribution (i.e., the whole sample) using the relationship (Sage & Melsa, 1971):

$$\sigma_N^2 = \sigma^2 / N$$

when:

σ_N = standard deviation of the sampling distribution with N test measured per sample,

σ = variation of the population distribution,

N = number of tests in per sample

The variation of the population distribution (whole sample N=35) of BOFS 5K was for; the late Holocene *G. bulloides* ±1.00‰ $\delta^{18}O$ and ±0.71‰ for $\delta^{13}C$, and for the LGM *N. pachyderma (s)* ±0.54‰ for $\delta^{18}O$ and ±0.98‰ for $\delta^{13}C$. Variation of the population distribution (whole sample) indicates the overall effects of ontongy, seasonality, sampling and bioturbation in the sample. A key objective of future studies will be to assess how this variance of the whole sample changes down core and between cores.

It appears that to obtain a reasonable stable isotope estimates, with error below 0.2‰ per measurement (95% confidence limit) then **at least** 20 specimens must be measured per sample in the North East Atlantic; if this is not possible due to the lack of foraminifera in the sample then authors should note the exact number used. It is also important that the number of specimens measured per sample is kept constant down-

core, since varying this adds another time-dependant noise function, which can not be taken into account by bioturbation deconvolution models (Bard et al., 1990 and Manighetti et al., 1995) or adaptive filtering (Trauth, 1995a and b). As standard we measure at Cambridge 30 planktic foraminifera per sample.

BENTHIC FORAMINIFERA

Stable isotopes measurements from benthic foraminifera should be more reproducible than planktics as the sediment they inhabit is bathed in deep water of near constant temperature (Shackleton, 1974). A major cause of variability in benthic stable isotopes is bioturbation (Zahn et al., 1986; Vogelsang, 1990). Vogelsang (1990) showed in cores from the Greenland Sea that repeat measurements of single *Cibicidoides wuellerstorfi* gave a range of up to 0.8‰ during both the LGM and the late Holocene. We, however, wished to investigate the intrinsic isotopic variability of a benthic foraminifera species, the bioturbation problem already being well documented (e.g. Zahn et al., 1986; Loubere, 1987; Vogelsang, 1990). To try and minimise the bioturbation effect we selected a sample from an interval with a high sedimentation rate and with no adjacent major climatic changes. The sample at depth 187-188 cm was chosen (mid Stage 3), as it has abundant benthics, a sedimentation rate of 8 cm/kyrs (Maslin, 1993; Manighetti, et al., 1995), and is at least 20 cm from the nearest Heinrich (ice rafting) event (Thomas et al., 1995; Maslin et al., 1995a). *Uvigerina peregrina* was selected as it is the benthic species most widely used in palaeoceanography (e.g., Shackleton, 1974, 1976; Zahn et al., 1986; Zahn et al., 1986 and 1991). We have used benthic foraminifera test weight as it is a better measure of ontogeny than size fraction (E. Thomas, pers. comm.). Each test was weighed using a Cahn26 microbalance and analysed singularly. Dunbar & Wefer (1984) analysed nine benthic foraminifera species at six different size fractions and found no influence of size on isotopic composition.

For single Uvigerina *peregrina* specimens it was found that above 25 µg the reproducibility of $\delta^{18}O$ and $\delta^{13}C$ was within the 95% confidence limits which were

estimated from repeat measurements of individuals larger than 99 μg (Fig 5). Below 25 μg the $\delta^{18}O$ and $\delta^{13}C$ of both single *Uvigerina peregrina* specimens (Fig 5) and the Carrcen Z standard show marked deviations from the 95% confidence limits. After extensive investigation we have come to the conclusion that this deviation is caused by a memory effect in the mass spectrometer source (N.B. not the common acid bath). This speculation was tested by repeating the Carrcen Z measurements but with a much longer pump-out time between the standard gas and the sample gas measurements. By using a pump-out time of 100 seconds (about 20 minutes per sample) compared to the normal 15 seconds (3 minutes per sample) we were able to remove most of the memory effect (Fig. 7). We believe this approach will be successful when used to measure the $\delta^{18}O$ of small *Uvigerina peregrina* as the deviation is consistently negative. Unfortunately due to the complex nature of the memory effect it has not been possible to devise a correction to the existing measurements. The $\delta^{13}C$ of small *Uvigerina peregrina* on the other hand deviates both negatively and positively, suggesting that below weights of 30-25 μg there are vital effects probably due to changes in metabolic activity associated with growth.

Benthic foraminifera growth and standing stocks are strongly influenced by the input of phytodetritus (i.e., food) from the surface waters (Gooday & Turley, 1990). In the North East Atlantic the isotopic composition of the phytodetritus is strongly influenced by the occurrence, intensity and duration of the spring blooms (Gooday, 1988; Gooday et al., 1992; Thomas et al., 1995). It has been suggested that changes in the isotopic composition of the phytodetritus may have an important effect on benthic $\delta^{13}C$ (Thomas et al., 1995). This could explain why the standard deviation of $\delta^{13}C$ for the larger single *Uvigerina peregrina* (-0.66‰ ± 0.22‰) is greater than that for $\delta^{18}O$ (4.45‰ ± 0.17‰). It also suggests that care should be taken interpreting down core benthic $\delta^{13}C$ records in areas with a large range of surface water productivity.

It appears that reasonably reproducible stable isotope estimates of single *Uvigerina peregrina* can be obtained if individuals larger than 25 μg are used. If reproducible $\delta^{18}O$ is required on individual *Uvigerina peregrina* below 25 μg then we

recommend a pump-out time between the standard gas and the sample gas measurements of at least 100 seconds.

CONCLUSION

If $\delta^{18}O$ and $\delta^{13}C$ are to be used beyond interglacial-glacial stratigraphy, i.e., for the determination of surface water salinity or global carbon storage, then this type of error analysis is essential.

For reliable stable isotope records we suggest that:

1/ Planktonic foraminifera must be picked from a discrete size fraction and where possible this should be made a standard for each species to allow direct comparison of isotopic records,

2/ Thirty planktic foraminifera should be measured if possible at each sample depth, if not the authors should make it clear how many were used and the possible errors associated. It is also recommended that the number of specimens analysed per sample should be kept constant down core to reduce possible noise.

3/ *Uvigerina peregrina* benthic foraminifera tests of weights greater than 25 μg should be used. If reproducible $\delta^{18}O$ is required on individual *U. peregrina* below 25 μg then we recommend a pump-out time between the standard gas and the sample gas measurements of at least 100 seconds.

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FIGURE CAPTIONS

Figure 1.

Preparation and analysis sequence for oxygen and carbon stable isotopes of calcium carbonate samples in the VG Prism mass spectrometer used at the Godwin Laboratory, Cambridge, U.K.

Figure 2

Variations in oxygen isotope content of planktic foraminifera at different test sizes. Vertical error bars are 2 s.d. error associated with measuring 50 foraminifera per sample (see Fig. 4A). The horizontal bars represent the range of size fractions from which the foraminifera were picked. Dotted lines and arrows show for each species the size fraction which is used as standard in Cambridge and in subsequent experiments in this study.

Figure 3

Variations in carbon isotope content of planktic foraminifera at different test sizes. Vertical error bars are the 2 s.d. error associated with measuring 50 foraminifera per sample (see Fig. 4B). The horizontal bars represent the range of size fractions from which the foraminifera were picked. Dotted lines and arrows show for each species the size fraction which is used as standard in Cambridge and in subsequent experiments in this study.

Figure 4

Various numbers of planktic foraminifera were analysed for $\delta^{18}O$ and $\delta^{13}C$, each with five repeats to obtain a standard deviation of the sampling distribution (σ_N), to determine the error associated with analysing small numbers of planktic foraminifera tests. Note that the weight of the small samples was always above 25 µg therefore avoiding the memory effect of the mass spectrometer source (see text).

Figure 5

Oxygen and carbon isotopic values of different weights of individual *Uvigerina peregrina* benthic foraminifera tests using the standard 15 seconds pump-out time between the sample gas and standard gas measurements. A) below 25 μg for $\delta^{18}O$ there is a strong negative deviation away from the 95% confidence limits, which were estimated from repeat measurements of samples weighing greater than 50 μg. B) below 30 μg for $\delta^{13}C$ there is initially a positive deviation and then a negative deviation away from the 95% confidence limits.

Figure 6

Oxygen and carbon isotopic values of different weights of the standard Carrcen Z using the standard 15 seconds pump-out time between the sample Carrcen Z gas and standard gas measurements. Note that below 25 μg there is a strong deviation away from the 95% confidence limits, estimated from repeat measurements of samples weighing greater than 99 μg.

Figure 7

Oxygen and carbon isotopic values of different weights of the standard Carrcen Z using 100 seconds pump-out time between the sample Carrcen Z gas and standard gas measurements. Note that the strong deviation below 25 μg away from the 95% confidence limits has been greatly reduced.

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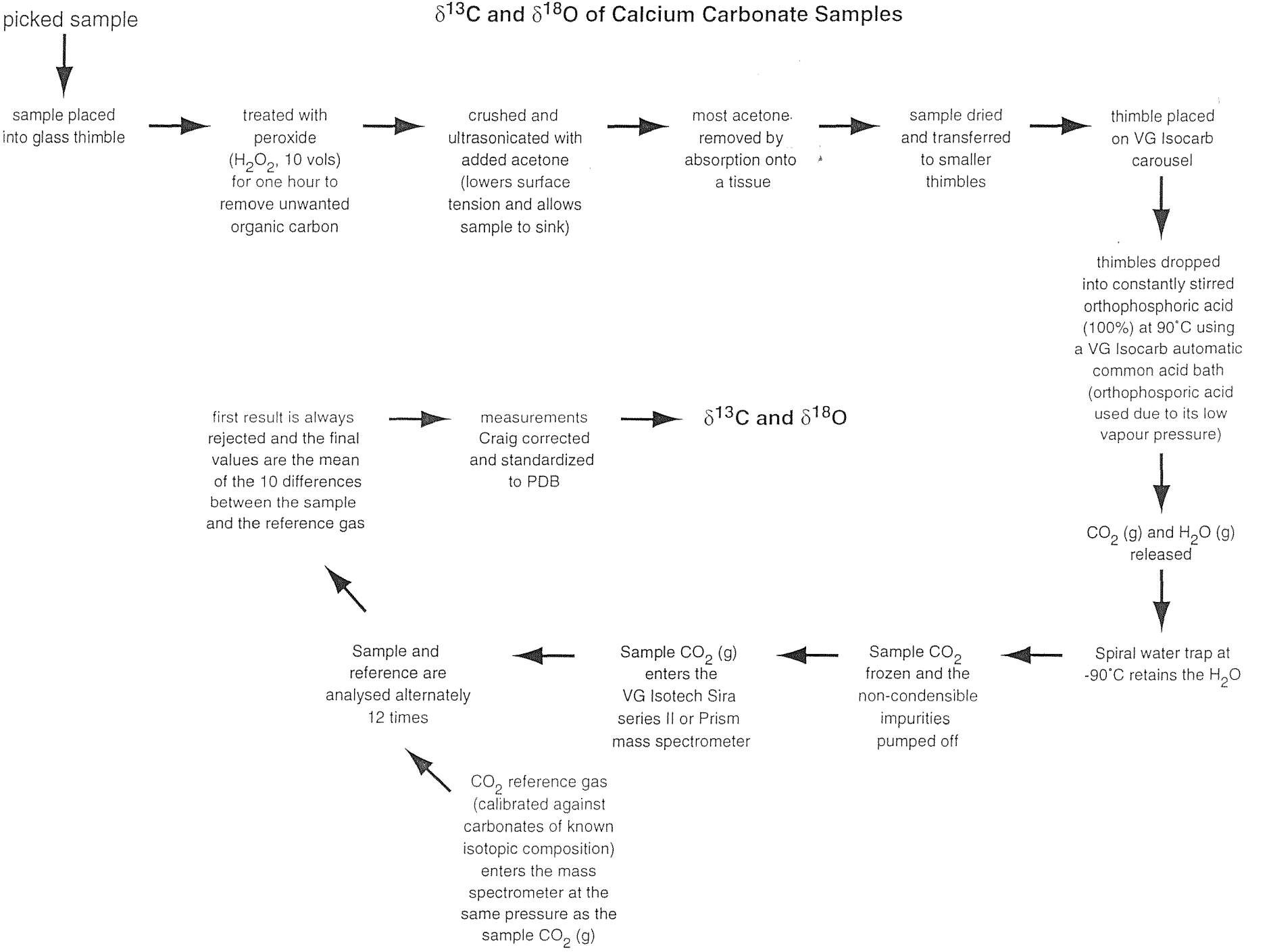
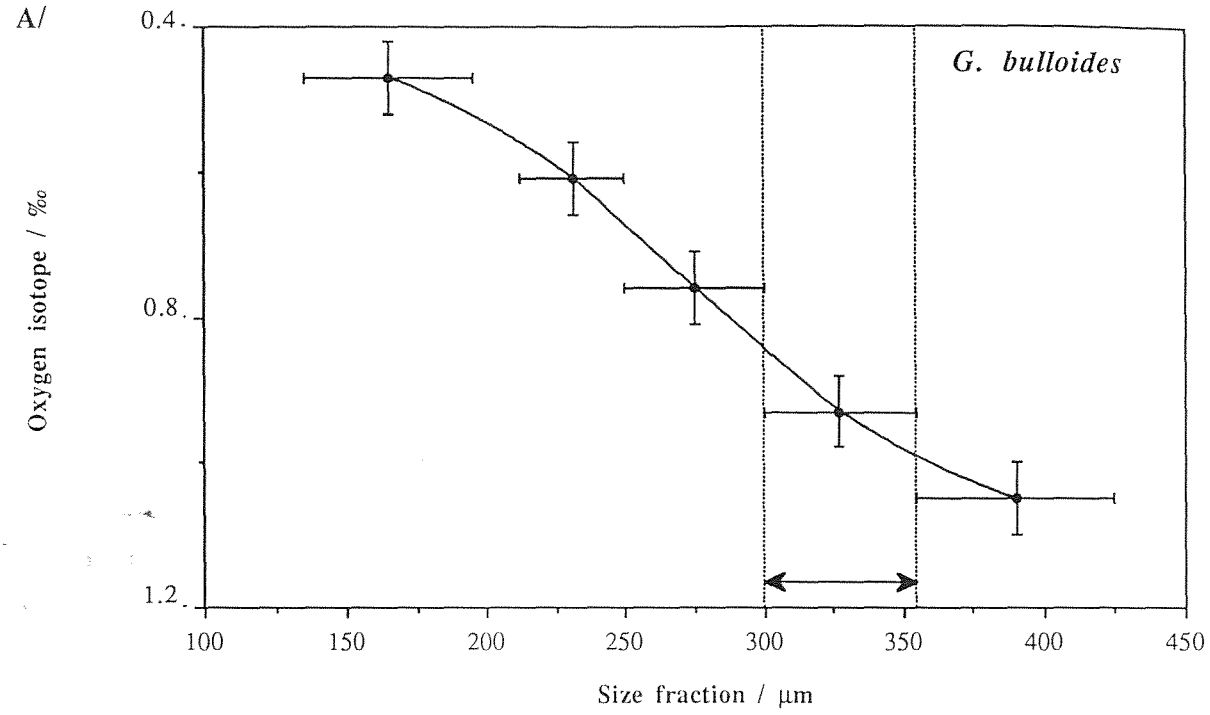
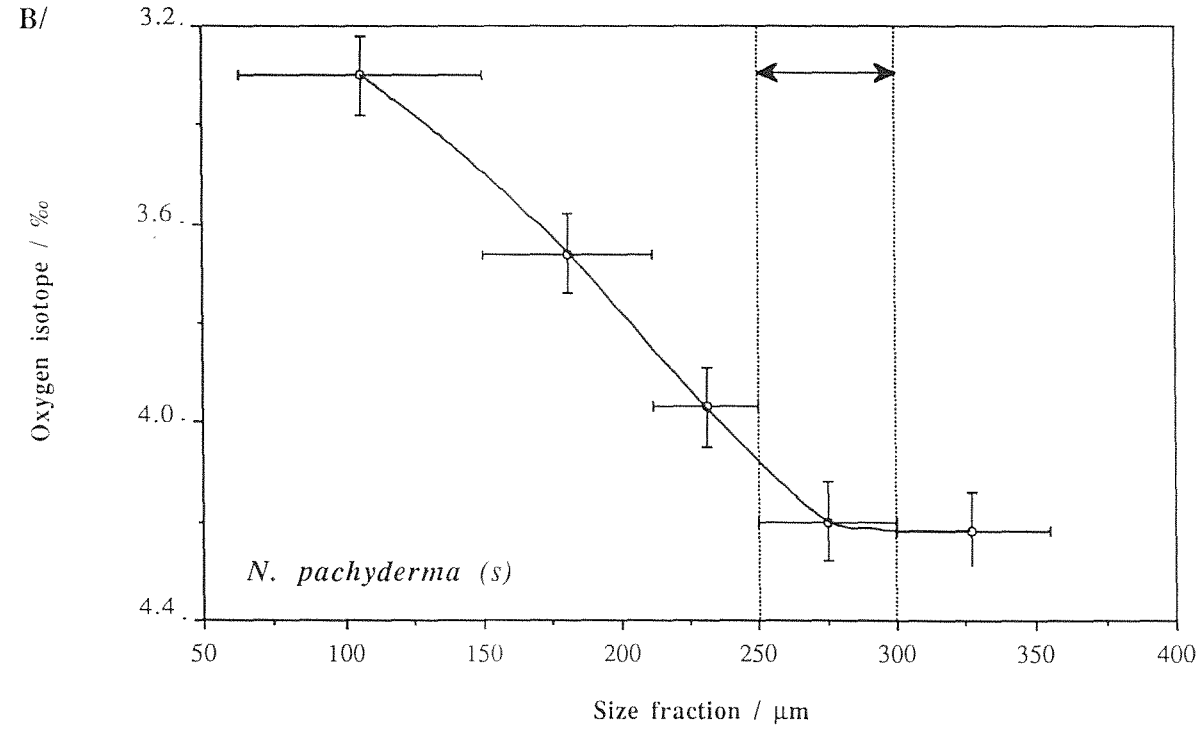
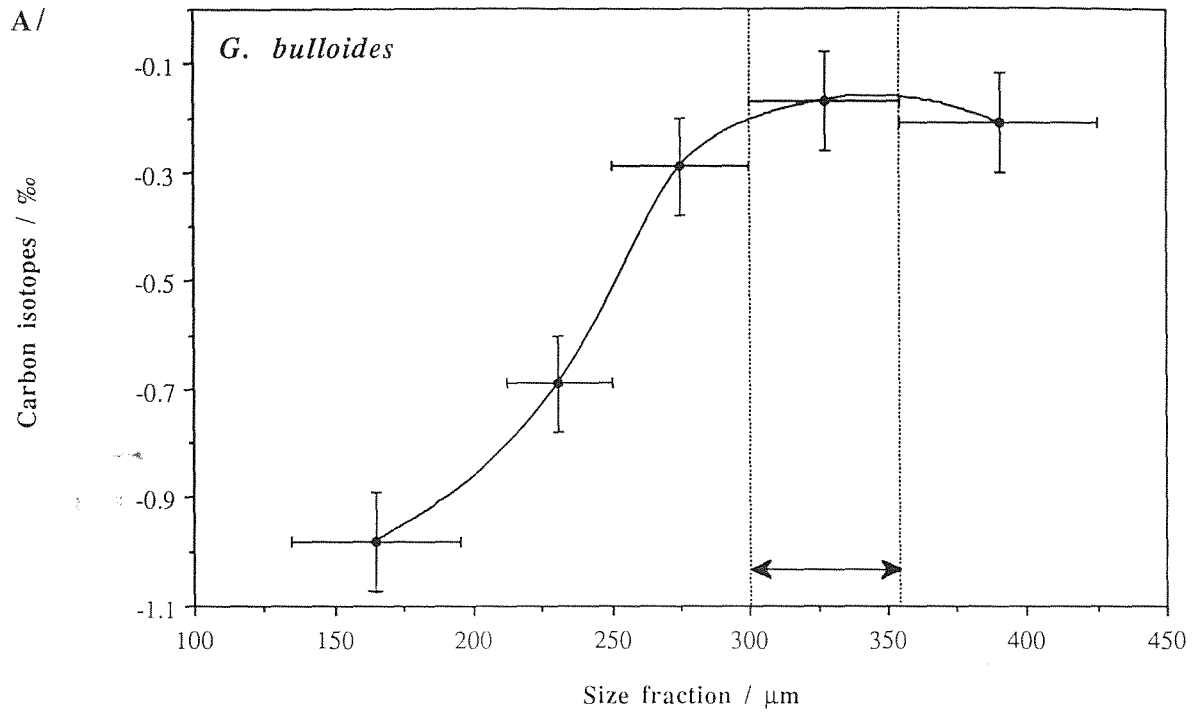
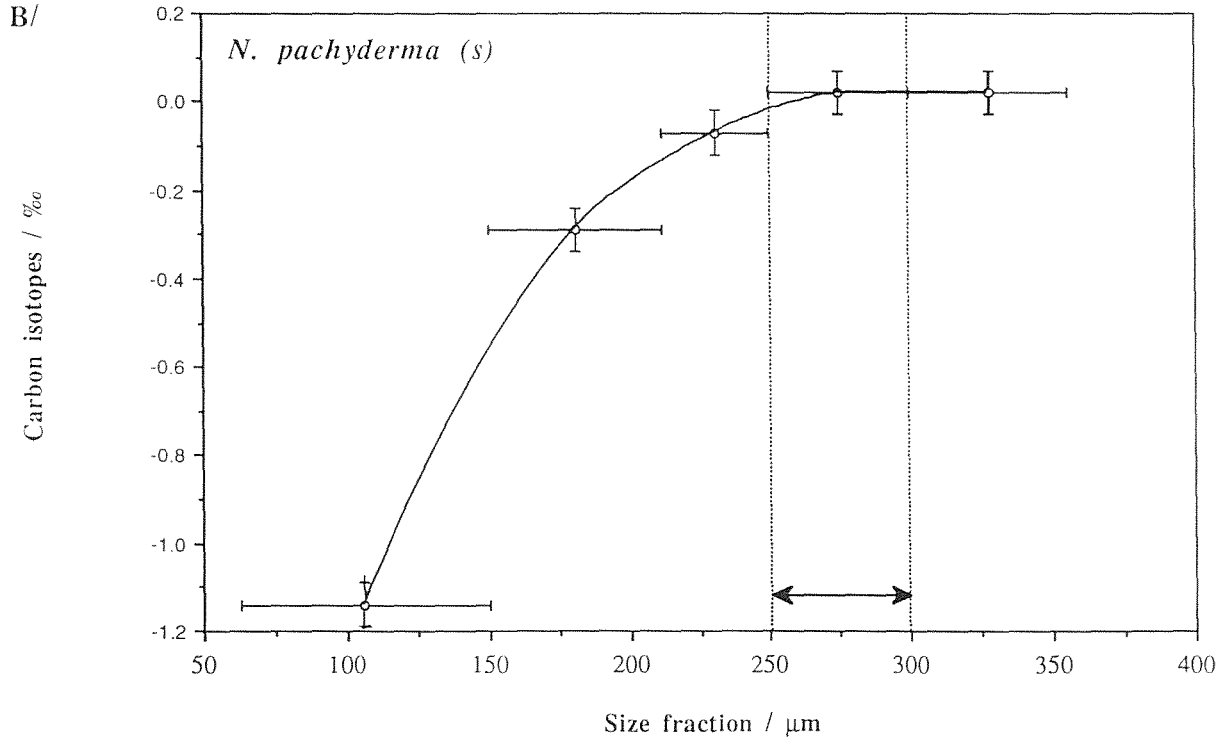
Fig. 1 Mass spectrometer: Preparation and Analysis of $\delta^{13}C$ and $\delta^{18}O$ of Calcium Carbonate Samples



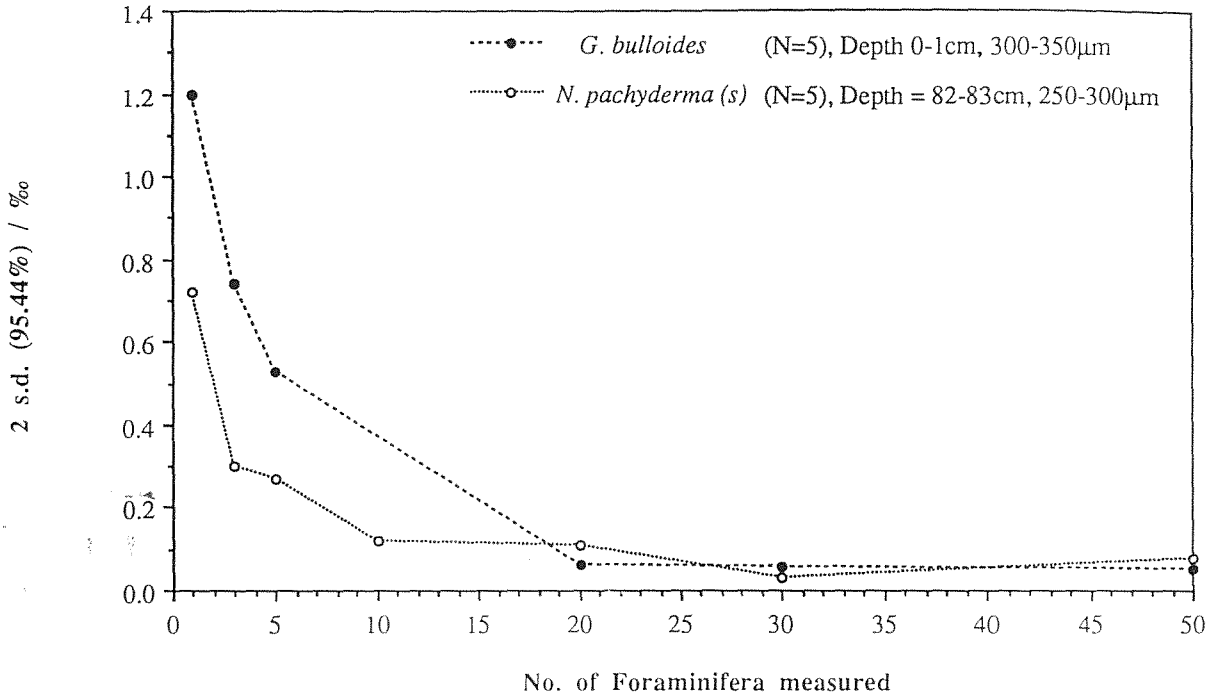

A/
0.4
0.8
1.2
Oxygen isotope / ‰
G. bulloides
100
150
200
250
300
350
400
450
Size fraction / µm


B/
3.2
3.6
4.0
4.4
Oxygen isotope / ‰
N. pachyderma (s)
50
100
150
200
250
300
350
400
Size fraction / µm

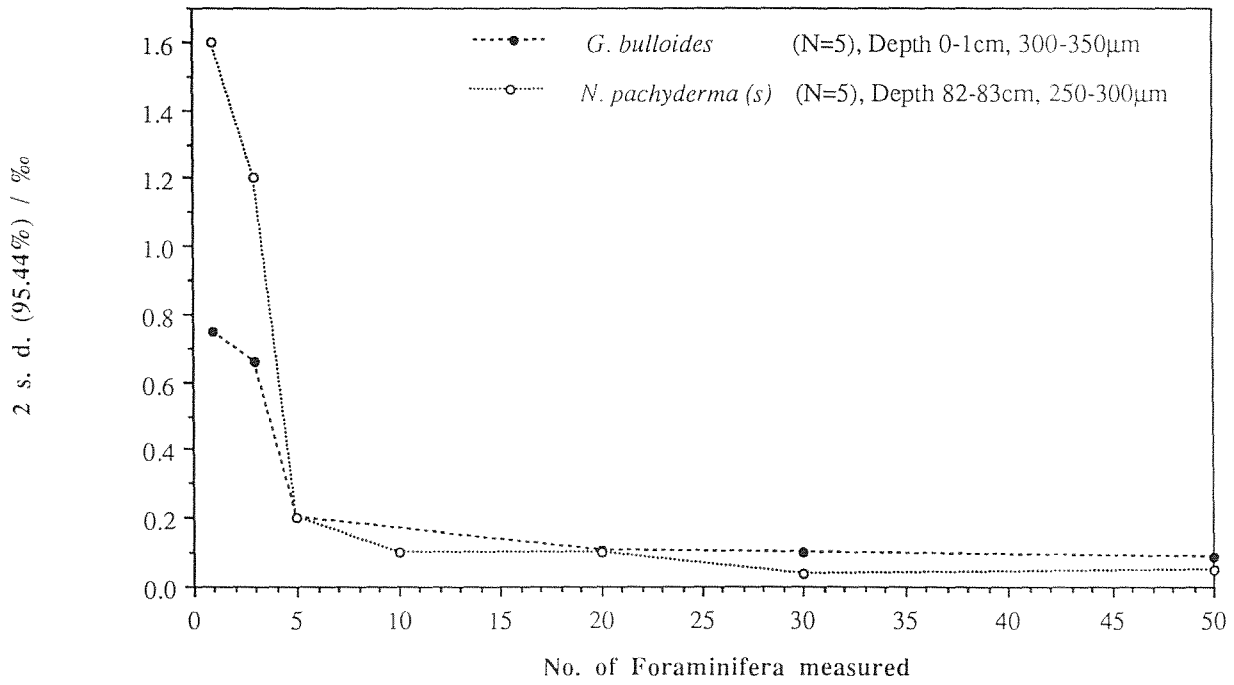

A/
G. bulloides
Carbon isotopes / ‰
-0.1
-0.3
-0.5
-0.7
-0.9
-1.1
100
150
200
250
300
350
400
450
Size fraction / µm


B/
N. pachyderma (s)
Carbon isotopes / ‰
0.2
0.0
-0.2
-0.4
-0.6
-0.8
-1.0
-1.2
50
100
150
200
250
300
350
400
Size fraction / µm

A/ Oxygen isotopes

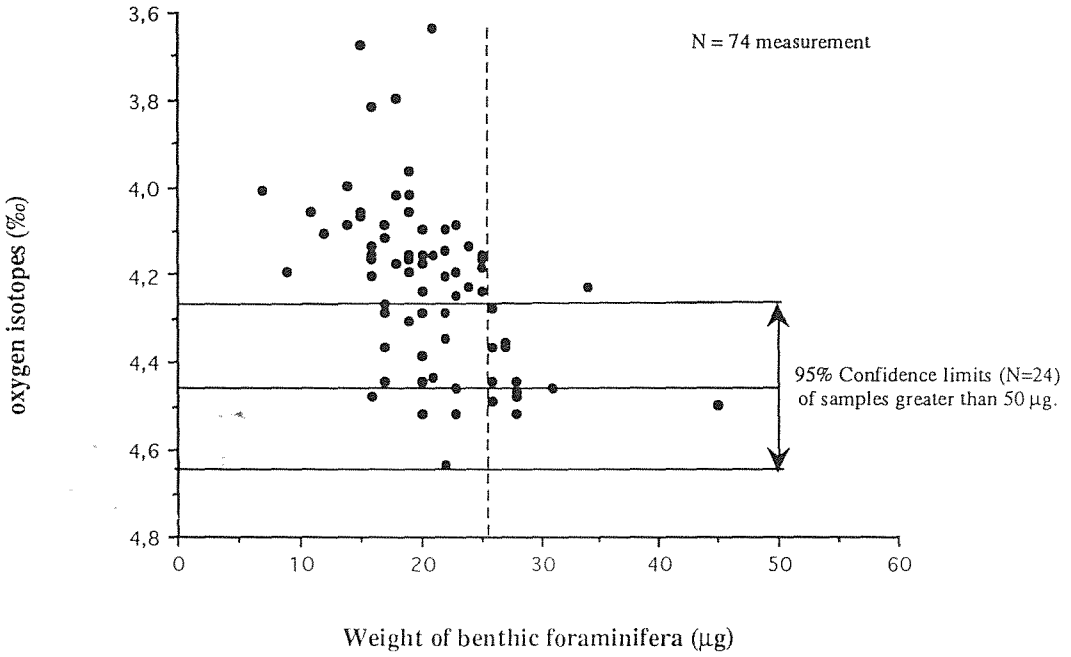


B/ Carbon isotopes

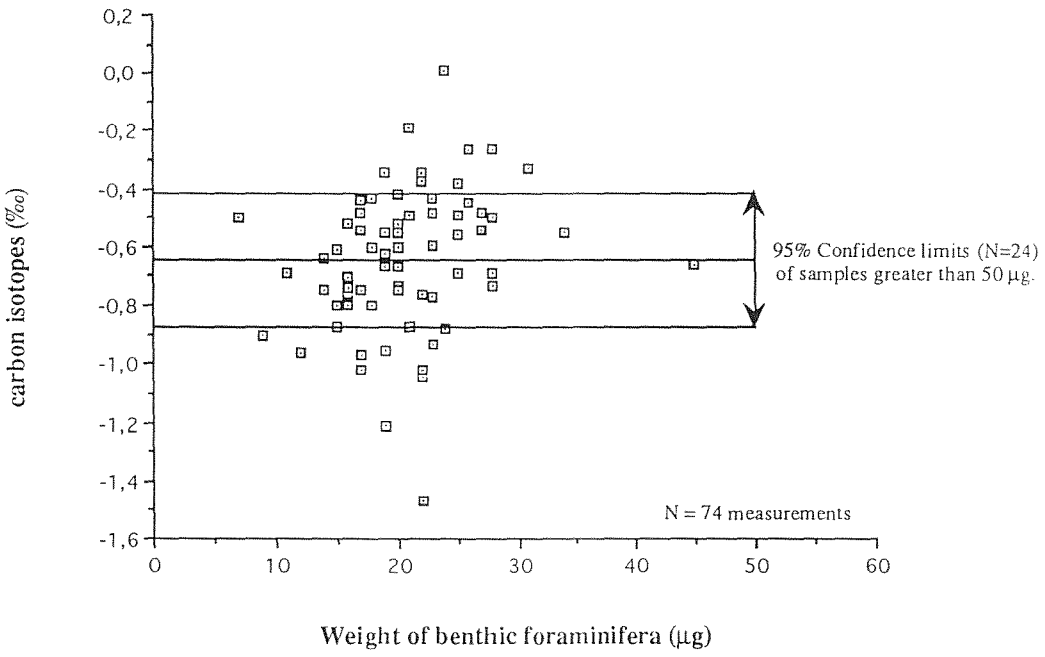


Single benthic foraminifera isotopic measurement

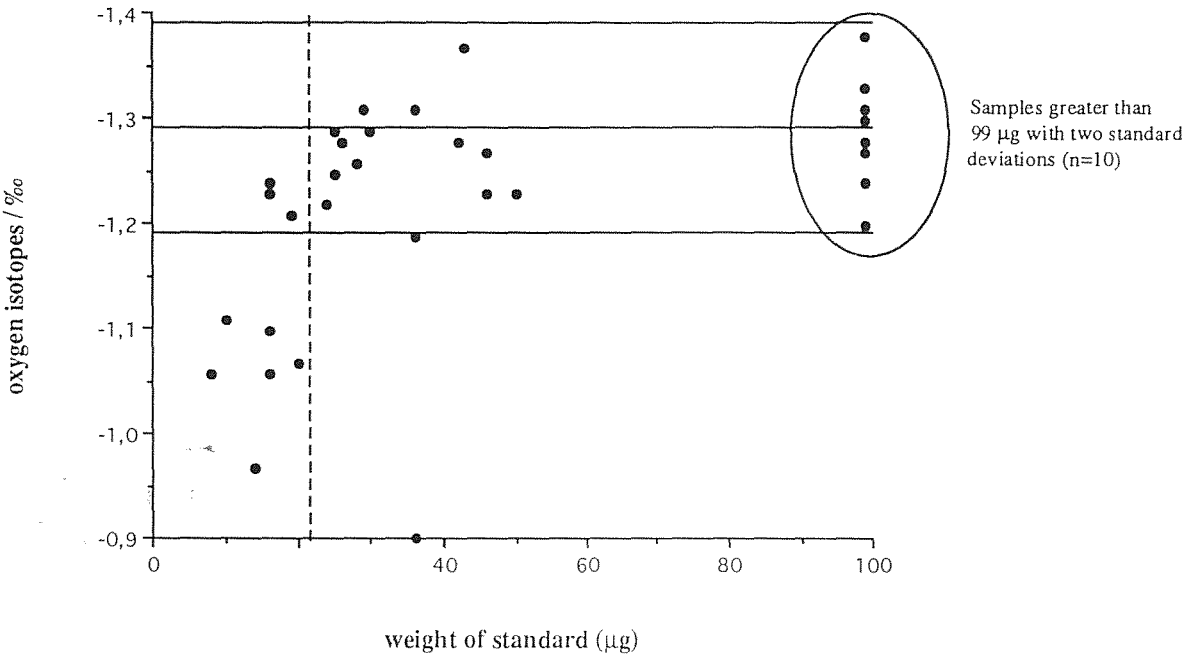
A) Oxygen isotopes



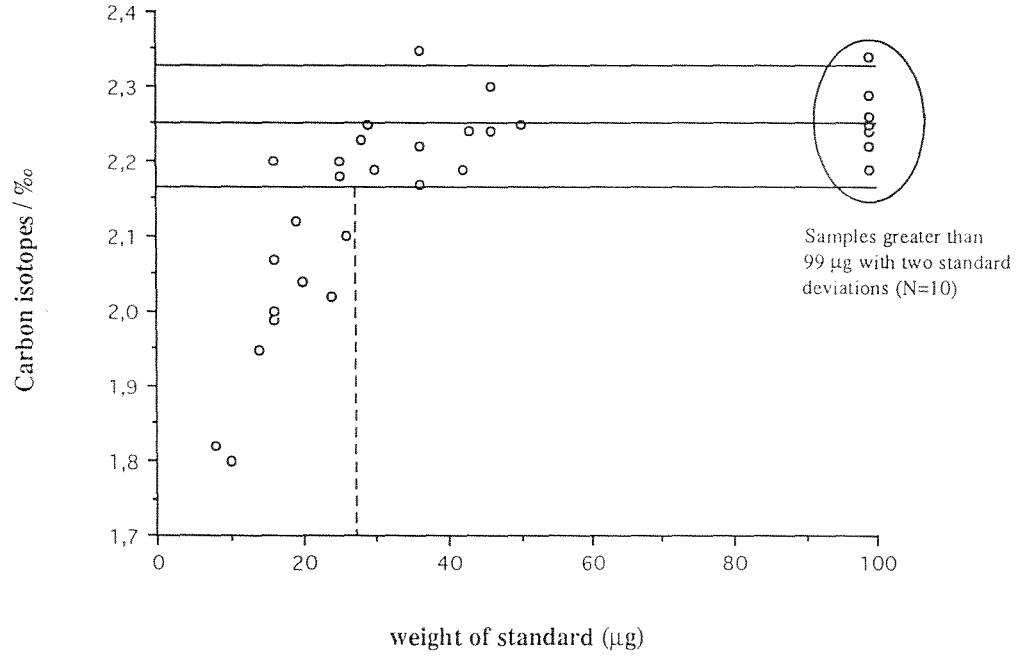
B) Carbon isotopes



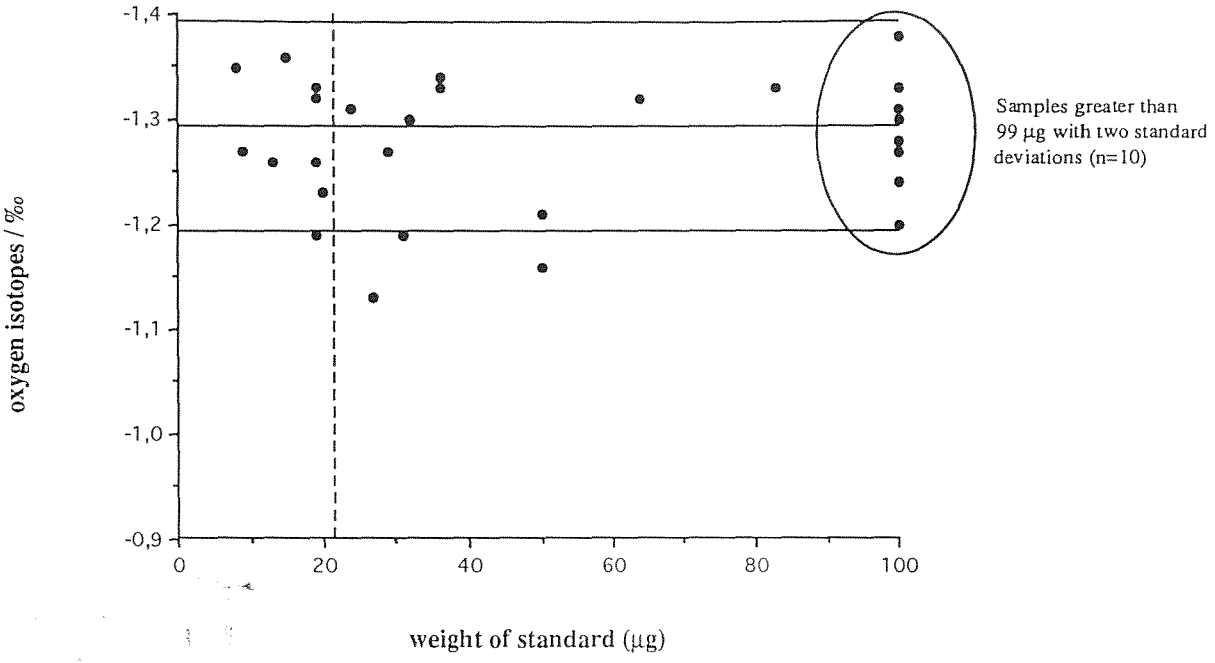
A / Oxygen isotopes



B/ Carbon isotopes



A / Oxygen isotopes



B/ Carbon isotopes