1	Late Neogene terrestrial climate reconstruction of the Central
2	Namib Desert derived by the combination of U-Pb silcrete and
3	TCN exposure dating
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16	Keywords: Namib Desert, U-Pb dating, groundwater sil-/calcretes, cosmogenic nuclides
17	Abstract:
18	The chronology of the Cenozoic 'Namib Group' of the Namib Desert <mark>is rather poorly understood</mark>
19	and lacks direct radiometric dating. Thus, the paleoclimate and landscape evolution of the Central
20	Namib Desert remains imprecise, complicating the detailed search for global and/or local forcing
21	factors for the aridification of the Namib. The widespread occurrence of calcretes and silcretes in
22	the Namib Desert allows to apply the novel application of the U-Pb laser ablation dating technique
23	on sil- and calcretes, to date important phases of landscape stability and to retrieve critical
24	paleoclimatic and environmental information on desertification and its paleoclimatic variability.
25	Microscale silcrete formation (max. 8 mm) due to pressure solution by expanding calcrete
26	cementation provides the opportunity to date multiple phases (multiple generation of silcrete as
27	growing layers or shells) of silcrete formation. Groundwater sil- and calcrete formation occurred
28	at our study site during the Pliocene, a period of relatively stable climate and landscape conditions
29	under semi-arid to arid conditions. Terrestrial Cosmogenic Nuclide (TCN) exposure ages from flat
30	canyon rim surfaces indicate the cessation of groundwater calcrete formation due to incision

31 during the Late Pliocene/Early Pleistocene and mark a large-scale landscape rejuvenation due to

32 climate shifts towards more arid conditions in the Pleistocene, which can be connected to global 33 climate patterns. This study demonstrates the feasibility of applying U-Pb laser ablation to groundwater sil- and calcretes, discusses several important issues associated with this technique 34 and opens up the possibility of dating numerous sedimentary sequences containing sil- and 35 calcretes in arid environments. In particular, the use of silcretes (as described above) reduces 36 37 potential effects of detrital components and bulk-signal measurements by using massive calcretes. 38 Our study redefines and improves the generally accepted Late Cenozoic chronostratigraphy of the 39 Namib Desert (Miller, 2008).

# 40 **1. Introduction**

41 In Namibia, widespread calcretes, together with (spatially) more restricted silcretes, are among 42 the most auspicious features of the Cenozoic surface cover and are outcropping along deeply 43 incised ephemeral or fossil drainage systems in terms of their ability to record past environmental 44 change (Miller, 2008; Candy et al., 2004; Summerfield, 1983a; Van Der Wateren and Dunai, 2001; 45 Ward, 1987). As well as being an important component in explaining the generally low denudation 46 rates due to their protective function (Stokes et al., 2007; Nash and Smith, 1998), these sil- and 47 calcretes are also thought to indicate relatively long periods of landscape and climate stability 48 during their formation (Goudie et al., 2015).

49 In general, calcretes are thought to form under semi-arid to arid conditions, with varying 50 interpretations and associations with specific precipitation ranges (Goudie, 2020; Summerfield, 51 1983a; Alonso-Zarza, 2003). Various models have been proposed to explain the different types of 52 calcrete (pedogenic, non-pedogenic, Goudie, 2020). In this study, we will mainly focus on the 53 non-pedogenic, groundwater-related calcrete formation, based on the *per-ascensum* hypothesis 54 (Goudie, 1996; Goudie et al., 2015), which are formed mainly by evaporation from the capillary 55 fringe or below the water table due to changing  $CO_2$  level (Goudie et al., 2015). Most prominent 56 calcrete formations are related to calcretes capping the Karpfenkliff Conglomerate of the Kuiseb 57 Canyon in the Central Namib and the Kamberg Calcrete Formation (Fig. 1, Ward, 1987). Secondary 58 silcrete formation by pressure solution and reprecipitation was synchronous with calcrete 59 formation in the Karpfenkliff conglomerates. It consists of microscale silcrete with discrete 60 multiple layers of silcrete encrusting quartz clasts. The Karpfenkliff Conglomerate overlies the 61 Tsondab Sandstone and was probably deposited in a proto-Kuiseb and a proto-Gaub valley (Ward 62 et al., 1983; Miller, 2008; Ward, 1987).

Calcretes in the central Namib are thought to be at least Early Pleistocene to Pliocene in age (Ward,
1987; Miller, 2008). Common dating techniques used to date calcretes are radiocarbon <sup>14</sup>C, U/Th
disequilibrium, or solution U-Pb. The first two dating methods are limited to ~45 kyr or 500 kyr,

respectively, Calcrete U-Pb laser ablation has recently been used to provide critical chronological 66 67 information on the age-depth relationship of the calcretized sediments from the Kalahari Group 68 (Houben et al., 2020). However, the dating of calcretes using the U-Pb system may be 69 influenced/biased by detrital components from the source area of the leached carbonates. The 70 variable growth rate makes it difficult to obtain individual ages from multiple generations of 71 calcrete formation when using the bulk sampling approach for solution U-Pb dating, as it can be 72 affected by the 'nugget' effect (Branca et al., 2005). Although calcrete formation pre-dates the 73 major canyon incision that can be dated using TCN exposure dating, calcrete formation post-dates 74 sediment deposition and is not age-equivalent to the host sediments. However, the time lag 75 between sediment deposition and calcrete formation may be negligible for any expected age in 76 the range of several millions of years. To avoid contamination by detrital components from the 77 catchment and to date multiple stages of sil-/calcrete formation, syndepositional (with calcrete 78 formation), microscale silcretes produced by the secondary effect of calcrete formation, pressure 79 solution and re-precipitation in the proximity (pressure shadow) might be a valuable target.

80 Evidence for climate change and major landscape change, as well as the reliability of dating of 81 Plio/Pleistocene sediments in the Namib, is relatively poor and not well constrained, and in part 82 shows discrepancies between different dating techniques and interpretations (Miller, 2008; Van 83 Der Wateren and Dunai, 2001; Goudie and Viles, 2014). In Namibia, calcretes and silcretes 84 commonly form prominent landscape features (i.e., cliffs) outcropping along deeply incised ephemeral or fossil drainage systems (Miller, 2008; Van Der Wateren and Dunai, 2001; Ward, 85 86 1987). The (relative) chronology of these fossil duricrusts is the backbone of the (late) Cenozoic chronostratigraphy of the Namib Desert ('Namib Group' of Miller, 2008) and past climate 87 reconstructions (Miller, 2008, and references therein). A major weakness of this 88 89 chronostratigraphy is its absolute chronology: essentially all early Quaternary to mid- Miocene 90 continental deposits in the Namib Desert are 'dated' with ostrich shells (Miller, 2008) or are 91 age-correlated with deposits dated with such shells (Miller, 2008). In general, the ostrich shell 92 biostratigraphy is linked to intracontinental correlations derived from fossil mammals (Pickford 93 and Senut, 2000). The catch is that only the oldest shells are 'dated' to 16-20 Ma (Aepyonithoid, Senut, 2000; Pickford et al., 1999; Pickford et al., 1995), whereas the ensuing eight ostrich species 94 95 are arbitrarily assigned to 2 to 3 Myr long periods (Senut, 2000) without any direct age control. 96 The ostrich shell biostratigraphy provides a valuable relative chronology, but its use in its current 97 form as an absolute chronology remains unverified for the time < 16 Ma. Consequently, the 98 generally accepted Late Cenozoic chronostratigraphy of the Namib Desert (Miller, 2008) requires 99 verification.

100 The use of Terrestrial Cosmogenic Nuclide (TCN) exposure dating in the Namib Desert has grown101 in recent years, demonstrating that this method is a reliable way to measure landscape change

102 (Van Der Wateren and Dunai, 2001; Vermeesch et al., 2010; Stone, 2013; Bierman and Caffee, 103 2001). According to Van Der Wateren and Dunai (2001), major changes in the Namib Desert, i.e. 104 rejuvenation of the landscape by intermittent fluvial phases during the predominant arid to 105 hyperarid climate, indicate major changes during the Plio-/Pleistocene. However, there are 106 doubts about the interpretation of the exposure ages in relation to the underlying deposited 107 sediments (Miller, 2008). The dating of groundwater connected sil- and calcretes beneath the 108 surfaces sampled for TCN exposure dating allows to verify the resulting TCN exposure ages. 109 Furthermore, the combination of both dating techniques can be used to build a robust chronology 110 of landscape change during the evolution and intensification of arid conditions in the Namib 111 Desert.

112 Here we present the new application of U-Pb laser ablation to groundwater silcretes from the 113 Namib Desert in combination with re-measured TCN exposure ages from the Karpfenkliff and 114 nearby equivalent sites. Laser ablation U-Pb dating of multiple microscale silcrete layers from the 115 Karpfenkliff Conglomerate Formation indicates groundwater cal-/silcrete formation during the 116 Pliocene. Re-measured surface clasts from Van Der Wateren and Dunai (2001) confirm and 117 substantiate the interpretation of a major landscape rejuvenation of the Central Namib during the 118 Plio-/Pleistocene transition. The combination of the two dating techniques allows a robust 119 chronological reconstruction of landscape evolution and the paleoclimate transition to 120 increasingly arid conditions in the central Namib Desert.

#### 121

#### 2. Sampling Site and Samples

122 The central Namib Desert, between the Atlantic Ocean to the west and the Great Escarpment to 123 the east, is a relatively flat landscape with numerous dispersed inselbergs and locally deeply 124 incised canyons formed by ephermal rivers such as the Kuiseb or Swakop (Fig. 1). Our study 125 focuses on the Kuiseb River canyon in the central Namib. The ephermal Kuiseb River marks the 126 prominent boundary between the stone desert in the north and the Namib Sand Sea to the south. 127 The Kuiseb River receives its water from precipitation in the Great Escarpment to the east, with 128 mean annual rainfall of 200-450 mm/yr (Ward, 1987; Jacobson et al., 1995). Annual floods of the 129 Kuiseb River clean its bed of all sand transported from the Namib Sand Sea to the south. They only 130 reach the sea during exceptionally high floods (Van Der Wateren and Dunai, 2001). The Kuiseb 131 River forms a distinctive deep and partly narrow canyon, which is up to 250 m deep and only 132 1000 m wide at its deepest part (Fig. 1, 2). The recent course of the Kuiseb River is south-133 southwest to Hudaob, where it is thought to have been redirected north-west by the activity of the 134 Namib Sand Sea (Miller, 2008). Prior to this deflection, the Proto-Kuiseb River may have flowed 135 westwards, as indicated by numerous outcrops within the interdune valleys of the Namib Sand 136 Sea (Fig. 25.18 Vol. 3 in Miller, 2008; Ward, 1987; Lancaster, 1984).



Fig. 1: (A) Overview map of the Central Namib Desert based on World Imagery (Earthstar 138 139 Geographics (TerraColor NextGen) imagery, ArcGIS Pro Version 3.1.0). Major drainage systems are 140 shown in white. Red rectangle indicates the study area. Red squares indicate sampling sites of Van 141 Der Wateren and Dunai (2001). Topographic profiles in Fig. 2 are marked as red lines. (B) Study area 142 (Earthstar Geographics (TerraColor NextGen) imagery, ArcGIS Pro Version3.1.0) including mapped 143 geology by the Geological Survey of Namibia (Geological Survey of Namibia, 2016). Relevant 144 geological formations are shown covering the Cenozoic sediment succession of the Central Namib 145 (Namib Group). Red squares indicate sampling sites from Van Der Wateren and Dunai (2001). The 146 sub-catchment of the Gaub River is shown in white.

### 147 <u>Sediment Succession Kuiseb Canyon</u>

- The outcrop sequence along the Kuiseb canyon in our study area comprises up to 100 m of
   sedimentary units (Fig. 2) resting on the Namib Unconformity Surface of Precambrian age (NUS,
   Ward, 1987; Miller, 2008), consisting of basal breccias from Precambrian basement, and solidified
   aeolian sands assigned to the Tsondab Sandstone Formation, overlain by calcretized
- 152 coarse-grained conglomerates, called the Karpfenkliff Conglomerate Formation (Ward, 1987).
- 153 Well-preserved terraces, resistant to weathering due to calcretization, are exposed at the rim of
- the canyon (Fig. 3).
- 155 The Tsondab Sandstone Formation rests on the Namib Unconformity Surface (NUS, Ward, 1987;
- 156 Miller, 2008), and is the oldest and first terrestrial Cenozoic deposit in the Central Namib (Ward,
- 157 1987; Miller, 2008), covering large areas of the Central Namib (Fig. 1, 2). The Tsondab Sandstone
- 158 Formation consists predominantly of cemented aeolianites (Miller, 2008; Ward, 1987) and is
- regarded as the precursor of the recent Namib Sand Sea (Ollier, 1977). The Tsondab Sandstone
- 160 Formation is thought to have been deposited under predominantly arid conditions (Ward, 1987),

between 20-16 Ma and 5 Ma based on the biostratigraphy of Struthious eggshells (Namoris
Oshanai, Struthio Karinagarabensis, Ward and Corbett, 1990; Pickford et al., 1995; Senut, 2000).



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Fig. 2: (A) Fig. 2: (A)Cross-sections of the Karpfenkliff and (B) Kamberg Cliff based on SRTM data 164 165 (created using ArcGIS Pro 3.1.0). Spatial information on geological units was extrapolated from 166 mapped geology (Geological Survey of Namibia, 2016). Samples were collected from the surface of 167 the Karpfenkliff (DWA980006,07) and from the subsurface at the canyon outcrop (DWA980008, -168 21). The identical sampling approach was used on the Kamberg Cliff, by sampling exposed quartz 169 clasts (DWA98013) and shielded clasts (DWA98014). The shielded quartz clasts were used to 170 investigate and date secondary micro-scale silcrete layers attached to quartz clasts. Due to the 171 unknown fluvial topography of the Proto-Kuiseb, the profile is just an approximation. The occurrence 172 of the Karpfenkliff Conglomerate Formation (TKk) is used from the geological map, however, its 173 outcrop condition in profile A in the eastern sector remains speculative. The exact transition from 174 the underlying Tsondab Sandstone (TTs) to the Karpfenkliff Conglomerate Formation is unclear and 175 is approximated. The elevation of the Kamberg Calcrete from its key position is marked in B and to 176 illustrate the potential discrepancy between the two formations.

177 Proto-Kuiseb Incision and aggradation of the Karpfenkliff Conglomerate Formation

The Karpfenkliff Conglomerate Formation (TKk, Ward, 1987) overlies the Tsondab Sandstone Formation and was deposited in a proto-Kuiseb and proto-Gaub River valley, a tributary of the Kuiseb River (Fig. 1, 2). Pre-depositional incision of the Kuiseb and Gaub rivers probably occurred during a wetter phase (Ward, 1987; Miller, 2008). The incision excavated a broad shallow valley and eroded the semi-consolidated Tsondab Sandstone without eroding the underlying Pre-Cambrian Damaran schists (Ward, 1987; Miller, 2008). The Karpfenkliff Conglomerate Formation consists of a medium- to fine-grained, sand-sized matrix of angular to subrounded 185 clasts (Fig. 3, Ward, 1987; Miller, 2008). Clasts are rounded to well-rounded with numerous 186 percussion marks (Ward, 1987; Van Der Wateren and Dunai, 2001). The Karpfenkliff 187 Conglomerate Formation thins to the west, indicating a depositional wedge (Miller, 2008). The 188 thickest accumulations are found at the foothills of the Great Escarpment ( $\sim 60$  m, Miller, 2008), 189 decreasing to  $\sim$  40 m (Ward, 1987) in the upper Gaub Valley, 20-30 m at the Karpfenkliff, and 190 thinning to ~5 m at Gomkaeb (Ward, 1987; Miller, 2008). Deposition took place in a wide, shallow, 191 braided river system (Ward, 1987; Miller, 2008), presumably during an intermittent pluvial phase 192 despite prevailing arid conditions and synchronous with the deposition of the Tsondab aeolianites 193 (Ward, 1987). Equivalent gravels in the Tsondab, Tsauchab and Swakop rivers are assigned to the 194 Karpfenkliff Conglomerate Formation (Miller, 2008). The conglomerate is cemented by a massive 195 groundwater calcrete that has caused significant volume expansion (Miller, 2008). The source 196 area of the carbonate ions is thought to be the outcropping and eroding Precambrian Nama Group 197 in the headwaters of the Kuiseb River and is therefore authogenic in origin. Calcretization caused 198 secondary precipitation of microscale silcrete by pressure solution and local re-precipitation.

199 The Karpfenkliff Conglomerate Formation is age-correlated with the occurrence of Diamantornis 200 corbetti (fossil ostrich shell) in the Tsondab Aeolianites at Elim (Pickford and Senut, 2000; Miller, 201 2008), implying a younger age of *Diamantornis corbetti* than 14-15 Ma, equivalent to the age of 202 the Arries Drift Formation (Miller, 2008). Youngest deposition age ( prior to  $2.81 \pm 0.11$  Ma) was 203 proposed by Van Der Wateren and Dunai (2001) based on <sup>21</sup>Ne exposure dating of abandoned 204 surfaces of the Kuiseb River. The latter indicates the minimum depositional age for the last 205 remnants of any fluvial transport and deposition of the Karpfenkliff Conglomerate Formation. 206 Although this age is controversial according to Miller (2008, page 25-27) based on the ostrich shell 207 biostratigraphy, it clearly indicates the onset of incision by the recent Kuiseb River.

#### 208 <u>Calcrete within Karpfenkliff Formation and Tsondab Sandstone - Kamberg calcrete formation</u>

209 The Kamberg Calcrete is described as a pedogenic calcrete up to 5 m in thickness (Miller, 2008; 210 Ward, 1987; Yaalon and Ward, 1982). According to Miller (2008), it cements the upper 211 Karpfenkliff Conglomerate Formation in places, as well as the Tsondab Sandstone, which covers a 212 large area east of Homeb in the Kuiseb River (Miller, 2008; Ward, 1987; Yaalon and Ward, 1982). 213 The Kamberg Calcrete, as well as equivalent calcretes in the study area, represent the surface 214 predating the recent canyon incision of the Kuiseb and Gaub rivers. They are used as an important 215 stratigraphic marker horizon in the Cenozoic 'Namib Group' (Miller, 2008; Ward, 1987). Whether 216 the Kamberg Calcrete is identical to the calcrete of the Karpfenkliff can be questioned (Fig. 2). The 217 pedogenic Kamberg Calcrete may be transitional to the groundwater calcrete found at the 218 Karpfenkliff and therefore be syndepositional. If the Kamberg Calcrete at the key site at Kamberg 219 correlates with the Kamberg Cliff and the Carp Cliff at Kuiseb canyon, this would imply that it is

stratigraphically equivalent to or younger than the groundwater calcrete cementing the Karpfenkliff Conglomerate Formation (Fig. 2). A late Miocene age has been suggested for the evolution of the Kamberg Calcrete (Yaalon and Ward, 1982; Ward, 1987). The calcrete is thought to have been formed under semi-arid conditions during a relatively long period of landscape stability (Goudie et al., 2015; Ward, 1987), with seasonal precipitation of potentially 350-450 mm

in the headwaters, decreasing drastically to the west (Ward, 1987).

A clear differentiation between the Kamberg Calcrete and any calcretes overlying and/or within the Karpfenkliff Conglomerate Formation is difficult. The Kamberg Calcrete is not specifically mapped in the published geological maps (Geological Survey of Namibia, 2016). For our study, we focused on near-surface clasts with silcrete at the Karpfenkliff. The clear spatial and evolutionary differentiation, as well as the connection between the two, should be the focus of future research to use their occurrence as a marker horizon in the Central Namib.

232 Kuiseb Incision – Phase of landscape rejuvenation

233 The incision of the Kuiseb River (and other adjacent rivers such as the Swakop to the north) is 234 thought to have begun at the end of the Neogene, synchronous with other major river systems in 235 South Africa (Ward, 1987; King, 1951; Partridge and Maud, 1987; Korn and Martin, 1957). The 236 recent incision was able to cut deeply into the Karpfenkliff Conglomerate Formation, the Tsondab 237 Sandstone Formation and also into the Pre-Cambrian Damaran schists (Miller, 2008; Van Der 238 Wateren and Dunai, 2001; Ward, 1987), forming a V-shaped valley and the famous Kuiseb canyon 239 (Fig. 3). The transition from the aggradation of the Karpfenkliff Conglomerate Formation and the 240 formation of calcretes, to the degradation and incision of the recent Kuiseb, Gaub and Swakop 241 rivers is thought to be related to either a tectonic- (King, 1955; Ward, 1987; Korn and Martin, 242 1957) or climatic control (Van Der Wateren and Dunai, 2001; Richards and Richards, 1987; 243 Weissel and Seidl, 1998).

## 244 Detailed sampling sites and sampling

245 We consider that the calcrete at our sampling sites (Karpfenkliff, and Kamberg Cliff, Fig. 1, 2, 3) 246 was formed primarily by groundwater interaction, due to its direct location near the present-day 247 Kuiseb canyon. We used sampled and dated (in-situ <sup>21</sup>Ne) surface quartz clasts from Van Der 248 Wateren and Dunai (2001), from abandoned exposed surfaces and shielded clasts from several 249 metres below the surface. Details of the sampling procedure and sampling sites are given in Van 250 Der Wateren and Dunai (2001). For this study we concentrated on surface quartz clasts from the 251 Carp Cliff (DWA98006, -07, -08, -21) and the Kamberg Cliff (DWA98013, -14) for re-measurement 252 of cosmogenic <sup>21</sup>Ne concentrations (Table 1). Eight quartz clasts from the Carp Cliff with visible silcrete cementation were prepared for U-Pb laser ablation (DWA98008, Table 1). The following 253

- descriptions are taken from Van Der Wateren and Dunai (2001) and partly adapted for additional
- 255 samples.



Fig. 3: Outcrop image compilation. (A) From Van Der Wateren and Dunai (2001) Kamberg Cliff, ~15 m of
Karpfenkliff Conglomerates overlying ~15 m of Tsondab Sandstones. (B) Carp Cliff (Field Campaign 2018,
Photo B. Ritter). (C) Close-up of the calcrete-cemented Karpfenkliff Conglomerate Formation (Photo B. Ritter).
Rounded quartz clasts float in a matrix-supported fabric which is cemented by calcrete. Some clasts are
fractured by volume expansion of the calcrete, resulting in pressure solution and formation of micro-scale
silcrete. (D) Surface of the calcrete cemented Karpfenkliff (Photo B. Ritter).

- 263 <u>Carp Cliff (Kuiseb highest terrace)</u>
- 264 DWA98006-07 (Site 6) is located on the horizontal upper surface of a mesa-shaped terrace 265 remnant 500 m west of the 200 m deep Kuiseb Canyon (Fig. 1, 2). The terrace has a surface area 266 of ~5 km<sup>2</sup> and is surrounded by steep, locally vertical and overhanging cliffs, which to the east and 267 south are nearly 50 m in height. The terrace surface consists of a desert pavement of mainly quartz 268 pebbles overlying up to 15 cm of sandy silt. This is underlain by 10–25 m of calcretized pebble 269 and boulder conglomerates of the Karpfenkliff Conglomerate Formation (Fig. 3). Van Der Wateren 270 and Dunai (2001) collected 40 rounded (DWA98007) and (sub-)angular pebbles (DWA98006) 271 with diameters between 2 and 6 cm. The site is located at the top of the mesa and is almost 272 horizontal, so that post depositional transport of the sampled pebbles by the Kuiseb River or local 273 precipitation can be excluded.

- DWA98008 and DWA980021 (Site 7 and 19) are located next to small gullies running from the
- 275 north side of the Carp Cliff mesa. At these sites, Van Der Wateren and Dunai (2001) collected
- shielded samples 5 m below the terrace surface. Van Der Wateren and Dunai (2001) sampled
- 277 **rounded pebbles from the ceilings of overhangs** to ensure that the measured <sup>21</sup>Ne concentrations
- 278 were derived only during hillslope and fluvial transport to their present site and not from
- 279 subsequent exposure at the sampling site.

# 280 <u>Kamberg Cliff (Kuiseb highest terrace)</u>

281 DWA98013 and DWA980014 are from a terrace on the Karpfenkliff Conglomerate Formation 282 immediately adjacent to the nearly 250 m deep Kuiseb Canyon, 30 km downstream of Carp Cliff (Fig. 1, 2, 3). The terrace surface is very similar to that of Carp Cliff, with a desert pavement of 283 284 pebbles and cobbles on a sandy silt overlying 25 m of calcretized conglomerates. The Karpfenkliff 285 Conglomerates rest on 30–50 m of the Tsondab Sandstone Formation, which forms the bulk of the 286 cliff adjacent to the canyon. DWA98013 sampling site is on the horizontal surface of the terrace, 287 where we sampled angular pebbles. At DWA98014, rounded pebbles (DWA98014) were sampled 288 from the ceiling of an overhang in the cliff face 6 m below.

# 289 **Table 1: General sample information.**

i un exposur e Duung										
Sample ID	Locality	Туре	Longitude [°]	Latitude [°]	Remark					
DWA98006Etch2	Carp Cliff	Quartz clasts	-23.339	15.744	amalgamated sample of 40 clasts					
DWA98007Etch	Carp Cliff	Quartz clasts	-23.339	15.744	amalgamated sample of 40 clasts					
DWA98008Etch2	Carp Cliff	Quartz clasts	-23.331	15.746	amalgamated sample of 40 clasts					
DWA98021Etch	Carp Cliff	Quartz clasts	-23.332	15.745	amalgamated sample of 40 clasts					
DWA98013Etch2	Kamberg Cliff	Quartz clasts	-23.608	15.580	amalgamated sample of 40 clasts					
DWA98014Etch	Kamberg Cliff	Quartz clasts	-23.608	15.580	amalgamated sample of 40 clasts					

## TCN Exposure Dating

### U-Pb LA-ICP-MS Dating

Sample ID	Locality	Туре	Longitude	Latitude	Remark
DWA98008- Silc3	Carp Cliff	Silcrete on clast	-23.331	15.746	Silcrete with distinct layering
DWA98008- Silc4	Carp Cliff	Silcrete on clast	-23.331	15.746	Silcrete with distinct layering
DWA98008- Silc7	Carp Cliff	Silcrete on clast	-23.331	15.746	Silcrete with distinct layering
DWA98008- Silc8	Carp Cliff	Silcrete on clast	-23.331	15.746	Silcrete with distinct layering

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### 3. Formation of Calcretes and microscale silcretes

294 In general, two types of calcretes can be differentiated, pedogenic and groundwater calcretes 295 (Alonso-Zarza and Wright, 2010), following the per descensum or the per ascensum evolutionary 296 model (Goudie, 2020). They occur preferentially in arid to semi-arid climates (Alonso-Zarza, 297 2003; Candy and Black, 2009; Goudie, 2020). Specific climatic and environmental conditions are 298 required for calcrete formation; (1) precipitation in the headwater/source area to promote 299 carbonate dissolution, (2) intermittent or seasonal precipitation downstream favour 300 groundwater systems capable of (3) causing high evaporation and evapotranspiration for 301 chemical precipitation of carbonate (Mann and Horwitz, 1979). Calcrete formation is dependent 302 on the supply of carbonate ions leached from the drainage bedrock. In this study we focus on 303 groundwater calcretes formed along the Kuiseb- and Gaub rivers. Groundwater calcretes form at 304 or above shallow groundwater tables/aquifers (Mann and Horwitz, 1979; Netterberg, 1969) and 305 do not require subaerial exposure, although shallow contacts and stable surfaces favour the 306 evolution of groundwater calcretes (Alonso-Zarza, 2003). They were originally called 'valley 307 calcretes' (Butt et al., 1977) because of their relationship with drainages. Groundwater calcretes 308 are rather restricted to local drainages, although groundwater calcretes can have lateral extents 309 of more than 100 km long and 10 km wide, depending on the drainage topography (Mann and 310 Horwitz, 1979). Groundwater calcretes do not have characteristic features compared to 311 pedogenic calcretes and are rather massive bodies (Alonso-Zarza, 2003). The permeability 312 (coarse channel sediments) of the host rock favours their formation (Alonso-Zarza and Wright, 313 2010). Calcretes have been frequently used to obtain paleo precipitation information, but the 314 specific ranges are still under discussion. The upper limit may be between 600 and 1000 mm/yr 315 (Mack and James, 1994). The lower limit may be as low as 50 mm/yr (Goudie, 1973; Retallack, 316 1994).

317 Silcretes can form as duricrust due to the accumulation of secondary silica within a soil or host 318 rock (Milnes and Thiry, 1992; Summerfield, 1983a). Prominent examples include silcretes from 319 Australia (Milnes et al., 1991; Taylor and Eggleton, 2017) or the Kalahari Desert (Summerfield, 320 1983b; Nash and Shaw, 1998). In this study, we focus on microscale silcretes, which are formed 321 by pressure solution (Mcbride, 1989; Rutter, 1983; Sorby, 1863; Wilson, 2020) due to calcrete 322 cementation and volume expansion within the host rock or sediment, and therefore cannot be 323 directly compared to the commonly used term 'silcrete'. Additional silica may be enriched in the 324 groundwater due to increased pH (favours the precipitation of calcite and the solution of silica, 325 Goudie, 1983; Nash and Shaw, 1998). Microscale silcrete formation is therefore thought to be 326 linked to paleo-environmental and climatic conditions favourable to calcrete formation. 327 Calcretization involves the precipitation of CaCO<sub>3</sub> within the pore spaces of the host rock or 328 sediment, causing significant volume expansion. A secondary effect of this process is to increase the differential pressure within the host rock or sediment, causing clast shattering, relocation and pressure solution at intergranular contacts (Sorby, 1863; Rutter, 1983). Increased stress at grain boundaries and intergranular contacts leads to dissolution, e.g., silica mobilisation. Mobilized solutions migrate to regions of lower compressive stress, the 'pressure shadow', to reprecipitate. Theoretically, depending on the remaining pore space, multiple pressure solution and reprecipitation cycles can be archived in the host rock as multiple silcrete layers or shells attached to quartz clasts.

336

# 4. Dating of Cal- and Silcretes

337 Quantifying the timing and duration of calcrete formation is quite difficult. Clear stratigraphic 338 relationships with the overlying and underlying sediments are not straightforward, as groundwater calcrete, for example, forms within sediments deposited close to the surface. 339 340 Numerous studies propose only relative age controls and estimates of the formation time, such as the application of the ostrich shell biochronostratigraphy used for the Namib Group (Pickford and 341 342 Senut, 2000; Senut, 2000; Miller, 2008). Many attempts have been made to date this type of 343 deposits using radiocarbon <sup>14</sup>C (e.g. Geyh and Eitel, 1997), U/Th (Kelly et al., 2000; Candy et al., 344 2004; Candy and Black, 2009) or U-Pb dating (Rasbury and Cole, 2009; Houben et al., 2020).

345 Silcretes are enriched in U relative to calcretes and occur in most soils in arid and semi-arid 346 environments. Uranium decays to Pb isotopes through a chain of intermediate daughter isotopes, 347 and ages of thousands- to millions-of-years-old samples can be estimated using parent-daughter pairs <sup>238</sup>U-<sup>206</sup>Pb, <sup>235</sup>U-<sup>207</sup>Pb, <sup>234</sup>U-<sup>230</sup>Th, and <sup>238</sup>U-<sup>234</sup>U. The use of a particular isotope pair depends 348 349 on how old the sample is compared to the half-life of the selected radioactive isotope within the U 350 decay chain (Neymark, 2011; Neymark et al., 2002, 2000). Considering that the samples are 2.85 Ma old or older (Van Der Wateren and Dunai, 2001), the U-Pb method using the parent-daughter 351 pairs <sup>238</sup>U-<sup>206</sup>Pb and <sup>235</sup>U-<sup>207</sup>Pb was chosen to date the samples in this work. 352 353 Many studies attempting to date massive cal-/silcretes are hampered by the dilution or averaging

effect of bulk analysis and by bias from non-carbonate detrital minerals or secondary
reprecipitated carbonate due to diagenesis. The "limestone dilution effect" (as a result of
contamination with detrital carbonate components of the host rock, Alonso-Zarza, 2003) or the
"averaging effect" (averaging of different phases of mineral precipitation, Candy and Black, 2009;
Neymark et al., 2000) are minimised (or even avoided) by the higher spatial resolution of laser
ablation compared to bulk analysis techniques. The possible effect of detrital components (e.g.

360 Zircon or clay minerals) on the U-Pb analyses is also neglected, as the signals from these inclusions

361 **can be filtered out of the time-resolved analyses.** 

362	The conventional method of calculating U-Pb isotope dates assumes that all intermediate
363	daughter isotopes in the $^{238}$ U and $^{235}$ U decay chains were in secular equilibrium at the time of
364	formation (Neymark, 2014). This is not necessarily true for calcretes and silcretes due to
365	differences in the geochemical behaviour of parent and daughter elements. The silcretes dated in
366	this study are sufficiently old (> c. 2.85 Ma) to have achieved secular equilibrium (at present), and
367	therefore (almost) all its initial excess of daughter isotopes to decay, or their initial depletion to
368	replenish (i.e. their activity ratios to be equal to 1) ergo a direct measurement of these deviations
369	is not feasible. Therefore, the values needed to correct for these disequilibriums were estimated
370	from previous works (see methodology chapter).

- 371
- 372 **5. Methods**
- 373 <u>Raman Spectroscopy</u>

374 We use Raman spectroscopy to obtain high resolution images of silcretes and to better 375 characterise the mineralogical composition. Raman spectra were collected with up to 1300 376 wavenumber (cm<sup>-1</sup>), using a WITec alpha 300R confocal Micro-Raman microscope, at the Goethe 377 University Frankfurt (GUF). The objective used was 50x, an excitation laser of 532 nm (using 10 378 mW laser power before the objective), and spectra integration time of 0.2 s with 5 accumulations 379 in total. Maps (400 x 400 μm<sup>2</sup>) were performed applying a step size of 1.3 μm with a holographic 380 grating of 600 grooves mm<sup>-1</sup>. The instrument was calibrated using an Ar-Hg spectral lamp and was 381 checked regarding its performance before the measurements with respect to the 1300 cm<sup>-1</sup> line 382 of silicon. The spectrum of each sample layer was confirmed at several locations on the same layer. 383 Raman spectra of reference compounds are found in the Rruff database (https://rruff.info/).

384 Dating of Silcretes – U-Pb Laser Ablation ICP-MS

385 Eight quartz clasts were cut in half to expose their silcrete coatings, mounted in epoxy mounts and 386 polished at the Department of Geosciences, University of Cologne (UoC). U-Pb analyses were 387 performed at the Goethe University Frankfurt (GUF) using a RESOlution 193 nm ArF excimer laser 388 (COMpex Pro 102), equipped with a two-volume ablation cell (Laurin Technic S155). The laser 389 was coupled to a ThermoScietific ElementXr sector field ICP-MS. The surfaces were cleaned with 390 8 pre-ablation laser pulses. Ablation was carried out in a He (0.3 l/min), Ar (1.01 l/min) and N 391 (0.012 l/min) atmosphere, with a high energy density (c. 5 J/cm<sup>2</sup>), a frequency of 15 Hz and round 392 50 μm diameter spots (SI2\_Supporting Information).

Artificial silicate glasses NIST SRTM 612 and 614 were used as reference materials (RM). Plots and dates are calculated using the in- house spreadsheet program (Gerdes and Zeh, 2009, 2006), 395 together with Isoplot (Ludwig, 2012). Ages are reported with and without systematic components 396 (i.e., date  $\pm 2s / 2s_{sys}$ ). Uncertainties include internal standard errors (SE), background, counting 397 statistics, excess scatter of the primary reference material (NIST SRTM 612), and excess variance 398 (calculated from NIST SRTM 614). Systematic uncertainties also propagate systematic errors, 399 which are the long-term excess variance (1.5%, 2s), decay constant uncertainties (Horstwood et 400 al., 2016) and the uncertainty derived from the initial activity ratio uncertainty. Dates are 401 calculated as Tera-Wasserburg lower intercepts (Tera and Wasserburg, 1972). Linear regressions 402 are anchored to a common-lead <sup>207</sup>Pb/<sup>206</sup>Pb ratio of 0.837. This is the Y-intercept of sample 403 "DWA98008-Silc4 Black Crack", which is where this ratio is better constrained. This value is in 404 good agreement with modelled crustal values at the time of formation (0.836, Stacey and Kramers, 405 1975).

406 The samples dated are sufficiently old to have reached secular equilibrium and hence activity 407 ratios cannot be measured (with the present techniques and assuming a closed system 408 behaviour). Consequently, the following initial activity ratios used are assumed. The silcretes 409 dated in this study have virtually no Th (average of  $\sim 89 \text{ ng/g}$ ) and therefore we consider  $[^{230}\text{Th}/^{238}\text{U}]_{i} = 0$  (initial  $^{230}\text{Th}/^{238}\text{U}$  activity ratio). Considering previous studies on calcretes and 410 411 silcretes formed in semi-arid and arid environments (Oster et al., 2017; Maher et al., 2007; 412 Neymark, 2011), the ground and surface waters from which these rocks are formed often have 413  $[^{234}\text{U}/^{238}\text{U}]_{i}$  values greater than 1. Therefore, the data in this study are calculated with  $[^{234}\text{U}/^{238}\text{U}]$ 414  $_{i}$  = 1.75 ± 0.32 (2s abs), which is a weighted average of the [<sup>234</sup>U/<sup>238</sup>U] i of the above-mentioned 415 studies. The uncertainty in this activity ratio is added to the final systematic uncertainties by 416 quadratic propagation (Scardia et al., 2019; SI2\_Supporting Information).

### 417 <u>Cosmogenic <sup>21</sup>Ne Exposure Dating</u>

418 We used prepared samples from Van Der Wateren and Dunai (2001) for in situ <sup>21</sup>Ne exposure 419 dating using the new noble gas mass spectrometer at the University of Cologne (Helix MC Plus 420 from Thermo Fisher Scientific, further information see Ritter et al., 2021). The <sup>21</sup>Ne analyses of 421 Van Der Wateren and Dunai (2001) were performed without an international standard (CREU). 422 For neon analysis we prepared amalgamated samples from each site containing between 35 and 423 40 quartz clasts (100mg/sample) using the already prepared 63-125 µm fraction. By also 424 analysing shielded pebbles, a pre-exposure correction (accumulated <sup>21</sup>Ne concentration during 425 transport) can be applied to analysed surface samples (Repka et al., 1997). The presumably 426 non-atmospheric <sup>21</sup>Ne concentration found in these samples can be subtracted from the 427 concentration in their exposed counterparts. The latter also corrects for any potential nucleogenic 428 <sup>21</sup>Ne that may be present in the samples. Samples were measured on the noble gas mass 429 spectrometer at the University of Cologne using the analytical methods outlined in Ritter et al.

(2021). CREU quartz standards were measured in Cologne for interlaboratory comparability and
quality control (Vermeesch et al., 2015). The spallogenic origin of the measured <sup>21</sup>Ne excess was
verified using the triple isotope plot. <sup>21</sup>Ne exposure ages were calculated using the 'nuclide
dependent scaling' after Lifton et al. (2014), calculated with "The online exposure age calculator
formerly known as the CRONUS-Earth online exposure age calculator." (Version 3,
http://hess.ess.washington.edu/math/v3/v3\_age\_in.html; Balco et al., 2008).

### 436 **6. Results**

#### 437 <u>Silcrete Imaging</u>

Digital microscope images show vein-contact parallel-layering with different crystal orientation (Fig. 4A and Supporting Information). The Raman spectra peaks at 129, 209 and 467 cm<sup>-1</sup> are indicative for quartz, which dominate the silcrete samples. Raman spectroscopy of DWA98008-Silc8 also indicate the presence of one major calcite band (dark colour in Fig. 5B, Raman spectra peaks at 156, 283, 466, 714 and 1088 cm<sup>-1</sup>). The calcite crack filling might be indicative for shattering of previous silcrete and crack filling by repeated and/or ongoing calcrete formation within the Karpfenkliff Conglomerate.

### 445 <u>U-Pb Laser Ablation Results Silcretes</u>

Four out of the eight silcrete samples yielded meaningful dates (DWA98008 – Silc3, Silc4, Silc7, Silc8), out of which 12 dates could be calculated from various silcrete layers (SI2\_Supporting Information). The dates range from 2.96  $\pm$  0.14 to 6.72  $\pm$  0.16 Ma, with maximum relative abundance peaks at around 3.4 Ma and about 5.5 Ma (see Fig. 6 and Table 2). All dates are calculated from multiple spot analyses, ranging from 9 to 28 spots per date. The majority of the analyses have U concentrations between ~30 and 70 µg/g, with an average of 42 µg/g, and very low Th concentrations, up to 200 ng/g, with an average of 89 ng/g.

Sample	Date (Ma)	2s abs	2s sys	Y- Interce	2s (5)	Anchored	MSWD	n (8)	n (9)	Date (Ma)	2s abs	2s sys
name	(1)	(2)	(3)	pt <sub>(4)</sub>		(6)	0	(used)	(total)	(10)	(11)	(12)
Silc3 Rim 1	5.756	0.124	0.132	0.837	0.008	WAHR	0.45	9	9	5.590	0.120	0.176
Silc3 Rim 2	5.246	0.107	0.115	0.837	0.008	WAHR	0.41	9	9	5.080	0.103	0.164
Silc3 Rim 3	5.488	0.068	0.081	0.837	0.008	WAHR	1.35	24	28	5.322	0.066	0.143
Silc3 Rim Outer	3.734	0.064	0.071	0.838	0.011	WAHR	1.63	28	28	3.569	0.061	0.138
Silc4 Black crack	5.927	0.244	0.248	0.8374	0.0066	FALSCH	1.50	21	25	5.761	0.237	0.270
Silc4 Rim	3.498	0.061	0.067	0.837	0.008	WAHR	1.24	16	17	3.332	0.058	0.136
Silc4 Rim Outer	3.456	0.061	0.067	0.837	0.008	WAHR	1.30	15	17	3.290	0.058	0.136
Silc4 Rim Inner	3.129	0.068	0.072	0.837	0.008	WAHR	1.63	9	9	2.964	0.064	0.138
Silc7	6.881	0.091	0.106	0.8528	0.0028	FALSCH	0.94	27	29	6.715	0.089	0.159
Silc8 Rim Out 1	6.116	0.081	0.095	0.837	0.008	WAHR	0.64	20	20	5.950	0.079	0.152
Silc8 Rim Out 2	5.131	0.132	0.139	0.837	0.014	WAHR	2.56	17	25	4.965	0.128	0.180

Silc8 Rim Center 3.847 0.131 0.135 0.837 0.008 WAHR 1.37 20 20 3.681 0.126 0.176													
	Silc8 Rim Center	3.847	0.131	0.135	0.837	0.008	WAHR	1.37	20	20	3.681	0.126	0.176

453

454 Table 2: 1. - Concordia curve lower intercept dates, Tera-Wasserburg diagram (Tera and 455 Wasserburg, 1972). 2. - 2s absolute uncertainties, considering within run precision (SE of the mean of ratios), excess of scatter, background, counting statistics and excess of variance (calculated from 456 the validating RM, SRTM NIST 614. 2.6%, 1s, on <sup>238</sup>U/<sup>206</sup>Pb and 0%, 1s, on <sup>207</sup>Pb/<sup>206</sup>Pb ratios). 3. -457 458 Previous uncertainties (2) expanded with systematic uncertainties (0.8 %, 2s, long term 459 reproducibility and decay constant uncertainties). See Horstwood et al. (2016). 4. - 207 Pb/206 Pb ratio 460 of the upper intercept. 5. - 2s absolute uncertainty of the upper intercept. 6. - If the linear regression on the Tera-Wasserburg was anchored or not. 7. - Mean squared weighted deviates. 8. - Number of 461 462 analyses considered. 9. - Total number of analyses. 10. - Dates calculated (following Wendt and Carl, 463 1985) taking into account an initial <sup>234</sup>U/<sup>238</sup>U activity ratio of 1.75, and initial <sup>230</sup>Th/<sup>238</sup>U activity ratios of 0. 11. - Uncertainties (2) recalculated to the new dates (10). 12. - Uncertainties (3) 464 recalculated to the new dates (10) and adding  $^{234}U/^{238}U$  activity ratio uncertainty (0.32, 2s abs). 465





469 Fig. 4: (A) Microscope image of sample DWA98008-Silc8 after LA-ICP-MS analysis. Laser spots are 470 visible, respective U-Pb Tera-Wasserburg plots are shown in (D-F). (B) Raman image of Silc8, area 471 not visible on microscope image. Grey crosses indicate measured quartz spectra. White cross marked 472 the occurrence of Calcite, which can be traced as a black line. Raman imaging shows multiple 473 layering of silcrete filling the crack of the shattered quartz clast. Color variations are indicative of 474 differences in crystal orientations (C) Respective Raman spectra for the quartz and calcite 475 identification. (D-F) Tera-Wasserburg plots of Silc8 and respective U-Pb ages (red ellipses are 476 considered outliers).

## 477 <u>TCN Exposure Age Results</u>

478 <sup>21</sup>Ne samples measured at the Noble Gas Laboratory in Cologne (Ritter et al., 2021) yield 479 concentrations of 1.52 - 1.95 x 107 atoms/gr for shielded samples (DWA98008, 014, 021) and 480  $6.30 - 9.60 \ge 10^7$  atoms/gr for surface samples (DWA98006,007, 013). All samples are within 2 481 sigma of the spallation line (Fig. 5). Compared to the <sup>21</sup>Ne concentrations of Van Der Wateren and 482 Dunai (2001), the etched samples measured in Cologne reveal lower concentrations of up to 13% 483 difference when comparing direct concentrations (average of five measured samples), however, within  $\pm 1\sigma$  it reduces to ~1.6%, agreeing within  $\pm 2\sigma$  on average. We have excluded sample 484 485 DWA98008, as it presumably contains a high abundance of non-cosmogenic Ne, as deduced from 486 the significant concentration differences between the sample measured by Van Der Wateren and 487 Dunai (2001) and the etched counterpart measured in Cologne (SI1\_Supporting Information). 488 Similar results and interpretations for sample DWA98008 were reported by Van Der Wateren and 489 Dunai (2001).

- 490 Using the mean difference of ~13% between VU Amsterdam and Cologne Ne concentrations
- 491 (SI1\_Supporting Information), the data from Van Der Wateren and Dunai (2001) can be corrected
- 492 for lab-specific differences. The corrected exposure ages are given in Table <mark>3</mark>.



- Fig. 5: Triple isotope diagram indicating single-heat-step extraction of the Cologne laboratory
  (orange circles) compared to the multiple-heat-step extraction (red circles) of Van Der Wateren and
  Dunai (2001). Uncertainties are 1o. The red stippled line indicates the Cologne laboratory spallation
  line (Ritter et al., 2021). Green circles indicate CREU1 measured during the analysis in Cologne.
- 498 Calculated exposure ages derived from Cronus Earth (Balco et al., 2008) are summarised in 499 Table **3**. For the Kuiseb terrace, <sup>21</sup>Ne concentrations in shielded, pre-exposed samples 500 (DWA98008, DWA98021), give a mean apparent age of  $0.65 \pm 0.04$  Ma (external uncertainty  $\pm 1\sigma$ ). 501 The latter indicates that the non-cosmogenic component of DWA98008 has been removed by 502 etching, indicating the identical apparent exposure age as DWA98021. Correction of the <sup>21</sup>Ne 503 concentration of exposed rounded pebbles (DWA98007) from the top of the Carp Cliff terrace 504 yields an exposure age of  $3.2 \pm 0.2$  Ma ( $\pm 1\sigma$  external uncertainty), being slightly older than 505 calculated by Van Der Wateren and Dunai (2001), however, identical within the uncertainty. 506 Exposed angular clasts (DWA98006) show a younger exposure age of 2.85  $\pm$ 0.19 Ma ( $\pm$ 1 $\sigma$  external 507 uncertainty). The latter is slightly older than in Van Der Wateren and Dunai (2001), which is 508 identical within their uncertainty. A similar exposure age of 2.75  $\pm$  0.18 Ma ( $\pm 1\sigma$  external 509 uncertainty) was derived from angular clasts from the Kamberg cliff (DWA98013) with. Angular 510 clasts are assumed to be derived from local sources without significant pre-exposure from long 511 transport times. Our results indicate that terrace abandonment and exposure to cosmic rays 512 started at  $\sim$ 2.8 Ma (Fig. 6).

		LSDn Exposure Age	
	Age [Ma]	Int. Unc. [Ma]	Ext. Unc. [Ma
DWA98006Etch2	2.85	0.07	0.19
DWA98007Etch	3.85	0.07	0.25
Corr. DWA98007Etch2	3.20	0.05	0.20
DWA98008Etch2	0.66	0.02	0.05
DWA98021Etch	0.65	0.02	0.04
DWA98013Etch2	2.75	0.05	0.18
DWA98014Etch	0.90	0.02	0.06
DWA98001VU	5.35	0.23	0.41
DWA98002VU	3.98	0.32	0.40
DWA98003VU	1.08	0.13	0.15
DWA98005VU	0.58	0.06	0.07
DWA98019VU	0.49	0.05	0.06
DWA98024VU	1.27	0.12	0.14

513 Table **3**: TCN exposure ages.

#### 514 **7. Interpretation and Discussion**

515 Our U-Pb ages are stratigraphically in the correct order, with the oldest U-Pb ages at the contact 516 between quartz clast and filled rock fracture and the youngest age in the centre of the filled 517 fracture (Table 2, Fig. 4). Recurrent U-Pb ages underpin and mark the main phase of silcrete, i.e. 518 calcrete, formation. Groundwater calcrete formation, i.e., microscale silcrete formation, within the 519 sediments of the proto-Kuiseb canyon (Karpfenkliff Conglomerate) took place between the Late 520 Miocene (~7 Ma) and the Late Pliocene (~3 Ma, Fig. 6). The U-Pb silcrete ages suggest either 521 persistent or alternating periods of wetter climate for groundwater calcrete formation.

- 522 Based on the causal relationship between silcrete and calcrete formation, our U-Pb silcrete ages 523 indicate that environmental and climatic conditions during the Pliocene were sufficient to allow 524 for carbonate leaching, transport and calcrete formation within the coarse-grained Karpfenkliff 525 Conglomerate. However, whether the sampled groundwater calcrete is identical or synchronous 526 with the prominent Kamberg Calcrete can be questioned, but we can narrow down the timing of 527 major groundwater calcrete formation, previously assigned to the Late Miocene (Goudie et al., 528 2015; Ward, 1987) or Plio-Pleistocene (Pickford and Senut, 2000). Calcrete formation ceased 529 during the Late Pliocene/Early Pleistocene by incision and groundwater lowering (Fig. 6).
- 530 Re-measured TCN <sup>21</sup>Ne surface exposure ages from amalgamated quartz clasts agree with the 531 derived U-Pb silcrete chronology and are younger than the youngest U-Pb silcrete age obtained 532 (Fig. 6), i.e., in stratigraphically correct order. The surface exposure ages mark the abandonment 533 of the fluvial terraces and the onset of the Kuiseb River incision at  $\sim$ 2.8 Ma. The latter caused a 534 groundwater lowering of the water table and the cessation of calcrete formation within the 535 Karpfenkliff Conglomerate Formation. Re-measurements of the quartz clasts from the Oswater 536 terrace downstream of the Karpfenkliff and Kamberg cliff sampling sites confirm the exposure 537 ages previously obtained by Van Der Wateren and Dunai (2001). The exposure ages of the 538 Karpfenkliff and Oswater terrace constrain the period of major canyon incision to  $\sim$ 2.8 -1.3 Ma 539 (Fig. 6).
- 540 With the aid of absolute U-Pb silcrete and surface exposure dating, it is now possible to redefine 541 depositional ages or depositional periods for sediments in the Central Namib Desert, some of 542 which are widely used as marker horizons. Our U-Pb silcrete ages constrain the timing of sediment 543 deposition within the Kuiseb Canyon (Karpfenkliff Conglomerate Formation) to be older than 544  $\sim$ 7 Ma, as silcrete formation within the conglomerates postdates deposition thereof (Fig. 6). The 545 incision age of the Proto-Kuiseb and the subsequent deposition by the Karpfenkliff Conglomerate as proposed by Miller et al. (2021) of  $\sim$ 5 Ma, does not agree with our absolute U-Pb ages. If the 546 547 relative biostratigraphic dating of Pickford and Senut (2000) is valid, the proto-Kuiseb canyon was filled by the Karpfenkliff Conglomerate Formation over a time period of up to 6-7 Ma 548

(Diamantornis corbetti at Elim ~14 15 Ma, see Pickford and Senut, 2000). Verification and
absolute direct dating of the Karpfenkliff Formation is still lacking and is a target for future studies.
Our fluvial chronology substantially supports the chronological data obtained by Van Der Wateren

552 and Dunai (2001).

### 553 Pliocene Calcrete Formation – Steady State Climate

554 Our U-Pb ages indicate a relatively calm or transitional phase between aggradation and backfilling 555 of the Proto-Kuiseb (and presumably other drainage systems such as the Swakop) and the 556 renewed incision by the recent Kuiseb River, throughout the Pliocene. U-Pb ages of microscale 557 silcrete from the same stratigraphic horizon indicate a long-term stable groundwater level, i.e. no 558 significant aggradation or degradation.

559 As the formation of groundwater calcrete is generally restricted to specific environmental 560 conditions, the existence and chronology of its formation in the Central Namib Desert now allows 561 us to relate these environmental conditions to specific episodes in the past and thus to obtain a 562 better and partly more quantitative paleoclimate and environmental reconstruction of the 563 Pliocene in the Central Namib Desert. We therefore interpret our U-Pb silcrete chronology as 564 marking the transition of the mean annual precipitation (MAP) from the upper potential limit of 565 approximately  $\sim 600 \text{ mm/yr}$  or the lower potential limit of 50 mm/yr in the Kuiseb catchment 566 during the Late Miocene. Whether there was a climate change from, or a return to, wetter 567 conditions during the Pliocene cannot be determined from our U-Pb chronology. Calcrete 568 formation ceased with the incision of the Kuiseb River and a significant lowering of the 569 groundwater table.

Age information from the Kalahari Basin by Houben et al. (2020) indicated a shift towards more 570 571 arid conditions since ~12 Ma, which intensified at ~4 Ma (Houben et al., 2020; Miller et al., 2010). 572 Marine records off Namibia (Fig. 7, ODP 1081, Hoetzel et al., 2017) suggest a shift to more arid 573 conditions over the course of the Mid to Late Miocene, controlled by a gradual increase in the 574 upwelling activity of the Benguela Current, initiated by a strengthening of the meridional gradient. 575 This shift is supported by pollen data (Hoetzel et al., 2015; Dupont et al., 2013), indicating the 576 expansion of savanna grasslands (C4 expansion) in Namibia since  $\sim$ 8 Ma, with a subsequent shift 577 during the Pliocene to more shrubland and desert vegetation (Hoetzel et al., 2015). 578 Compound-specific hydrogen isotopes (ODP 1085, Dupont et al., 2013) indicate a change in the 579 precipitation source from the Atlantic to the Indian Ocean since  $\sim 8$  Ma (Dupont et al., 2013). 580 Therefore, our U-Pb chronology of calcrete formation ( $\sim$ 7-3 Ma) tracks the shift to more arid 581 conditions with a corresponding reduction in the MAP to allow calcrete formation. Nevertheless, 582 this transition and aridification of the Namib was slow, and regional SST records (ODP 1082, 583 Etourneau et al., 2009; ODP 1081, Rosell-Melé et al., 2014, Fig. 7), as well as global paleoclimate

- records (benthic  $\delta$ 180, Westerhold et al., 2020, Fig. 7) indicate a relatively stable climatic period.
- 585 Rosell-Melé et al. (2014) proposed, based on their marine SST record off Namibia (ODP 1082),
- that the persistently warm Pliocene, with conditions analogue to a persistent Benguela '*El Niño*',
- 587 ended at the transition to the Pleistocene (Fig. 6).

### 588 Plio/Pleistocene Transition

- 589 During the transition from the late Pliocene to the early Pleistocene, the Central Namib Desert 590 underwent large-scale landscape rejuvenation with drainage reorganisation and incision. This is 591 the same period, in which Miller et al. (2010) reconstructed the major desiccation of the Etosha 592 paleolake (Fig. 6). Based on U-Pb calcrete ages from the Kalahari basin, Houben et al. (2020) 593 proposed an intensification of arid conditions since  $\sim$  3.8 Ma, older than our onset of more arid 594 conditions in the Central Namib at around 3 Ma. Higher offshore sedimentation rates off Namibia 595 may be associated with increased input of terrestrial material (Dupont et al., 2005) due to incision 596 of E-W flowing drainages into the Atlantic. The propagation of the Horingbaai fan-delta (between 597 Omaruru and the Ugab river) occurred approximately at the same time (2.7 -2.4 Ma) according to 598 Stollhofen et al. (2014), supporting the idea of large-scale landscape rejuvenation and incision of 599 multiple E-W flowing drainages in the Namib Desert.
- The underlying mechanism is still questionable, as several forcing factors could be responsible for the incision of major E-W flowing river systems: climate change and variability, sea level change and/or tectonic uplift. The latter was previously suggested by Ward (1987) and attributed to a late Neogene epeirogenic uplift. Stollhofen et al. (2014) also suggest that uplift could be one of the causes and/or at least a contributor to other factors, such as climate.
- 605 Data from the marine realm off Namibia suggest a further step towards more extreme arid 606 conditions during the Plio-/Pleistocene transition (Fig. 6). Local SST records (ODP 1082, 607 Etourneau et al., 2009; ODP 1081, Rosell-Melé et al., 2014, Fig. 7) indicate the onset of decreasing 608 SSTs at ~2.7-2.5 Ma and the significant shift towards increased upwelling activity of colder water 609 masses in the Benguela Current since ~2.2 Ma (Dupont et al., 2005; Marlow et al., 2000; Etourneau 610 et al., 2009). The significant decrease in SSTs correlates with the further intensification of 611 Northern Hemisphere glaciation since  $\sim 2.7$  Ma (Ruggieri et al., 2009). The pollen record (ODP 612 1082) of (Dupont, 2006) shows that arid to semi-arid biomes were rather limited prior to  $\sim$ 2.7 613 Ma, and that their concentration increases with higher variability since then, reflecting the 614 intensification of arid conditions in the Central Namib Desert (Dupont, 2006).
- 615 Vegetation change may be a major cause of the exposure of landscapes to accelerated erosion.
- 616 Major river incision in the Central Namib Desert thus occurred during a period of climate change
- and greater climate variability compared to the more persistently stable Pliocene (Rosell-Melé et

618 al., 2014), with the intensification of arid conditions in southern Africa, synchronous with major 619 global changes. We therefore propose that the major river incision of the Kuiseb River at the Plio-/Pleistocene transition was caused by a shift to more arid conditions with decreasing 620 621 precipitation, resulting in reduced river discharge, river steepening and incision (e.g. Whipple and Tucker, 1999; Bonnet and Crave, 2003; Molnar, 2001; Cooper et al., 2016). Catchment and river 622 623 systems such as the Kuiseb (and/or river systems such as the Swakop), which had reached a 624 steady-state during the more stable Pliocene, had to adapt to the new boundary conditions, which 625 is in line with the global increase in erosion rates at the Plio-/Pleistocene transition (Herman et 626 al., 2013; Herman and Champagnac, 2016). A major vegetation shift towards more arid biomes 627 and sparser vegetation cover increased the susceptibility of landscapes to erosion. The global 628 sea-level drop at the Plio-/Pleistocene transition may have had an additional impact on drainage 629 base levels.

The incision of the recent Kuiseb River can be constrained to a period between the derived terrace ages of ~2.8 and ~1.3 Ma (minimum age of the Oswater bedrock river terrace). The actual period of incision may be even shorter, given the cessation of fluvial sediment deposition offshore at ~2 Ma (Dupont et al., 2005). Deposition of the Oswater Formation indicates a phase of aggradation sometime after ~1.3 Ma, followed by an incision into the recent bed of the Kuiseb River.



Fig. 6: Compilation of paleoclimate records. (A) Alkenone SSTs from ODP 1082 (Etourneau et al., 637 638 2009) and ODP 1081 (Rosell-Melé et al., 2014). Intensification of Benguela upwelling according to Etourneau et al. (2009). (B) Sedimentation rate of ODP 1082 for the Plio/Pleistocene transition 639 640 (Dupont, 2006). (C) Desiccation of the Etosha paleolake from Miller et al. (2010). (D) U-Pb silcrete and surface exposure ages (this study). Numbers indicate identical clasts. 1 - DWA98008-Silc3, 2 -641 DWA98008-Silc4, 3 - DWA98008-Silc7, 4 - DWA98008-Silc8, 5 - DWA98013, 6 - DWA98007, 7-642 643 DWA98006, 8 – DWA98024. (E) Global Sea-Level curve from Hansen et al. (2013). Black dashed line 644 indicates relative mean sea-level during the Pliocene, followed by the global decrease since the Plio-645 /Pleistocene transition. (F) Global Cenozoic reference benthic foraminifer oxygen isotope dataset 646 (CENOGRID) from Westerhold et al. (2020).

#### 647 **Conclusion**

648 Our study demonstrates that microscale silcrete from the Central Namib Desert can be dated using 649 U-Pb LA-ICP-MS, and that layered silcrete incrustations can be used as paleoclimate archives. 650 LA-ICP-MS U-Pb dating of silcrete has advantages over bulk carbonate analysis because it is less 651 affected by potential interferences and contamination. The combined dating approach with 652 additional <sup>21</sup>Ne exposure age dating allows us to reconstruct major paleoclimate and landscape 653 changes since the Late Miocene for the Central Namib Desert. We can corroborate previously 654 obtained chronological data from Van Der Wateren and Dunai (2001) and place absolute age 655 constraints on some sediments from the Central Namib Desert, some of which are used as marker 656 horizons throughout the region. Our chronology of groundwater calcrete formation and river 657 incision adds crucial information with absolute dates to the 'Namib Group'. Although specific 658 precipitation ranges for calcrete formation are still being debated, we can assign potential 659 precipitation ranges and their shifts to specific time episodes and thus provide a semi-quantitative 660 picture of the aridification of the Central Namib Desert during the Late Miocene to the 661 Plio-/Pleistocene. Our terrestrial paleoclimate record of microscale silcrete formation, i.e., 662 calcrete formation, supports the marine evidence for a persistently stable Pliocene climate in the 663 Central Namib Desert. The cessation of groundwater calcrete formation was caused by the deep 664 incision of the Kuiseb River (presumably synchronous with other E-W flowing drainage systems 665 of the Central Namib Desert) at the Plio-/Pleistocene transition, which can be explained by the 666 intensification of aridity, vegetation change, and presumably global sea-level drop. Global climate 667 change with the onset of the Pleistocene was most likely the major forcing factor for major 668 landscape rejuvenation and change in the Central Namib Desert. Precipitation decline in the 669 Kuiseb River catchment is identified as the tipping point for the local climate and landscape 670 response.

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### 678 Author Contribution:

B.R. fieldwork, sample preparation, <sup>21</sup>Ne noble gas analytic, data evaluation, manuscript writing.
R.A. U-Pb dating, sample analysis thin section, Raman, data analysis, manuscript writing. A.R.

Raman, F.M.v.d.W. fieldwork, T.J.D. fieldwork, data evaluation. A.G. data analysis. All authors
reviewed the manuscript.

## 683 Additional Information

- Declaration of interest: The authors declare that the research was conducted in the absence of
   any commercial or financial relationships that could be construed as a potential conflict of
   interest.
- 687 Data availability statement: All data generated or analysed during this study are included in this
  688 published article (and its supporting information files).

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