# Amino acid racemization in Neogloboquadrina pachyderma and Cibicidoides wuellerstorfi from the Arctic Ocean and its implications for age models

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#### 15 Abstract

We report the results of amino acid racemization (AAR) analyses of aspartic and glutamic acids in the planktic foraminifera, Neogloboquadrina pachyderma, and the benthic species, Cibicidoides wuellerstorfi, collected from sediment cores from the Arctic Ocean. The cores were retrieved at various deep-sea sites of the Arctic, which cover a large geographical area; from the Greenland and Iceland seas to the Alpha and Lomonosov Ridges in the central Arctic Ocean. Age models for the investigated sediments were developed by multiple

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dating and correlation techniques, including oxygen isotope stratigraphy, magnetostratigraphy, bio-, litho-, and cyclostratigraphy. The extent of racemization (D/L values) was determined on 95 samples (1028 subsamples) and shows a progressive increase downcore for both foraminifera species. Differences in the rates of racemization between the species were established by analysing specimens of both species from the same stratigraphic levels (n = 21). Aspartic acid and glutamic acid racemize on average 16±2 % and 23±3 % faster, respectively, in C. wuellerstorfi than in N. pachyderma. D/L values typically-increase with sample age in nearly all cases, with a trend that follows a simple power function. Scatter around least square regression fits are larger for samples from the central Arctic Ocean than for those from the Nordic Seas. Calibrating the rate of racemization in C. wuellerstorfi using independently dated samples from the Greenland and Iceland seas for the 30 past 400 ka enables estimation of sample ages from the central Arctic Ocean, where bottom water temperatures are similarmight have been are presently relatively similar. The resulting ages are older than expected when considering the existing age models for the central Arctic Ocean cores. These results confirm that the differences are not due to taxonomic effects on AAR and further warrant a critical evaluation of existing Arctic

Ocean age models. A better understanding of temperature histories at the investigated sites, and the-other environmental factors that may influence racemisation racemization rates in central Arctic Ocean sediments is

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also needed.

#### 1. Introduction

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The first application of amino acid geochronology to Arctic Ocean sediments analysed the extent of racemization epimerization in the protein amino acid, isoleucine, over time in samples of the planktic foraminifera, Neogloboquadrina pachyderma and the benthic species, Cibicidoides wuellerstorfi (Sejrup et al., 1984). Not only did this study provide some of the first amino acid racemization (AAR) data from a polar environment, but it also exposed crucial chronological issues associated with Arctic Ocean sediments. The results contradicted available age interpretations obtained from palaeomagnetic data (Sejrup et al., 1984; Backman, 2004). The problems of dating Pleistocene Arctic marine sediments continue to exist today and are well known (e.g. Alexanderson et al., 2014). Over the past few decades, amino acid geochronology received limited attention in the Arctic, but several studies provided promising results (Sejrup & Haugen, 1992; Kaufman et al., 2008, 2013) that highlighted its potential as a dating technique, and the need for its continued development in Arctic settings. This is particularly desirable, since theoretically, it could provide age control up to a few million years, using even limited amounts of biocarbonatecalcium carbonate.

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N. pachyderma and C. wuellerstorfi are commonly used in stable isotope stratigraphy and paleoceanographic reconstructions (e.g. Shackleton et al., 2003) and are associated with cold water masses. N. pachyderma is considered to be a primarily 'high-latitude' species (Darling et al., 2017), and C. wuellerstorfi is thought to show strong preference for bottom waters below ~5°C (Rasmussen and Thomsen, 2017). These characteristics and their frequent occurrence in sediment cores from the Arctic Ocean make them particularly useful for amino acid geochronology studies in this region.

The rates of racemization for aspartic and glutamic acids were previously calibrated in N. pachyderma from central Arctic Ocean samples by Kaufman et al. (2008) for the past 150 ka. The calibration relied on the 65 established age of upper Quaternarymodels of sediments from the Lomonosov Ridge (O'Regan et al., 2008). Subsequently, however, the extent of racemization in these samples was shown to be higher than expected when compared with those of similar ages from other cold bottom water sites from the Atlantic and Pacific oceans (Kaufman et al., 2013), despite the cold bottom water in the central Arctic Ocean. The reasons for this apparently higher extent of racemization in N. pachyderma from central Arctic Ocean samples is unclear, but 70 not considered to be caused by taxonomic effects, since the rate of racemization observed is lower in this species is lower than in other observed taxa (Kaufman et al., 2013). Either the established ages that were used to calibrate the rate of racemization in the central Arctic Ocean sediments are too young, such that units currently correlated with substages of marine oxygen isotope stage (MIS) 5 instead represent MIS 9, 7 and 5, or otherundetermined --processes influence protein degradation and preservation in the-central Arctic Ocean 75 foraminifera. At the Yermak Plateau in the eastern Arctic Ocean (Fig. 1), racemization rates for N. pachyderma generally conform to the rates determined for other cold bottom water sites (West et al., 2019), further challenging the established ages previously used to calibrate the rate of AAR from the Lomonosov Ridge in the central Arctic Ocean. However, it is unknown how racemization progresses in other regions of the Arctic Ocean.

- 80 If the apparently higher extent of racemization in *N. pachyderma* from the central Arctic Ocean is not the result of taxonomic effects, a higher rate of racemization can also be anticipated in other taxa from the area, e.g. in *Cibicidoides wuellerstorfi*. However, little is known about racemization of amino acids in this species. The earliest studies involving *C. wuellerstorfi* investigated taxonomical applications (Haugen et al., 1989), or focused on the epimerization of isoleucine (Sejrup et al., 1984; Sejrup and Haugen, 1992) utilising HPLC ion
- 85 exchange analysers. Since the publication of these seminal papers, analysis of amino acids has become significantly faster, with reduced sample size-mass requirements, due to improvements in analytical methods (Kaufman & Manley, 1998), yet no studies have addressed amino acid racemization in *C. wuellerstorfi* despite its palaeoceanographical importance (e.g. Yu & Elderfield, 2008; Wollenburg et al., 2015; Burkett et al., 2016; Raitzsch et al., 2020), and the relatively faster and easier sample processing offered by its larger tests (up to ~4-
- 90 5 times the size) when compared to *N. pachyderma*.

Here we report the results of aspartic acid and glutamic acid racemization analyses of *Neogloboquadrina pachyderma* and *Cibicidoides wuellerstorfi* obtained from well-dated Quaternary deep-sea sediment cores from the Greenland and Iceland seas, and from sediment cores from the central Arctic Ocean, where sediment ages continue to be debated (Purcell et al., 2022). The long-term rates of racemization in the two species are compared, and the relationship between the extent of racemization and sample age is investigated in both species.

#### 2. Materials and methods

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#### a) <u>Investigated sediment cores</u>

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Foraminifera samples were taken from sediment cores from the Greenland and Iceland seas, the Lomonosov Ridge, and the Alpha Ridge (Fig. 1). The Greenland and Iceland seas, although part of the Arctic Ocean, are under the direct influence of the Atlantic surface waters, and thus are predominantly characterised by open water conditions, unlike the sea ice covered areas of the Lomonosov and Alpha Ridges. The studied sediment cores (Table 1) were collected from deep water (811 – 2952 m) environments, which presently—based on modern estimates— experience similar, very cold (<0 °C), and relatively stable bottom water temperatures.

Region	Core	Latitude (°)	Longitude (°)	Water depth (m)	Bottom water temperature est. (°C)	Age reference
Alpha Ridge	AO16-9-PC1	85.95570	-148.32580	2212	-0.4	Cronin et al. (2019)
Lomonosov Ridge	AO16-5-PC1	89.07800	-130.54700	1253	-0.3	ACEX age model*
Lomonosov Ridge	LOMROG07-PC04	86.70117	-53.76720	811	0.1	Hanslik et al. (2013)
Lomonosov Ridge	LOMROG12-PC03	87.72470	-54.42528	1607	-0.4	O'Regan et al. (2020)
Lomonosov Ridge	LOMROG12-PC07	88.19760	-55.68450	2952	-0.8	ACEX age model*
Lomonosov Ridge	LOMROG12-PC09	89.02672	-73.73444	1318	-0.3	ACEX age model*
Iceland Sea	ODP 151/ 907A	69.24982	12.69823	1801	-0.8	Jansen et al. (2000a, b)
Greenland Sea	PS17 / 1906-2	76.84630	-2.15050	2901	-1.0	Bauch (2002, 2013)

Table 1: Sediment cores investigated in this study. Current bottom water temperatures were approximated by using annual mean temperature observations from the nearest location from the World Ocean Atlas (Locarnini et al., 2018). \*The ACEX age model is based on Backman et al. (2008), Frank et al. (2008), and O'Regan et al. (2008).



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Figure 1: Location of sediment cores referred to in this study. Basemap: Jakobsson et al. (2020).

Age-depth models for the investigated sediment cores <u>were-have been</u> developed using a variety of dating techniques. Cores from the Nordic Seas (ODP151/907A and PS17/1906-2) primarily relied on oxygen isotope stratigraphy, complemented by magnetostratigraphy in the case of ODP151/907A (Jansen et al., 2000a, b), and by carbon isotope stratigraphy for core PS17/1906-2 (Bauch 2002, 2013). The age-depth model of the latter is

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by carbon isotope stratigraphy for core PS<u>17/</u>1906-2 (Bauch, 2002, 2013). The age-depth model of the latter is less certain beyond marine isotope stage (MIS) 6 (Bauch, 2013), due to the large uncertainty associated with isotope stratigraphy, a characteristic issue of Arctic Ocean records.

The age-depth models of sediment cores from the central Arctic Ocean utilise a more diverse toolset, reflecting the difficulties of dating Arctic marine sediments, and heavily depend on a combination of bio- and lithostratigraphy (e.g. Cronin et al., 2019). The lithostratigraphy of the central Arctic Ocean cores investigated in this study can be correlated to-with that of the Integrated Ocean Drilling Program Expedition 302, the Arctic Coring Expedition (ACEX). This correlation is most apparent when in bulk density profiles are used (Fig. 2), but it has been shown to be also coherent when other sedimentological properties including grain size and a variety of XRF-scanning properties (O'Regan et al., 2019) arealso correlate coherently among cores-utilised (O'Regan et al., 2019). The currently accepted age model for the ACEX sedimentary sequence was developed using cyclostratigraphic analysis (O'Regan et al., 2008) and produced similar estimated Quaternary sedimentation rates as obtained by the decay of beryllium isotopes (Frank et al., 2008). The late Quaternary chronology (MIS 1 – 6) for ACEX included constraints from <sup>14</sup>C dating, the correlation to-with near-by records AO96/12-1PC (Jakobsson et al., 2001) and PS2185 (Spielhagen et al., 2004), where MIS 5 was identified based on the occurrence of the calcareous nannofossil *Emiliania huxleyi* (Jakobsson et al., 2001), and further supported by results from optically stimulated luminescence dating of quartz grains (Jakobsson et al., 2003). The age model of core LOMROG07-PC04 is based on correlation to-with PS2185 (Hanslik et al., 2013).

LOMROG12-7PC AO16-5PC LRG12-9PC LOMROG12-3PC/TWC AO16-9PC ACEX Zr/Ti Zr/Ti Zr/Ti Zr/Ti 0.2 0.3 0.4 0.5 0.7 0.3 0.4 0.5 0.3 0.4 0.5 0.6 0.2 0.3 0.4 0.5 2 3 4 Munner . Depth (rmcd) 1.5 1.7 1.9 1.5 17 1 9 BD (g/cm<sup>3</sup>) BD (g/cm<sup>3</sup>) 6 5 8 1.6 1.8 2.0 BD (g/cm3) 9 12 1.4 1.6 1.8 BD (g/cm<sup>3</sup>) 1.4 1.6 1.8 2.0 BD (g/cm<sup>3</sup>) 10 11 12 1.3 1.5 1.7 1.9 BD (g/cm<sup>3</sup>)

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Figure 2: a)-Bulk density (BDp) (black line) and XRF-scanning Zr/Ti (red line) profiles of sediment cores from the central Arctic Ocean, with correlations among cores and with illustrating the correlative sequences in each of the records compared to the ACEX composite section. . b) Correlation of bulk density ( $\rho$ ) profiles of sediment cores from the central Arctic Ocean. D<sub>A</sub> and D<sub>B</sub> are prominent diamict units found in all the cores, while PL is a characteristic fine-grained peach coloured layer. Locations Depth profiles were all scaled linearly to match the corresponding depths in the Arctic Coring Expedition (ACEX) core.Samples of N. pachyderma (circles) and C. wuellerstorfi (triangles) samples analysed in this study are shown for each core.

b) Analytical procedures

Sediment samples were wet sieved (63 µm), and air dried prior to picking foraminifera tests of *N. pachyderma* and *C. wuellerstorfi*. Initially, the  $\geq$ over-250-µm fraction was targeted to isolate the largest and best-preserved tests. For some samples this was not possible, and the tests were collected from the 180—250 µm fraction instead. The tests were kept in glass vials, and stored in a refrigerator prior to racemization analyses. A total of 95 stratigraphic depths were sampled, with some depths containing both foraminifera species (*n* = 21). Each sample was further subsampled – on average with 9.6 *N. pachyderma* and 11.2 *C. wuellerstorfi* subsamples per

- sample was further subsampled on average with 9.6 *N. pachyderma* and 11.2 *C. wuellerstorfi* subsamples per sample, producing  $\frac{1028-1009}{2}$  analysed subsamples. Each subsample comprised between 10 and 12 *N. pachyderma*, or 2 and 4 *C. wuellerstorfi* tests.
- 160 The analytical procedures followed that of previous analyses as described in detail by Kaufman et al. (2013) and West et al. (2019), and were performed at the Amino Acid Geochronology Laboratory (AAGL), Northern Arizona University. The foraminifera tests were first sonicated (1-30 s) to remove any loose sediment particles, treated with 1 ml-mL hydrogen peroxide (3%) to remove surficial organic matter, and then rinsed three times with reagent grade (grade I) water. Multiple tTests were picked into micro-reaction hydrolysis vials (defining one subsample), and dissolved in 8 µl-µL hydrochloric acid (6 M) was added to dissolve the tests. The vials 165 were then sealed with nitrogen gas, and the subsamples hydrolysed for 6 hours at 110°C. After the hydrolysis was complete, the subsamples were evaporated in a vacuum desiccator, and then rehydrated in 4  $\mu$ L of 0.01 M HCl spiked with 10 µM L-homoarginine. Each subsample was injected onto a high-performance liquid chromatograph (HPLC) with a fully automated, reversed phase procedure (Kaufman & Manley, 1998) to 170 separate pairs of D- and L-amino acids. The peak-area ratio of D and L stereoisomers of eight amino acids (aspartic acid, glutamic acid, serine, alanine, valine, phenylalanine, isoleucine and leucine) were analysed to determine the extent of racemization. T-but this study only utilised the racemization results of aspartic acid (Asp) and glutamic (Glu) acid-racemization, which are among the two most abundant and chronographically well-resolved amino acids. Asp and Glu reported in this study also include any Asn and Gln present in the 175 biomineral.

## 3. Results

#### a) Data screening

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Initial data screening was based on the procedure of Kosnik & Kaufman (2008). First, subsamples with L-Ser / L-Asp  $\ge 0.8$  – an indicator of potential contamination by modern amino acids – were excluded. The D/L values of Asp and Glu positively covary in fossil proteins. Subsamples <u>that</u>, which did not adhere to this expected trend were omitted. Finally, subsamples with D/L Asp or Glu values <u>beyondnot within ±2</u> $\sigma$  of the sample mean were also removed (Supplementary Fig. S1). As a result of this screening process, <u>1617.9</u>:0 % of all subsamples were rejected (Supplementary Table S1).

Following the subsample screening process, sample means, and related standard deviation values were calculated for Asp and Glu for all samples. Stratigraphically reversed samples (mean D/L values lower than expected for their stratigraphic depths with no overlap within  $1\sigma$  with the sample from shallower depth) were

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identified within each core (Table 2). <u>These include five samples of *N. pachyderma* and three *C. wuellerstorfi*. Of these, two samples contained sufficient tests of both species to analyse AAR, but only *C. wuellerstorfi* were stratigraphically reversed.</u>

17337       AO16-9-PC1       0.210       22       10       1       10       0.210       0.010       0.140       0.000       0.140       0.010       0.140       0.020       0.242       0.020         17338       AO16-9-PC1       1.260       1.88       10       5       55       0.0445       0.015       0.224       0.020         Lommonsov Ridge       -       -       -       -       -       -       -       -         21406       LOMROG12-PC03       0.495       60       7       1       14       0.133       0.017       0.168       0.022       0.163       0.022       0.163       0.022       0.163       0.021       0.031       0.016       0.017       0.168       0.021       0.032       0.163       0.021       0.032       0.163       0.021       0.031       0.016       0.040       0.212       0.021       0.03	Lab ID (UAL)	Core	Core depth (m)	Age (ka)	nª	Excluded	Excl. ratio (%)	Asp D/L	1σ	Glu D/L	1σ
Apha Ridge         Apple Note         Apple N	Neoalob	oquadrina pachvderm	a								
17337       AO16-9-PC1       0.190       16       10       1       10       0.141       0.014       0.069       0.011         17337       AO16-9-PC1       0.210       0.22       10       1       10       0.210       0.010       0.164       0.000         17338       AO16-9-PC1       1.200       188       10       5       50       0.445       0.021       0.024       0.022       0.024       0.022       0.024       0.024       0.024       0.024       0.024       0.024       0.027       0.011         21406       LOMROG12-PC03       0.095       38       7       1       14       0.133       0.017       0.016       0.02       0.168       0.021       0.020       0.017       0.168       0.022       0.168       0.021       0.020       0.017       0.168       0.021       0.020       0.017       0.168       0.021       0.020       0.021       0.020       0.021       0.020       0.021       0.020       0.021       0.020       0.021       0.020       0.021       0.024       0.017       0.040       0.024       0.017       0.040       0.024       0.017       0.042       0.024       0.017       0.024       0.024	J		-								
17337       AO16-9-PC1       0.210       22       10       1       10       0.210       0.010       0.140       0.000       0.140       0.010       0.140       0.020       0.242       0.020         17338       AO16-9-PC1       1.260       1.88       10       5       55       0.0445       0.015       0.224       0.020         Lommonsov Ridge       -       -       -       -       -       -       -       -         21406       LOMROG12-PC03       0.495       60       7       1       14       0.133       0.017       0.168       0.022       0.163       0.022       0.163       0.022       0.163       0.021       0.031       0.016       0.017       0.168       0.021       0.032       0.163       0.021       0.032       0.163       0.021       0.031       0.016       0.040       0.212       0.021       0.03	17336	• •	0.190	16	10	1	10	0.141	0.014	0.069	0.011
17338         AO16-9-PC1         1.260         1.27         8         3         38         0.419         0.020         0.242         0.020           17339         AO16-9-PC1         1.260         188         10         5         50         0.445         0.020         0.242         0.020           Lommosov Ridge	17337	AO16-9-PC1	0.210	-	10	1	10	0.210	0.010	0.104	0.005
17339         AO16-9-PC1         1.260         1188         10         5         50         0.445         0.015         0.254         0.02           Lumonosov Ridge	17338	AO16-9-PC1	0.970		8	3	38	0.419	0.020	0.242	0.029
Lomonosov Ridge         N         N         N         N         N           21406         LOMROG12-TWC03         0.095         38         7         1         144         0.193         0.017         0.017         0.017           21407         LOMROG12-TWC03         0.695         61         8         2         25         0.340         0.032         0.161         0.026         0.017         0.188         0.026         0.017         0.016         0.021         0.206         0.011         0.036         0.021         0.206         0.011         0.046         0.021         0.206         0.011         0.040         0.212         0.023         0.212         0.021         0.230         0.021         0.230         0.040         0.212         0.230         0.041         0.230         0.021         0.230         0.021         0.230         0.021         0.230         0.021         0.230         0.021         0.230         0.031         0.032         0.181         0.041         0.032         0.181         0.041         0.032         0.181         0.041         0.032         0.031         0.030         0.030         0.030         0.030         0.030         0.030         0.030         0.030         0.0		AO16-9-PC1			10	5	50	0.445		0.254	0.022
21406         LOMROG12-TWC03         0.095         38         7         1         14         0.193         0.017         0.017           21407         LOMROG12-TWC03         0.495         60         7         1         144         0.323         0.017         0.168         0.022           21408         LOMROG12-PC03         0.610         76         8         1         13         0.395         0.021         0.206         0.161           21410         LOMROG12-PC03         0.610         76         8         1         13         0.395         0.021         0.206         0.240         0.011           21411         LOMROG12-PC03         1.291         204         8         2         25         0.468         0.015         0.240         0.017           21413         LOMROG12-PC03         1.551         290         6         2         33         0.464         0.025         0.155         0.240         0.077         0.040           21414         LOMROG12-PC03         1.781         391         5         0         0         0.377         0.060         0.337         0.060         0.336         0.037         0.060         0.336         0.0377         0.040		Lomonosov Ridge									
21408         LOMROG12-PC03         0.505         61         8         2         25         0.340         0.032         0.163         0.02           21409         LOMROG12-PC03         0.610         76         8         1         13         0.395         0.27         0.206         0.011           21410         LOMROG12-PC03         1.291         204         8         2         25         0.468         0.015         0.240         0.01           21411         LOMROG12-PC03         1.511         290         6         2         33         0.446         0.066         0.242         0.07           21414         LOMROG12-PC03         1.511         280         6         2         33         0.446         0.066         0.242         0.07           21414         LOMROG12-PC03         1.781         391         5         0         0         0.377         0.660         0.420         0.014         0.011         0.014         0.011         0.014         0.011         0.014         0.011         0.014         0.011         0.014         0.011         0.014         0.011         0.014         0.011         0.014         0.011         0.014         0.012         0.060	21406	-	0.095	38	7	1	14	0.193	0.017	0.077	0.010
21408         LOMROG12-PC03         0.505         61         8         1         13         0.335         0.021         0.026         0.011           21409         LOMROG12-PC03         0.610         76         8         1         13         0.335         0.021         0.206         0.011           21410         LOMROG12-PC03         1.291         204         8         2         25         0.468         0.015         0.240         0.011           21411         LOMROG12-PC03         1.511         290         6         2         33         0.446         0.066         0.242         0.07           21413         LOMROG12-PC03         1.511         290         6         2         33         0.446         0.066         0.242         0.07           21414         LOMROG12-PC03         1.781         391         5         0         0         0.377         0.600         1.010         0.044         0.011         0.140         0.012         0.018         0.0037         0.007         0.044         0.012         0.018         0.014         0.014         0.014         0.014         0.014         0.014         0.025         MIS 1.37 (-19)         13         2         15 <td>21407</td> <td>LOMROG12-TWC03</td> <td>0.495</td> <td>60</td> <td>7</td> <td>1</td> <td>14</td> <td>0.332</td> <td>0.017</td> <td>0.168</td> <td>0.022</td>	21407	LOMROG12-TWC03	0.495	60	7	1	14	0.332	0.017	0.168	0.022
21409         LOMROG12-PC03         0.610         76         8         1         13         0.395         0.021         0.206         0.011           21410         LOMROG12-PC03         1.291         204         8         2         25         0.488         0.015         0.240         0.01           21411         LOMROG12-PC03         1.551         290         6         2         33         0.443         0.021         0.82         0.72           21413         LOMROG12-PC03         1.601         311         8         3         0.417         0.060         0.91         0.042         0.07           21414         LOMROG12-PC03         1.601         311         8         3         8         0.417         0.060         0.91         0.04           21415         LOMROG7-PC04         0.025         MIS 1.3? (-19)         17         4         24         0.102         0.18         0.040         0.041         0.021         0.040         0.041         0.215         0.006         0.033         0.007         0.006         0.036         0.020         0.140         0.025         MIS 1.7 (-77)         8         3         38         0.276         0.008         0.123         0.	21408	LOMROG12-PC03	0.505		8	2	25	0.340	0.032	0.163	0.026
21410         LOMROG12-PC03         0.761         108         8         3         38         0.415         0.040         0.212         0.02           21411         LOMROG12-PC03         1.291         204         8         2         25         0.48         0.015         0.240         0.01           21412         LOMROG12-PC03         1.601         2316         6         2         33         0.443         0.026         0.027         0.021           21414         LOMROG12-PC03         1.601         3111         8         3         38         0.417         0.032         0.185         0.027           21414         LOMROG12-PC03         1.781         391         5         0         0         0.377         0.006         0.191         0.044         0.141           21415         LOMROG07-PC04         0.040         MIS 1.37 (~39)         8         3         38         0.077         0.006         0.036         0.000           21587         LOMROG07-PC04         0.025         MIS 5.17 (~42)         1         1         9         0.278         0.008         0.133         0.000           21581         LOMROG07-PC04         1.025         MIS 5.57 (~123)         8<	21409	LOMROG12-PC03			8	1		0.395	0.021	0.206	0.015
21411         LOMROG12-PC03         1.291         204         8         2         25         0.468         0.015         0.240         0.011           21412         LOMROG12-PC03         1.551         290         6         2         33         0.466         0.066         0.242         0.07           21414         LOMROG12-PC03         1.551         290         6         2         33         0.466         0.066         0.242         0.07           21414         LOMROG12-PC03         1.781         391         5         0         0         0.377         0.060         0.191         0.04           21416         LOMROG07-PC04         0.025         MIS 1.3? (~19)         17         4         24         0.102         0.018         0.044         0.01           21769         LOMROG07-PC04         0.205         MIS 1.3? (~19)         8         3         38         0.977         0.060         0.323         0.001         1.014         0.02           22760         LOMROG07-PC04         1.205         MIS 5.57 (~12)         13         2         15         0.320         0.011         0.15         0.01           22761         LOMROG07-PC04         1.025         MIS	21410	LOMROG12-PC03			8	3	38	0.415	0.040	0.212	0.025
21412         LOMROG12-PC03         1.401         236         6         2         33         0.443         0.021         0.23           21413         LOMROG12-PC03         1.551         290         6         2         33         0.443         0.021         0.22         0.02           21414         LOMROG12-PC03         1.611         311         8         3         38         0.417         0.032         0.185         0.022           21414         LOMROG12-PC03         1.781         391         5         0         0         0.377         0.060         0.191         0.04           21414         LOMROG07-PC04         0.025         MIS 1.3? (~19)         17         4         24         0.102         0.18         0.044         0.01           21587         LOMROG07-PC04         0.025         MIS 5.1? (~77)         8         3         38         0.278         0.034         0.114         0.02           21581         LOMROG07-PC04         1.100         MIS 5.5? (~12)         13         2         15         0.387         0.029         0.146         0.02           21581         LOMROG07-PC04         1.325         MIS 5.7 (~40)         13         2         15 </td <td></td> <td>LOMROG12-PC03</td> <td></td> <td></td> <td>8</td> <td></td> <td>25</td> <td>0.468</td> <td>0.015</td> <td></td> <td>0.010</td>		LOMROG12-PC03			8		25	0.468	0.015		0.010
21413         LOMROG12-PC03         1.551         290         6         2         33         0.466         0.066         0.242         0.07           21414         LOMROG12-PC03         1.761         391         5         0         0.417         0.032         0.185         0.02           21416         LOMROG12-PC03         2.711         449         8         5         63         0.392         0.037         0.207         0.04           21787         LOMROG07-PC04         0.025         MIS 1-3? (~19)         17         4         24         0.102         0.018         0.040         0.011           21587         LOMROGO7-PC04         0.040         MIS 5.1? (~77)         8         3         38         0.278         0.008         0.123         0.002           21589         LOMROGO7-PC04         1.025         MIS 5.7 (~12)         11         1         9         0.276         0.038         0.113         0.021         0.140         0.022           21589         LOMROGO7-PC04         1.055         MIS 5.7 (~12)         13         2         15         0.387         0.029         0.140         0.022         0.140         0.025         0.33         2155         0.036	21412	LOMROG12-PC03	1.401		6	2	33	0.443	0.021	0.239	0.026
21414         LOMROG12-PC03         1.601         311         8         3         38         0.417         0.032         0.185         0.022           21415         LOMROG12-PC03         1.781         391         5         0         0         0.377         0.060         0.191         0.04           21416         LOMROG7-PC04         0.025         MIS 1-3? (~19)         17         4         24         0.102         0.018         0.044         0.011           21587         LOMROG7-PC04         0.025         MIS 1-3? (~19)         17         4         24         0.102         0.016         0.034         0.014         0.012           21687         LOMROG7-PC04         0.025         MIS 5.1? (~77)         8         3         38         0.077         0.008         0.031         0.114         0.022         0.011         0.151         0.011           21760         LOMROG7-PC04         1.025         MIS 5.5? (~115)         13         2         15         0.320         0.011         0.151         0.011           21769         LOMROG7-PC04         1.100         MIS 5.5? (~123)         8         3         38         0.331         0.036         0.175         0.044 <tr< td=""><td></td><td></td><td></td><td></td><td>6</td><td></td><td></td><td>0.466</td><td></td><td></td><td>0.070</td></tr<>					6			0.466			0.070
21415         LOMROG12-PC03         1.781         391         5         0         0         0.377         0.060         0.191         0.044           21416         LOMROG12-PC03         2.101         489         8         5         63         0.322         0.037         0.207         0.044           22759         LOMROG7-PC04         0.024         MIS 1-3? (~19)         17         4         24         0.102         0.018         0.044         0.01           21587         LOMROG7-PC04         0.140         MIS 5.1? (~77)         8         3         38         0.027         0.034         0.114         0.02           21589         LOMROG07-PC04         1.025         MIS 5.7 (~15)         13         2         15         0.327         0.030         0.011         0.116         0.012           21589         LOMROG7-PC04         1.310         MIS 5.7 (~12.8)         8         3         38         0.337         0.036         0.175         0.031           21591         LOMROG7-PC04         1.780         MIS 5.1? (~74)         12         4         33         0.38         0.036         0.175         0.04           22761         LOMROG7-PC04         1.785         MIS 5.	21414	LOMROG12-PC03			8	3	38	0.417	0.032	0.185	0.026
21416         LOMROG12-PC03         2.101         489         8         5         63         0.392         0.037         0.207         0.044           22759         LOMROG07-PC04         0.025         MIS 1-3? (~19)         17         4         224         0.102         0.018         0.044         0.01           21587         LOMROG07-PC04         0.104         MIS 5.1? (~77)         8         3         38         0.077         0.006         0.036         0.00           22760         LOMROG07-PC04         0.225         MIS 5.1? (~72)         8         3         8         0.278         0.008         0.113         0.002           22761         LOMROG07-PC04         1.025         MIS 5.7 (~115)         13         2         15         0.327         0.029         0.146         0.02           22762         LOMROG07-PC04         1.385         > MIS 1-3? (~40)         13         2         15         0.347         0.029         0.146         0.02           22763         LOMROG07-PC04         1.720         MIS 1-3? (~12.)         8         3         38         0.391         0.035         0.175         0.041           21592         LOMROG07-PC04         1.705         MIS 5.1?	21415	LOMROG12-PC03			5	0	0	0.377	0.060		0.048
22759         LOMROGO7-PC04         0.025         MIS 1-3? (~19)         17         4         24         0.102         0.018         0.044         0.01           21587         LOMROGO7-PC04         0.040         MIS 1-3? (~39)         8         3         38         0.097         0.006         0.036         0.000           21588         LOMROGO7-PC04         0.226         MIS 5.17 (~77)         8         3         38         0.278         0.008         0.123         0.00           22760         LOMROGO7-PC04         1.225         MIS 5.57 (~15)         13         2         15         0.327         0.009         0.146         0.02           22761         LOMROGO7-PC04         1.10         MIS 5.57 (~15)         13         2         15         0.387         0.029         0.146         0.02           22762         LOMROGO7-PC04         1.410         MIS 1-37 (~12)         8         3         38         0.387         0.029         0.146         0.02           22763         LOMROGO7-PC04         1.720         MIS 1-37 (~40)         8         3         38         0.387         0.040         0.175         0.04           22764         LOMROGO7-PC04         2.025         MIS		LOMROG12-PC03	2.101		8	5	63				0.040
21587         LOMROG07-PC04         0.040         MIS 1-3? (~39)         8         3         38         0.097         0.006         0.036         0.000           21588         LOMROG07-PC04         0.140         MIS 5.1? (~77)         8         3         38         0.278         0.034         0.114         0.00           22760         LOMROG07-PC04         0.225         MIS 5.1? (~72)         8         3         38         0.278         0.008         0.112         0.00           22761         LOMROG07-PC04         1.025         MIS 5.5? (~115)         13         2         15         0.387         0.029         0.146         0.02           22762         LOMROG07-PC04         1.385         > MIS 7? (~405)         13         2         15         0.387         0.029         0.146         0.02           21591         LOMROG07-PC04         1.702         MIS 5.1? (~74)         12         4         33         0.386         0.036         0.175         0.04           21591         LOMROG07-PC04         1.785         MIS 5.1? (~74)         12         4         33         0.386         0.023         0.06           21594         LOMROG07-PC04         2.010         MIS 5.5? (~115)		LOMROG07-PC04			17	4	24	0.102	0.018	0.044	0.011
21588         LOMROG07-PC04         0.140         MIS 5.1? (~77)         8         3         38         0.278         0.034         0.114         0.02           22760         LOMROG07-PC04         0.225         MIS 5.1? (~82)         11         1         9         0.278         0.008         0.123         0.001         0.151         0.011         0.151         0.012           21589         LOMROG07-PC04         1.110         MIS 5.5? (~115)         13         2         15         0.387         0.029         0.146         0.02           22762         LOMROG07-PC04         1.140         MIS 1-3? (~40)         8         3         38         0.318         0.035         0.177         0.03           21591         LOMROG07-PC04         1.720         MIS 1-3? (~40)         8         3         38         0.318         0.035         0.175         0.04           21592         LOMROG07-PC04         1.785         MIS 5.1? (~74)         12         4         33         0.366         0.023         0.187         0.01           21592         LOMROG07-PC04         2.025         MIS 5.5? (~113)         9         2         22         0.417         0.069         0.230         0.06	21587	LOMROG07-PC04		. ,		3		0.097	0.006	0.036	0.004
22760         LOMROG07-PC04         0.225         MIS 5.1? (-82)         11         1         9         0.278         0.008         0.123         0.002           22761         LOMROG07-PC04         1.025         MIS 5.5? (-115)         13         2         15         0.320         0.011         0.151         0.011           21589         LOMROG07-PC04         1.110         MIS 5.5? (-123)         8         4         50         0.347         0.029         0.146         0.02           22762         LOMROG07-PC04         1.385         > MIS 7? (-405)         13         2         15         0.387         0.029         0.146         0.02           21591         LOMROG07-PC04         1.410         MIS 1-3? (-40)         8         3         38         0.391         0.036         0.175         0.04           22763         LOMROG07-PC04         1.720         MIS 5.1? (-74)         12         4         33         0.386         0.023         0.187         0.014           22764         LOMROG07-PC04         2.025         MIS 5.5? (-115)         9         2         22         0.417         0.069         0.330         0.066           21586         LOMROG12-PC07         0.020	21588	LOMROG07-PC04	0.140	. ,	8	3	38	0.278	0.034	0.114	0.021
22761         LOMROG07-PC04         1.025         MIS 5.5? (~115)         13         2         15         0.320         0.011         0.151         0.011           21589         LOMROG07-PC04         1.110         MIS 5.5? (~123)         8         4         50         0.347         0.029         0.146         0.02           22762         LOMROG07-PC04         1.385         > MIS 7? (~405)         13         2         15         0.387         0.029         0.146         0.03           "21590         LOMROG07-PC04         1.410         MIS 1.3? (~400)         8         3         38         0.318         0.035         0.137         0.03           21591         LOMROG07-PC04         1.720         MIS 5.1? (~74)         12         4         33         0.386         0.023         0.187         0.014           22763         LOMROG07-PC04         2.010         MIS 5.1? (~78)         8         7         88         0.357         0.140         0.230         0.06           22763         LOMROG07-PC04         3.000         MIS 5.5? (~123)         7         4         57         0.270         0.099         0.136         0.05           15866         LOMROG12-PC07         0.165	22760	LOMROG07-PC04	0.225	. ,	11	1	9	0.278	0.008	0.123	0.009
21589         LOMROG07-PC04         1.110         MIS 5.5? (~123)         8         4         50         0.347         0.029         0.146         0.022           22762         LOMROG07-PC04         1.385         > MIS 7? (~405)         13         2         15         0.387         0.029         0.195         0.033           *21590         LOMROG07-PC04         1.410         MIS 1-3? (~40)         8         3         38         0.318         0.035         0.137         0.03           21591         LOMROG07-PC04         1.720         MIS 1-3? (~40)         8         3         38         0.386         0.023         0.175         0.04           21592         LOMROG07-PC04         1.785         MIS 5.1? (~715)         9         2         22         0.417         0.069         0.230         0.06           *21592         LOMROG07-PC04         2.025         MIS 5.5? (~123)         7         4         57         0.270         0.099         0.136         0.05           15866         LOMROG12-PC07         0.165         31         10         1         10         0.025         0.036         0.090         0.02           15868         LOMROG12-PC07         0.185         52	22761	LOMROG07-PC04	1.025	. ,	13	2	15	0.320	0.011	0.151	0.012
22762         LOMROG07-PC04         1.385         > MIS 7? (~405)         13         2         15         0.387         0.029         0.195         0.033 <sup>b</sup> 21590         LOMROG07-PC04         1.410         MIS 1-3? (~12.8)         8         3         38         0.318         0.035         0.137         0.03           21591         LOMROG07-PC04         1.720         MIS 1-3? (~40)         8         3         38         0.318         0.036         0.175         0.04           22763         LOMROG07-PC04         1.785         MIS 5.1? (~715)         9         2         22         0.417         0.069         0.230         0.060           22764         LOMROG07-PC04         2.025         MIS 5.5? (~123)         7         4         57         0.270         0.099         0.136         0.05           15866         LOMROG12-PC07         0.165         31         10         1         10         0.025         0.036         0.090         0.02           15868         LOMROG12-PC07         0.165         31         10         1         10         0.225         0.026         0.122         0.011           15869         LOMROG12-PC07         0.315         40         <	21589	LOMROG07-PC04	1.110	, ,	8	4	50	0.347	0.029	0.146	0.022
*21590         LOMROG07-PC04         1.410         MIS 1-3? (~12.8)         8         3         38         0.318         0.035         0.137         0.03           21591         LOMROG07-PC04         1.720         MIS 1-3? (~40)         8         3         38         0.391         0.036         0.175         0.04           22763         LOMROG07-PC04         1.785         MIS 5.1? (~74)         12         4         33         0.386         0.023         0.187         0.01           21592         LOMROG07-PC04         2.010         MIS 5.1? (~78)         8         7         88         0.357         0.140           22764         LOMROG07-PC04         3.000         MIS 5.5? (~123)         7         4         57         0.270         0.099         0.36         0.001           15866         LOMROG12-PC07         0.020         9         9         2         22         0.081         0.004         0.034         0.00           15868         LOMROG12-PC07         0.165         31         10         1         10         0.226         0.22         0.272         0.026         0.122         0.01           15869         LOMROG12-PC07         0.315         40         10 <td>22762</td> <td>LOMROG07-PC04</td> <td>1.385</td> <td>, ,</td> <td>13</td> <td>2</td> <td>15</td> <td>0.387</td> <td>0.029</td> <td>0.195</td> <td>0.032</td>	22762	LOMROG07-PC04	1.385	, ,	13	2	15	0.387	0.029	0.195	0.032
21591         LOMROG07-PC04         1.720         MIS 1-3? (~40)         8         3         38         0.391         0.36         0.175         0.04           22763         LOMROG07-PC04         1.785         MIS 5.1? (~74)         12         4         33         0.386         0.023         0.187         0.010           21592         LOMROG07-PC04         2.010         MIS 5.1? (~78)         8         7         88         0.357         0.140           22764         LOMROG07-PC04         2.025         MIS 5.5? (~115)         9         2         22         0.417         0.069         0.230         0.06           "21594         LOMROG07-PC04         3.000         MIS 5.5? (~123)         7         4         57         0.270         0.099         0.136         0.05           15866         LOMROG12-PC07         0.020         9         9         2         22         0.81         0.004         0.034         0.00           15867         LOMROG12-PC07         0.315         40         10         2         20         0.272         0.025         0.124         0.01           15867         LOMROG12-PC07         0.780         52         9         1         11	<sup>b</sup> 21590	LOMROG07-PC04	1.410	. ,	8	3	38	0.318	0.035	0.137	0.035
22763         LOMROG07-PC04         1.785         MIS 5.1? (~74)         12         4         33         0.386         0.023         0.187         0.010           21592         LOMROG07-PC04         2.010         MIS 5.1? (~78)         8         7         88         0.357         0.140           22764         LOMROG07-PC04         2.025         MIS 5.5? (~115)         9         2         22         0.417         0.069         0.230         0.069           *21594         LOMROG07-PC04         3.000         MIS 5.5? (~123)         7         4         57         0.270         0.099         0.136         0.05           15866         LOMROG12-PC07         0.020         9         9         2         22         0.81         0.004         0.034         0.00           15867         LOMROG12-PC07         0.315         40         10         2         20         0.272         0.026         0.122         0.011           15870         LOMROG12-PC07         0.780         52         9         1         11         0.244         0.163         0.022           15871         LOMROG12-PC07         1.770         79         9         3         33         0.399         0.0	21591	LOMROG07-PC04		. ,	8	3	38	0.391	0.036	0.175	0.043
21592LOMROG07-PC042.010MIS 5.1? (~78)87880.3570.14022764LOMROG07-PC042.025MIS 5.5? (~115)92220.4170.0690.2300.06*21594LOMROG07-PC043.000MIS 5.5? (~123)74570.2700.0990.1360.0515866LOMROG12-PC070.020992220.0810.0040.0340.0015867LOMROG12-PC070.16531101100.2050.0360.9900.0215868LOMROG12-PC070.31540102200.2720.0250.1240.0115869LOMROG12-PC070.7805291110.2940.0260.1220.0115870LOMROG12-PC071.707993330.3730.0290.1890.0215871LOMROG12-PC071.4259693330.3850.0240.1680.0215873LOMROG12-PC071.58011192220.3680.0300.1680.0215874LOMROG12-PC071.5873749000.3850.0240.1730.0215875LOMROG12-PC073.53735093330.3650.0480.1850.0315875LOMROG12-PC073.5673749000.3540.0480.1850.03<	22763	LOMROG07-PC04	1.785		12	4	33	0.386	0.023	0.187	0.016
22764         LOMROG07-PC04         2.025         MIS 5.5? (~115)         9         2         22         0.417         0.069         0.230         0.069           *21594         LOMROG07-PC04         3.000         MIS 5.5? (~123)         7         4         57         0.270         0.099         0.136         0.05           15866         LOMROG12-PC07         0.020         9         9         2         22         0.081         0.004         0.034         0.00           15867         LOMROG12-PC07         0.165         31         10         1         10         0.205         0.036         0.090         0.02           15868         LOMROG12-PC07         0.315         40         10         2         20         0.272         0.026         0.122         0.01           15869         LOMROG12-PC07         0.780         52         9         1         11         0.294         0.026         0.122         0.01           15871         LOMROG12-PC07         1.170         79         9         3         33         0.373         0.029         0.189         0.02           15871         LOMROG12-PC07         1.580         111         9         2         22<	21592	LOMROG07-PC04	2.010	. ,	8	7	88	0.357		0.140	
**21594         LOMROG07-PC04         3.000         MIS 5.5? (~123)         7         4         57         0.270         0.099         0.136         0.051           15866         LOMROG12-PC07         0.020         9         9         2         22         0.081         0.004         0.034         0.001           15867         LOMROG12-PC07         0.165         31         10         1         10         0.205         0.036         0.090         0.022           15868         LOMROG12-PC07         0.315         40         10         2         20         0.272         0.025         0.124         0.013           15869         LOMROG12-PC07         0.780         52         9         1         11         0.294         0.026         0.122         0.011           15870         LOMROG12-PC07         1.700         79         9         3         33         0.373         0.029         0.189         0.022           15871         LOMROG12-PC07         1.425         96         9         3         33         0.373         0.029         0.183         0.022           15873         LOMROG12-PC07         1.580         1111         9         2         22		LOMROG07-PC04		. ,	9	2	22		0.069	0.230	0.066
15866         LOMROG12-PC07         0.020         9         9         2         22         0.081         0.004         0.034         0.001           15867         LOMROG12-PC07         0.165         31         10         1         10         0.205         0.036         0.090         0.021           15868         LOMROG12-PC07         0.315         40         10         2         20         0.272         0.025         0.124         0.013           15869         LOMROG12-PC07         0.780         52         9         1         11         0.294         0.026         0.122         0.011           15870         LOMROG12-PC07         0.780         52         9         1         11         0.294         0.026         0.122         0.011           15870         LOMROG12-PC07         1.170         79         9         3         33         0.373         0.029         0.189         0.021           15871         LOMROG12-PC07         1.425         96         9         3         33         0.339         0.355         0.183         0.021           15873         LOMROG12-PC07         1.580         111         9         2         22         0.36	<sup>b</sup> 21594	LOMROG07-PC04	3.000	,	7	4	57	0.270	0.099	0.136	0.057
15868         LOMROG12-PC07         0.315         40         10         2         20         0.272         0.025         0.124         0.012           15869         LOMROG12-PC07         0.780         52         9         1         11         0.294         0.026         0.122         0.012           15870         LOMROG12-PC07         0.980         63         6         1         177         0.354         0.024         0.163         0.021           15871         LOMROG12-PC07         1.170         79         9         3         33         0.373         0.029         0.189         0.021           15872         LOMROG12-PC07         1.425         96         9         3         33         0.399         0.035         0.188         0.021           15873         LOMROG12-PC07         1.425         96         9         3         33         0.399         0.035         0.188         0.021           15873         LOMROG12-PC07         1.580         111         9         2         22         0.368         0.030         0.168         0.021           15874         LOMROG12-PC07         3.537         350         9         3         33         0.	15866	LOMROG12-PC07	0.020		9	2	22	0.081	0.004	0.034	0.002
15869         LOMROG12-PC07         0.780         52         9         1         11         0.294         0.026         0.122         0.012           15870         LOMROG12-PC07         0.980         63         6         1         17         0.354         0.024         0.163         0.021           15871         LOMROG12-PC07         1.170         79         9         3         33         0.373         0.029         0.189         0.021           15872         LOMROG12-PC07         1.425         96         9         3         33         0.399         0.035         0.183         0.021           15873         LOMROG12-PC07         1.580         111         9         2         22         0.368         0.030         0.168         0.021           15874         LOMROG12-PC07         1.720         124         9         3         33         0.355         0.024         0.173         0.024           15875         LOMROG12-PC07         3.537         350         9         3         33         0.355         0.036         0.133         0.014           15876         LOMROG12-PC07         3.657         374         9         0         0         0.3	15867	LOMROG12-PC07	0.165	31	10	1	10	0.205	0.036	0.090	0.022
15869         LOMROG12-PC07         0.780         52         9         1         11         0.294         0.026         0.122         0.012           15870         LOMROG12-PC07         0.980         63         6         1         17         0.354         0.024         0.163         0.021           15871         LOMROG12-PC07         1.170         79         9         3         33         0.373         0.029         0.189         0.021           15872         LOMROG12-PC07         1.425         96         9         3         33         0.399         0.035         0.183         0.021           15873         LOMROG12-PC07         1.580         111         9         2         22         0.368         0.030         0.168         0.021           15874         LOMROG12-PC07         1.720         124         9         3         33         0.355         0.024         0.173         0.024           15875         LOMROG12-PC07         3.537         350         9         3         33         0.355         0.036         0.133         0.014           15876         LOMROG12-PC07         3.657         374         9         0         0         0.3	15868	LOMROG12-PC07	0.315	40	10	2	20	0.272	0.025	0.124	0.013
15871         LOMROG12-PC07         1.170         79         9         3         33         0.373         0.029         0.189         0.021           15872         LOMROG12-PC07         1.425         96         9         3         33         0.399         0.035         0.189         0.021           15872         LOMROG12-PC07         1.425         96         9         3         33         0.399         0.035         0.183         0.021           15873         LOMROG12-PC07         1.580         111         9         2         22         0.368         0.030         0.168         0.021           15874         LOMROG12-PC07         1.720         124         9         3         33         0.385         0.024         0.173         0.024 <sup>b</sup> 15875         LOMROG12-PC07         3.537         350         9         3         33         0.305         0.036         0.133         0.014           15876         LOMROG12-PC07         3.657         374         9         0         0         0.354         0.048         0.185         0.03           15877         LOMROG12-PC07         4.210         485         6         3         50	15869	LOMROG12-PC07	0.780	52	9	1	11	0.294	0.026	0.122	0.019
15872         LOMROG12-PC07         1.425         96         9         3         33         0.399         0.035         0.183         0.02           15873         LOMROG12-PC07         1.580         111         9         2         22         0.368         0.030         0.168         0.02           15873         LOMROG12-PC07         1.720         124         9         3         33         0.385         0.024         0.173         0.02 <sup>b</sup> 15875         LOMROG12-PC07         3.537         350         9         3         33         0.305         0.036         0.133         0.01 <sup>b</sup> 15875         LOMROG12-PC07         3.657         374         9         0         0         0.354         0.048         0.185         0.03           15876         LOMROG12-PC07         3.657         374         9         0         0         0.354         0.048         0.185         0.03           15877         LOMROG12-PC07         3.787         402         4         3         75         0.348         0.174           15878         LOMROG12-PC07         4.210         485         6         3         50         0.402         0.012         0	15870	LOMROG12-PC07	0.980	63	6	1	17	0.354	0.024	0.163	0.022
15872LOMROG12-PC071.4259693330.3990.0350.1830.0215873LOMROG12-PC071.58011192220.3680.0300.1680.02415874LOMROG12-PC071.72012493330.3850.0240.1730.024b15875LOMROG12-PC073.53735093330.3050.0360.1330.01415876LOMROG12-PC073.6573749000.3540.0480.1850.0315877LOMROG12-PC073.78740243750.3480.1740.01415878LOMROG12-PC074.21048563500.4020.0120.1930.01b15879LOMROG12-PC074.40550272290.3350.0140.1550.014b15879LOMROG12-PC074.51051053600.3070.0290.1450.03b15870LOMROG12-PC074.51051053600.3070.0290.1450.0322765LOMROG12-PC090.80055172120.3790.0120.1970.01422766LOMROG12-PC091.25012910000.4000.0290.2090.0322767LOMROG12-PC091.840205122170.4160.0230.2070.02 </td <td>15871</td> <td>LOMROG12-PC07</td> <td>1.170</td> <td>79</td> <td>9</td> <td>3</td> <td>33</td> <td>0.373</td> <td>0.029</td> <td>0.189</td> <td>0.028</td>	15871	LOMROG12-PC07	1.170	79	9	3	33	0.373	0.029	0.189	0.028
15874         LOMROG12-PC07         1.720         124         9         3         33         0.385         0.024         0.173         0.024 <sup>b</sup> 15875         LOMROG12-PC07         3.537         350         9         3         33         0.385         0.024         0.173         0.024           15875         LOMROG12-PC07         3.537         350         9         3         33         0.305         0.036         0.133         0.013           15876         LOMROG12-PC07         3.657         374         9         0         0         0.354         0.048         0.185         0.03           15877         LOMROG12-PC07         3.787         402         4         3         75         0.348         0.174           15878         LOMROG12-PC07         4.210         485         6         3         50         0.402         0.012         0.193         0.01 <sup>b</sup> 15879         LOMROG12-PC07         4.405         502         7         2         29         0.335         0.014         0.155         0.014 <sup>b</sup> 15879         LOMROG12-PC07         4.510         510         5         3         60         0.307         0.029	15872	LOMROG12-PC07	1.425		9	3	33	0.399	0.035	0.183	0.027
b15875         LOMROG12-PC07         3.537         350         9         3         33         0.305         0.036         0.133         0.011           15876         LOMROG12-PC07         3.657         374         9         0         0         0.354         0.048         0.185         0.03           15876         LOMROG12-PC07         3.657         374         9         0         0         0.354         0.048         0.185         0.03           15877         LOMROG12-PC07         3.787         402         4         3         75         0.348         0.174           15878         LOMROG12-PC07         4.210         485         6         3         50         0.402         0.012         0.193         0.01           b15879         LOMROG12-PC07         4.405         502         7         2         29         0.335         0.014         0.155         0.011           b15880         LOMROG12-PC07         4.510         510         5         3         60         0.307         0.029         0.145         0.031           22765         LOMROG12-PC09         0.800         55         17         2         12         0.379         0.012	15873	LOMROG12-PC07	1.580	111	9	2	22	0.368	0.030	0.168	0.024
15876         LOMROG12-PC07         3.657         374         9         0         0         0.354         0.048         0.185         0.03           15877         LOMROG12-PC07         3.787         402         4         3         75         0.348         0.174           15878         LOMROG12-PC07         4.210         485         6         3         50         0.402         0.193         0.01 <sup>b</sup> 15879         LOMROG12-PC07         4.405         502         7         2         29         0.335         0.014         0.155         0.012 <sup>b</sup> 15879         LOMROG12-PC07         4.510         510         5         3         60         0.307         0.029         0.145         0.03           15880         LOMROG12-PC09         4.510         510         5         3         60         0.307         0.029         0.145         0.03           22765         LOMROG12-PC09         0.800         55         17         2         12         0.379         0.012         0.197         0.011           22766         LOMROG12-PC09         1.250         129         10         0         0         0.400         0.029         0.209         <	15874	LOMROG12-PC07	1.720	124	9	3	33	0.385	0.024	0.173	0.025
15876         LOMROG12-PC07         3.657         374         9         0         0         0.354         0.048         0.185         0.03           15877         LOMROG12-PC07         3.787         402         4         3         75         0.348         0.174           15878         LOMROG12-PC07         4.210         485         6         3         50         0.402         0.012         0.193         0.01 <sup>b</sup> 15879         LOMROG12-PC07         4.405         502         7         2         29         0.335         0.014         0.155         0.012 <sup>b</sup> 15879         LOMROG12-PC07         4.510         510         5         3         60         0.307         0.029         0.145         0.03           15880         LOMROG12-PC09         4.510         510         5         3         60         0.307         0.029         0.145         0.03           22765         LOMROG12-PC09         0.800         55         17         2         12         0.379         0.012         0.197         0.01           22766         LOMROG12-PC09         1.250         129         10         0         0         0.400         0.029 <t< td=""><td><sup>b</sup>15875</td><td>LOMROG12-PC07</td><td>3.537</td><td></td><td>9</td><td>3</td><td>33</td><td>0.305</td><td>0.036</td><td>0.133</td><td>0.018</td></t<>	<sup>b</sup> 15875	LOMROG12-PC07	3.537		9	3	33	0.305	0.036	0.133	0.018
15877         LOMROG12-PC07         3.787         402         4         3         75         0.348         0.174           15878         LOMROG12-PC07         4.210         485         6         3         50         0.402         0.012         0.193         0.01 <sup>b</sup> 15879         LOMROG12-PC07         4.405         502         7         2         29         0.335         0.014         0.155         0.012           15880         LOMROG12-PC07         4.510         510         5         3         60         0.307         0.029         0.145         0.033           12765         LOMROG12-PC09         0.800         55         17         2         12         0.379         0.012         0.197         0.014           22766         LOMROG12-PC09         1.250         129         10         0         0         0.400         0.029         0.209         0.033           22766         LOMROG12-PC09         1.250         129         10         0         0         0.400         0.029         0.209         0.033           22767         LOMROG12-PC09         1.840         205         12         2         17         0.416         0.023	15876	LOMROG12-PC07			9	0	0	0.354	0.048	0.185	0.031
15878         LOMROG12-PC07         4.210         485         6         3         50         0.402         0.012         0.193         0.01 <sup>b</sup> 15879         LOMROG12-PC07         4.405         502         7         2         29         0.335         0.014         0.155         0.012         0.193         0.011           15880         LOMROG12-PC07         4.510         510         5         3         60         0.307         0.029         0.145         0.032           22765         LOMROG12-PC09         0.800         55         17         2         12         0.379         0.012         0.197         0.014           22766         LOMROG12-PC09         1.250         129         10         0         0         0.400         0.029         0.209         0.033           22766         LOMROG12-PC09         1.250         129         10         0         0         0.400         0.029         0.209         0.033           22767         LOMROG12-PC09         1.840         205         12         2         17         0.416         0.023         0.207         0.023	15877	LOMROG12-PC07	3.787		4	3	75	0.348		0.174	
b15879         LOMROG12-PC07         4.405         502         7         2         29         0.335         0.014         0.155         0.011           15880         LOMROG12-PC07         4.510         510         5         3         60         0.307         0.029         0.145         0.033           22765         LOMROG12-PC09         0.800         55         17         2         12         0.379         0.012         0.197         0.011           22766         LOMROG12-PC09         1.250         129         10         0         0         0.400         0.029         0.209         0.033           22767         LOMROG12-PC09         1.840         205         12         2         17         0.416         0.023         0.207         0.024		LOMROG12-PC07			6		50		0.012		0.011
15880         LOMROG12-PC07         4.510         510         5         3         60         0.307         0.029         0.145         0.037           22765         LOMROG12-PC09         0.800         55         17         2         12         0.379         0.012         0.197         0.014           22766         LOMROG12-PC09         1.250         129         10         0         0         0.400         0.029         0.209         0.033           22767         LOMROG12-PC09         1.840         205         12         2         17         0.416         0.023         0.207         0.024	<sup>b</sup> 15879	LOMROG12-PC07	4.405		7	2	29	0.335	0.014	0.155	0.012
22765         LOMROG12-PC09         0.800         55         17         2         12         0.379         0.012         0.197         0.013           22766         LOMROG12-PC09         1.250         129         10         0         0         0.400         0.029         0.209         0.033           22767         LOMROG12-PC09         1.840         205         12         2         17         0.416         0.023         0.207         0.023	15880	LOMROG12-PC07					60		0.029	0.145	0.034
22766         LOMROG12-PC09         1.250         129         10         0         0         0.400         0.029         0.209         0.039           22767         LOMROG12-PC09         1.840         205         12         2         17         0.416         0.023         0.207         0.023	22765	LOMROG12-PC09			17	2	12	0.379		0.197	0.010
22767 LOMROG12-PC09 1.840 205 12 2 17 0.416 0.023 0.207 0.02											0.030
											0.027
	22768	LOMROG12-PC09	1.980	233	17	3	18	0.435	0.019	0.232	0.024

22769	LOMROG12-PC09	2.160	276	13	1	8	0.475	0.053	0.278	0.040
⁰22770	LOMROG12-PC09	3.400	603	11	4	36	0.407	0.005	0.203	0.006
	Iceland Sea									
22745	ODP 151/ 907A	1.050	42	14	2	14	0.200	0.008	0.079	0.006
22746	ODP 151/ 907A	1.870	107	13	2	15	0.281	0.005	0.114	0.003
22747	ODP 151/ 907A	2.350	131	17	2	12	0.325	0.011	0.146	0.009
22748	ODP 151/ 907A	3.060	168	15	0	0	0.355	0.011	0.171	0.009
22749	ODP 151/ 907A	5.060	309	17	2	12	0.395	0.016	0.192	0.014
22750	ODP 151/ 907A	6.810	398	12	1	8	0.388	0.013	0.183	0.016
22751	ODP 151/ 907A	15.210	781	10	3	30	0.482	0.036	0.250	0.046
	Greenland Sea									
22732	PS17 / 1906-2	0.150	10	12	3	25	0.129	0.003	0.067	0.004
22733	PS17 / 1906-2	1.805	85	17	0	0	0.263	0.008	0.111	0.008
22734	PS17 / 1906-2	2.005	111	12	0	0	0.306	0.006	0.138	0.005
22735	PS17 / 1906-2	2.105	117	12	4	33	0.312	0.010	0.141	0.009
22736	PS17 / 1906-2	2.205	122	12	3	25	0.347	0.011	0.169	0.012
22737	PS17 / 1906-2	3.290	207	8	1	13	0.343	0.011	0.171	0.008
22738	PS17 / 1906-2	3.605	225	12	2	17	0.355	0.010	0.178	0.010
22739	PS17 / 1906-2	5.505	398	5	1	20	0.439	0.030	0.227	0.036
Cibicido	oides wuellerstorfi									
	Alpha Ridge									
17341	AO16-9-PC1	0.190	16	10	0	0	0.235	0.061	0.099	0.040
17342	AO16-9-PC1	0.210	22	10	1	10	0.326	0.027	0.158	0.020
17343	AO16-9-PC1	0.970	127	10	0	0	0.502	0.005	0.311	0.010
17344	AO16-9-PC1	1.260	188	10	0	0	0.522	0.007	0.321	0.008
17346	AO16-9-PC1	2.415	599	10	2	20	0.523	0.101	0.302	0.094
	Lomonosov Ridge									
17331	AO16-5-PC1	0.090	30	10	1	10	0.342	0.012	0.155	0.006
17333	AO16-5-PC1	1.370	105	7	1	14	0.525	0.020	0.339	0.032
22752	LOMROG12-TWC03	0.025	16	13	1	8	0.144	0.012	0.047	0.006
22753	LOMROG12-TWC03	0.095	38	18	3	17	0.157	0.014	0.053	0.006
17327	LOMROG12-TWC03	0.215	44	8	0	0	0.341	0.006	0.161	0.006
22754	LOMROG12-PC03	0.520	66	13	3	23	0.412	0.014	0.208	0.013
17328	LOMROG12-PC03	0.540	98	8	0	0	0.433	0.009	0.234	0.009
17329	LOMROG12-PC03	0.741	198	8	1	13	0.485	0.008	0.284	0.008
17330	LOMROG12-PC03	1.271	63	8	1	13	0.541	0.010	0.328	0.019
<sup>b</sup> 22755	LOMROG12-PC03	1.441	247	9	1	11	0.491	0.017	0.267	0.035
22756	LOMROG12-PC09	0.800	73	12	1	8	0.433	0.011	0.236	0.011
22757	LOMROG12-PC09	1.250	140	22	2	9	0.476	0.007	0.266	0.011
22758	LOMROG12-PC09	1.980	224	5	0	0	0.508	0.015	0.281	0.015
	Iceland Sea									
22740	ODP 151/ 907A	1.050	42	9	3	33	0.166	0.026	0.059	0.013
22741	ODP 151/907A	1.870	107	21	1	5	0.337	0.015	0.151	0.010
<sup>b</sup> 22742	ODP 151/ 907A	2.350	131	15	6	40	0.160	0.013	0.064	0.006
22743	ODP 151/ 907A	3.060	168	10	1	10	0.388	0.025	0.198	0.026
22744	ODP 151/ 907A	15.210	781	18	1	6	0.549	0.016	0.345	0.032
0070	Greenland Sea	0.155					0.1==	0.000	0.055	
22724	PS17 / 1906-2	0.150	10	15	0	0	0.175	0.009	0.059	0.004
22725	PS17 / 1906-2	1.805	85	18	3	17	0.328	0.010	0.144	0.007
22726	PS17 / 1906-2	2.005	111	20	1	5	0.351	0.016	0.160	0.007
22727	PS17 / 1906-2	2.105	117	18	1	6	0.357	0.009	0.171	0.007
22728	PS17 / 1906-2	2.205	122	21	1	5	0.379	0.016	0.191	0.015
22729	PS17 / 1906-2	3.290	207	6	1	17	0.443	0.050	0.258	0.056
<sup>b</sup> 22730	PS17 / 1906-2	3.605	225	6	0	0	0.343	0.014	0.160	0.023
22731	PS17 / 1906-2	5.505	398	6	0	0	0.517	0.018	0.331	0.026

Table 2: Extent of racemization (D/L) for aspartic acid (Asp) and glutamic (Glu)-acid (Glu) in samples of Neogloboquadrina pachyderma and Cibicidoides wuellerstorfi collected from the Arctic Ocean sediment cores. Reported ages are from published age models (Table 1).

<sup>a</sup> Number of subsamples used to calculate the mean and standard deviation <sup>b</sup> Stratigraphically reversed sample

#### b) Inter-species comparison

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The overall subsample rejection rate was higher for N. pachyderma (18.222.7 %) than for C. wuellerstorfi (9.8-9%); (and only subsamples of N. pachyderma were destroyed during laboratory analyses (Table 3). Only one C. wuellerstorfi subsample was rejected due to high serine content (L-Ser / L-Asp  $\geq 0.8$ ), significantly fewer than those for N. pachyderma, implying that secondary amino acids (contamination<sup>2</sup>) were not introduced during core storage or laboratory analysis because both species were treated similarly. C. wuellerstorfi subsamples were rejected more frequently as statistical outliers than were N. pachyderma. This could reflect the true heterogeneity of ages of individual tests, which is revealed when fewer individuals are averaged together in a 210 single subsample, especially where sedimentation rates are low (e.g. Dolman et al., 2021).

Species	Total number	L-Ser / L-Asp	Non-covarying D/L Asp	D/L Asp or D/L Glu not within
	of subsamples	≥ 0.8	and D/L Glu	$\pm 2\sigma$ of sample mean
N. pachyderma	635	82 <u>81</u>	<u>2220</u>	15 <u>43</u>
C. wuellerstorfi	374	1	-18 <u>16</u>	48 <u>20</u>

Table 3: Number of rejected subsamples per rejection criterion.

215 The proportion of samples with high intra-sample variability (coefficient of variation for D/L Asp and Glu > 10%, following data screening) was approximately twice as high for N. pachyderma as for C. wuellerstorfi.

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As some samples (n = 21) from certain stratigraphic intervals contained specimens of both N. pachyderma and C. wuellerstorfi, this allowed a direct comparison of the rates of racemization in the two species (Fig. 3). One stratigraphically reversed sample was omitted prior to analysis. The slope of the least-squares regression fit to the D/L versus D/L data indicates that Asp racemized, on average,  $\frac{1615\pm2}{2}$  % ( $n = \frac{2019}{2}$ ) faster in C. wuellerstorfi than in N. pachyderma, and similarly, Glu also racemized faster  $(23\pm3-2\%)$  in C. wuellerstorfi than in N. pachyderma.

The rate of amino acid racemization varies between different foraminifera species (King and & Neville, 1977).



Figure 3: Extent of racemization for aspartic acid (Asp) and glutamic acid (Glu) in *Neogloboquadrina pachyderma* and *Cibicidoides wuellerstorfi* from samples from the same stratigraphic depths collected from different regions of the Arctic Ocean. Error bars represent  $\pm 1\sigma$  intra-sample variability, solid line is least squares regression with 95% confidence shown in grey, and the dashed line corresponds tois the line of equality. Open symbols mark the samples excluded from regression analysis.

### c) <u>Relationship between D/L values and age / stratigraphic depth</u>

The extent of racemization (D/L values) for Asp and Glu show a systematic increasing trend with increasing sample age in of samples of both species from the Greenland and Iceland seas, and follow a simple power function (Fig. 4). A Ssimple power lawtransformation was used to model the forward rate of racemization in this study, as this is typical for racemization of amino acids in biominerals held under isothermal conditions (e.g. Kaufman, 2006; Clarke and Murray-Wallace, 2006).

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Figure 4: Extent of racemization for aspartic acid (Asp) and glutamic acid (Glu) in samples of *N. pachyderma* and *C. wuellerstorfi* from sediment cores PS17/1906-2 (Greenland Sea) and ODP 151/907A (Iceland Sea). Error bars represent  $\pm 1\sigma$  intra-sample variability and curves are power functions. Stratigraphically reversed samples are marked with unfilled symbols and were excluded to maximise-improve the goodness of fit.

D/L values obtained from sediment cores from the central Arctic Ocean, where age-depth models are less certain, are displayed on the ACEX composite depth scale based on correlations using bulk density profiles (Fig. 5). The extent of racemization follows a simple power function (stratigraphically reversed samples excluded), as also observed in samples from the Nordic Seas, and D/L values generally overlap between correlative samples from multiple cores. This directly supports the accuracy of the lithostratigraphic correlations established between among the sites. D/L values from *N. pachyderma* appear to reach a plateau at ~0.4 for Asp and ~0.2 for Glu (~6 m depth in the ACEX record). Plateauing of D/L Asp values was previously documented in other Arctic Ocean cores as well (Kaufman et al., 2008). It is unclear whether plateauing is present in *C. wuellerstorfi* 

samples, as only a few samples were analysed in the corresponding age range, and the oldest sample has the largest intra-sample variability.



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Figure 5: Extent of racemization for aspartic acid (Asp) and glutamic acid (Glu) in *N. pachyderma* and *C. wuellerstorfi* from the central Arctic Ocean displayed on the ACEX composite depth. Sediment cores from investigated in this study were grouped based on their geographical location (Alpha Ridge – red, Lomonosov Ridge – blue). Error bars are  $\pm 1\sigma$  intra-sample variability, <u>curves are power functions</u> and unfilled symbols mark stratigraphically reversed samples.

#### 265 4. Discussion

Aspartic acid and glutamic acid racemization analyses of multiple foraminifera taxa obtained from cold bottom water sites across the globe showed that sample ages can be confidently estimated by calibrated age equations, which relate the extent of racemization of the amino acids to independently determined sample age (Kaufman et al., 2013). These globally derived age equations are based on both planktic and benthic foraminifera species

with *N. pachyderma* contributing up to ~21% of all samples, but <u>exclude</u>-*C. wuellerstorfi* is not represented.-Ages derived using the global age equations generally agree with independently derived ages at the Yermak Plateau (West et al., 2019), but do not agree with <u>working previously published</u> age models applied to from the central Arctic Ocean, beyond about 40 ka. If existing age models in the central Arctic Ocean are correct, the extent of racemization for *N. pachyderma* is higher than expected when compared to <u>with</u> other oceans (Kaufman et al., 2008, 2013).

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The results of AAR presented here show that Asp and Glu racemize in a predictable manner in both *N. pachyderma* and *C. wuellerstorfi* samples from the Nordic Seas (Fig. 4), although the rates differ systematically between the species (Fig. 3). Both D/L Asp and D/L Glu values obtained from *N. pachyderma* samples from the Nordic Seas clearly follow a trend previously observed at the Yermak Plateau (Fig. 6), implying that racemization kinetics are indistinguishable in these areas. These data from independently dated cores from coldwater sites provide further support for the integrity of the AAR technique, and for the globally calibrated age equations of Kaufman et al. (2013).



Figure 6: Extent of racemization in aspartic acid (Asp) and glutamic acid (Glu) in Neogloboquadrina pachyderma and Cibicidoides wuellerstorfi from the Greenland and Iceland seas and the Yermak Plateau. Black,/ green and /red lines mark best fit linescurves are power functions fit to the data. Blue curves are D/L Asp and Glu values and associated sample ages, as predicted by the globally calibrated age equations of Kaufman et al. (2013), are shown in blue. Data for the Yermak Plateau from West et al. (2019). Error bars represent  $\pm 1\sigma$  intra-sample variability.

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300 this species suggests that D/L values should not be used in combination with the equations without adjustment (Fig. 6).

The purportedly higher rates of racemization in *N. pachyderma* from the central Arctic Ocean were argued to be caused by factors other than taxonomic effects (Kaufman et al., 2013). Either racemization generally progresses

- 305 at a higher rate here due to an unknown reason, or existing age models used to constrain the rate of AAR from the central Arctic Ocean underestimate the true age of deposition. Given the existing age models, higher-thanexpected rates of racemization are also observed in this study in both *N. pachyderma* and *C. wuellerstorfi* samples from the central Arctic Ocean (Fig. 7). Differences between ages predicted by the global calibrated age equation and currently available sample ages based on the ACEX age model are larger for *C. wuellerstorfi*, as
- 310 | expected, since we established that AAR is racemization proceeds faster in this taxon than in *N. pachyderma*.



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Figure 7: Extent of racemization for aspartic acid (Asp) and glutamic acid (Glu) in *N. pachyderma* and *C. wuellerstorfi* from the central Arctic <u>Ocean</u>. D/L values are displayed against the ACEX age model. D/L values versus age trend defined by the globally calibrated age equations (Kaufman et al., 2013) is shown in blue. Error bars represent  $\pm 1\sigma$  intra-sample variability.

320 The AAR trends in *C. wuellerstorfi* samples from the Nordic Seas, where age models are better constrained, can also be compared with those in the central Arctic Ocean samples. The racemization of Asp and Glu in *C. wuellerstorfi* samples from both the Greenland and Iceland seas (GIS) follow the same trend (Fig. 4), which can be approximated with regression analysis. Following the removal of stratigraphically reversed samples (*n* = 3) and samples with high analytical uncertainty (coefficient of variation > 10 %, (*n*=5), simple power models
325 <u>functions (Eq. 1 and 2)</u>

t =  $3827.6 * (D/L Asp)^{3.395}$  (1) t =  $4979.1 * (D/L Glu)^{2.138}$  (2) (where t = age in ka) fit the D/L Asp and D/L Glu versus age relationship well for the past 400 ka (the period for which these models are robust). <u>A, and can be applying edthese equations</u> to D/L values in *C. wuellerstorfi* from the central Arctic Ocean. This reveals that sample ages are younger than predicted by the model based on the Nordic Sea samples (Fig. 8). The higher-than-expected D/L values observed for *C. wuellerstorfi* from the central Arctic Ocean confirm earlier findings, which argued that higher racemization rates in the central Arctic Ocean are not the result of taxonomic effects (Kaufman et al., 2013).



Figure 8. Aspartic acid (Asp) and glutamic acid (Glu) racemization versus sample age in *C. wuellerstorfi* from the central Arctic Ocean. <u>Black curves are the age equations determined Model predictions based on the trend defined</u> by samples from the Greenland and Iceland seas is also shown ('GIS trend)? <u>blue black-line</u>; <u>blue black</u> dashed <u>curveslines</u> mark 95% confidence intervals). Error bars represent  $\pm 1\sigma$  intra-sample variability.

The discrepancy between the globally (*N. pachyderma*) and GIS (*C. wuellerstorfi*) calibrated AAR ages and the established chronology for central Arctic Ocean sediments is illustrated in Figure 9 and Table 4. The calibrated
345 AAR ages suggest that what is intervals previously interpreted as substages in MIS 5 are instead separate interglacial periods extending back to MIS 9. This interpretation would indicate that the diamict unit previously assigned to MIS 6, and representing the onset of a fundamentally different depositional regime in the Arctic characterised by recurrent coarse-grained facies (O'Regan et al., 2010), is likely older than MIS 8 and possibly as old as MIS 12. Exact age determinations are difficult due to a paucity of data from these lower depths, and increased downcore scatter in the stacked AAR results (*discussed below*).

It cannot be excluded that the established chronologies of the central Arctic Ocean sediments underestimate their true ages, but significant shifts (representing multiple glacial cycles) in sediment ages are required. It is important to recognise that this -and-results in a number of fundamental inconsistencies when compared to-with other geochronological data. For example, recent work (O'Regan et al., 2020) placed the first occurrence of the

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7 (~220 ka). The AAR age equations (1) and (2), defined by the trend in *C. wuellerstorfi* samples from the Greenland and Iceland seas, yield estimated ages of  $475\pm12$  ka (D/L Asp) or  $459\pm21$  ka (D/L Glu) (MIS 12/13) based on the mean D/L ±1SE for a sample from 1.27 m in this core. Similarly, the globally calibrated age

equation (Kaufman et al., 2013) returns an MIS 12 age of 427±20 ka for *N. pachyderma* samples from 1.29 m in LOMROG12-PC03. These ages are substantially older than the global evolutionary occurrence of *E. huxleyi* during MIS 8 (Thierstein et al., 1977; Anthonissen and Ogg, 2012). Furthermore, *E. huxleyi* is abundant in the 0.58—0.82 mbsf core interval of LOMROG12-PC03, which was assigned an MIS 5 age (O'Regan et al., 2020). In contrast, the AAR trend from the Greenland and Iceland seas assigns ages of 189±6 —to 328±7 ka (MIS 6—

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In contrast, the AAR trend from the Greenland and Iceland seas assigns ages of 189±6 <u>to</u>\_328±7 ka (MIS 6— 9) for the 0.52—0.74 m core depths (Table 4). Although <u>T</u>this could <u>only</u> be reconciled with the existence of *E*. *huxleyi* in this interval, <u>assuming-if</u> it <u>had</u> entered the Arctic shortly after its first evolutionary occurrence<sub>25</sub> <u>However, the-tThe</u> MIS 6-9 age is not consistent with results from optically stimulated luminescence dating in stratigraphically co-eval sediments from another core on the Lomonosov Ridge, which support the MIS 5 age assignment (Jakobsson et al., 2003).

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Stratigraphic depth (m)	ACEX equivalent depth (m)	<u>Marine Isotope Stage (MIS) correlation</u> Estimated age (ka)							
		ACEX age model (Backman et al., 2008; O'Regan et al., 2008) and <i>E. huxleyii</i> occurrences (O'Regan et al., 2020)	AAR geochronology – aspartic acid in <i>N.</i> <i>pachyderma</i> (Kaufman et al., 2013)	AAR geochronology – aspartic acid in <i>C.</i> <i>wuellerstorfi</i> (this study)					
0.52-0.74	1.96-3.35	MIS 5	MIS 6-9	MIS 6-9					
1.27-1.39	4.62-5.44	MIS 7	MIS 9-12	MIS 9-12					

Table 4: Age estimates for selected stratigraphic intervals in <u>core</u> LOMROG12-PC03 based on the ACEX age model and occurrences of *E. huxleyi*, and AAR geochronology. <u>MIS – marine oxygen isotope stage</u>.

On the other hand, sedimentation rates predicted byestimated using the AAR global and GIS calibration equations are consistent with radiocarbon derived estimates over the past 40 ka, but beyond this point remain intermediate between the proposed age model for the ACEX record (O'Regan et al., 2008) and those recently inferred from <sup>230</sup>Th and <sup>231</sup>Pa extinction ages in sedimentary sequences from this region of the Lomonosov Ridge (Hillaire-Marcel et al., 2017; Purcell et al., 2022). Significant Additional work is required to understand the origin and resolve the differences between these geochronological approaches. However, a critical observation is that even the older estimated ages from the global and GIS calibration equations remain considerably younger than the long-overturned paleomagnetic-derived age model that would place the Bruhnes-Matuyama boundary (~780 ka) at approximately 5 m depth in the ACEX record (O'Regan et al., 2008) (Fig. 9).



Figure 9: Alternative age—depth relationships in sediment cores from the central Arctic Ocean. Circles and diamonds mark ages estimated by globally calibrated rates of racemization of aspartic acid in *N. pachyderma* (Kaufman et al., 2013) and GIS-calibrated rates of racemization of aspartic acid in *C. wuellerstorfi* (this study), respectively. Error bars represent 95 % confidence intervals. The ACEX age model is based on Backman et al., (2008), Frank et al., (2008), and O'Regan et al., (2008). Also shown are ACEX digital core image, marine oxygen isotope stage (MIS) boundaries based on the ACEX age model, bulk density (ρ) profile of the ACEX core, and the global benthic δ<sup>18</sup>O record and corresponding oxygen isotope stages (MIS 1-15) based on Lisiecki & Raymo (2005).

An alternative to central Arctic Ocean age models underestimating the true age of sediments is that the assumption that the rate of AAR in foraminifera from the central Arctic Ocean is faster than in the eastern Arctic 395 Ocean (West et al., 2019), the Nordic Seas (this study), and the same as from other globally distributed cold water sites (Kaufman et al., 2013)-could be invalid. A fundamental premise of this study is that the investigated central Arctic Ocean deep-sea sites have experienced similar temperatures histories to those other cold water sites, which changed similarly over time. Modern bottom water temperatures near the coring sites are very similar (Table 1), but they may have differed in the past. Cronin et al. (2012) showed that the central Arctic 400 basin waters, at ~1000-2500 m depth interval, were 1-2°C warmer during the past 50-11 ka than today. While this might account for part of the apparently higher rate of AAR in the central Arctic Ocean, West et al. (2019) showed that more substantial differences ( $\sim >4$  °C) in effective diagenetic temperatures would be required to achieve the differences observed between D/L values of equivalent age samples from sediment cores from the central Arctic Ocean and those from the Nordic Seas. Available heat flow data (Shephard et al., 2018) show that 405 geothermal flux is not unusually high in the central Arctic Ocean when compared to the Yermak Plateau, and thus cannot explain the apparently higher rates of racemization. Therefore, while offsets in D/L values in stratigraphically co-eval sections of different central Arctic Ocean cores in this study might, in part, be attributed to differences in bottom water temperatures, such differences are unlikely able to explain the overall higher inferred rates of racemization compared to other global sites.

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D/L values in many samples of *N. pachyderma* from the central Arctic Ocean cores exhibit a distinct plateauing below the ~5.5—6 m composite ACEX depth (Fig. 5 and 9). Some of these samples <u>can be considered asare</u> stratigraphically reversed (i.e. their D/L values are lower than of those from shallower depths). Such plateauing of D/L values was previously observed in <u>cores from</u> other areas of the Arctic Ocean (Kaufman et al., 2008), but its cause remains unclear. While sediment mass movements, glacial erosion and subsequent re-deposition are known to occur across the Arctic Ocean (e.g. Jakobsson & O'Regan, 2016; Boggild et al., 2020; Pérez et al., 2020; Schlager et al., 2021), this explanation would require that almost all of the material above 5.5—6 m composite depth was reworked similarly across multiple cores, which seems <u>highly untenableunlikely</u>.

Local differences in sedimentation rates might also account for some of the observed scatter in AAR results. For example, there are pronounced regional differences in the thickness of correlative units in the studied cores (Fig. 2), implying highly variable sedimentation rates between the sites. In cores with lower sedimentation rates, the influence of bioturbation could conceivably introduce significant scatter though mixing of individuals withof different depositional ages.

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Overall, sSedimentation rates in the central Arctic Ocean are much lower than other, morein marginal areas closer to the shelves (Backman et al., 2004), and can be greatly reduced during glacials in some regions of the Arctic Ocean (Jakobsson et al., 2014). Conversely, thick diamict units suggest rapid influxes of ice rafted material during some glacials or glacial terminations. These punctuated episodes of sedimentation can not only introduce hiatuses, but also impact the length of time biocarbonates are exposed to microbial activity on the seafloor (Sejrup and Haugen, 1994), which in turn can speed up or slow down organic diagenetic processes that influence the rate of AAR. For example, w- Local differences in sedimentation rates (and temperatures) might also account for some of the observed scatter in AAR results.

- 435 Microbial influences in central Arctic Ocean sediments might account for a unique control on racemisation racemization rates and could explain the large offsets in the inferred AAR rates between the Arctic and global oceans. More specifically, where sedimentation rates are low, greater microbial activity could increase organic diagenesis and lead to higher apparent rates of AAR. On the other hand, continuous turnover and reworking of microbial necromass within the foraminifera test could instead lower the apparent rate of racemization via
   440 regeneration of L<sub>-</sub>-amino acids, and alter the apparent AAR rates, as has been documented for bulk organic matter in marine sediments (Braun et al., 2017). The composition of the microbial community may also have an
- <u>impact.</u> Furthermore, Kubota et al. (2016) isolated alphaproteobacteria from deep-sea sediments from the Sagami Bay (Japan), which exclusively utilised D amino acids as a carbon and nitrogen source. If such microbes were also present within foraminifera tests of the central Arctic Ocean, they could potentially alter the progress of racemization. Apparent plateauing of D/L values (e.g. Fig. 5, 7, 9) could be associated with these processes, but would require a distinctively different microbial sedimentary environment in the central Arctic Ocean than elsewhere (such as the Yermak Plateau or Norwegian-Greenland Sea, where the global age-equation appears

applicable). This is not improbable inconceivable, ; for example, as recently Yu et al. (2020) suggested that different marine environments could be characterised by distinct bacterial groups which that utilise D-amino acids.

In the future, pre-treating the foraminifera tests with bleach to isolateIsolation of the intra-crystalline fraction, which approximates a closed behaves as an open-system during diagenesis (Penkman et al., 2008; Wheeler et al., 2021), could-by means of bleaching could minimise the influence of bacterial activity on racemization rates. However, recent work (Millman et al., 2022) showed that bleaching does not necessarily improve the quality of AARthe results in foraminifera, thus the current study used the standard weak oxidative pre-treatment. Future work should investigate how bleaching impacts AAR results from central Arctic Ocean cores. Furthermore, <u>u</u>Understanding of microbial communities and their interactions with their immediate environment in the Arctic Ocean is insufficient and requires future research.

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If the rate of AAR in foraminifera is indeed higher in the central Arctic Ocean, its exact cause remains unknown. The data reported in this study confirm that it is not caused by taxonomic effects. It is observed not only in the planktic *N. pachyderma* but also in the benthic species *C. wuellerstorfi*. We have highlighted the discrepancies that arise in central Arctic Ocean age models if the global (*N. pachyderma*) and GIS (*C. wuellerstorfi*) AAR age equations are applied. This alternate geochronological interpretation should be considered in future attempts to reconcile the currently disparate results that are obtained by applyingfrom different dating techniques applied to Arctic Ocean sediments.

470 If the rate of AAR in foraminifera is indeed higher in the central Arctic Ocean, its exact cause remains
470 unknown, but the data reported in this study confirm that it is not caused by taxonomic effects, as it can be observed not only in the planktic *N. pachyderma* but also in the benthic species *C. wuellerstorfi*. We have highlighted the discrepancies that arise in central Arctic Ocean age models if the global (*N. pachyderma*) and GIS (*C. wuellerstorfi*) AAR age equations are applied. The results warrant a critical evaluation of existing Arctic Ocean age models and the need to more fully assess the environmental factors that may influence
475 racemisation racemization rates in central Arctic Ocean sediments.

#### 5. Conclusions

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- Aspartic <u>acid</u> and glutamic acids racemize faster in *C. wuellerstorfi* than in *N. pachyderma*, and the extent of racemization for these amino acids increases progressively with sample age in both species from multiple sediment cores. Their trends conform to a simple power function.
- *C. wuellerstorfi* samples are characterised by lower intra-sample variability than those of *N. pachyderma*, and this, coupled with a reduced subsample rejection rate and faster sample processing offered by its larger tests make it an preferred appealing target of future AAR studies.

• Ages of *N. pachyderma* samples from the Greenland and Iceland seas agree very well-with ages predicted by previous-globally calibrated age equations for aspartic acid and glutamic acids (Kaufman et al., 2013), and confirm their applicability in these <u>polar</u> regions.

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• The rate of racemization for *C. wuellerstorfi* was calibrated for the past 400 ka using samples from the Greenland and Iceland seas, where sample ages are robust, and D/L values conform tightly to a power trend. Applying this calibration to the *C. wuellerstorfi* samples from the central Arctic <u>Ocean</u> indicates that they are older than their currently accepted ages. This confirms that higher-than-expected D/L values in *N. pachyderma* from in the central Arctic Ocean are not the result of taxonomic effects.

- We cannot find a clear reason why age calibration equations that work globally in the eastern Arctic Ocean, and in the Nordic Seas cannot be applied in the central Arctic Ocean. As such the older ages predicted by these equations for central Arctic Ocean sediments remain a viable option when comparing and assessing results from different approaches to dating Arctic marine sediments.
  - Regardless of the age equation applied, there remains substantial scatter in the AAR age
    predictionsestimates for stratigraphically co-eval intervals in central Arctic Ocean cores. This likely
    reflects a combination of -enhanced mixing in lower sedimentation rate environments, differences in
    effective diagenetic burial temperatures, and potentially the poorly defined role of microbial activity.

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### **Supplementary Material**

Supplementary material associated with this article can be found in the online version.

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#### Data availability

The results of amino acid analyses of all <u>1028–1009</u> subsamples included in this study will be archived at the World Data Service for Paleoclimatology (https://www.ncdc.noaa).

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