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Strontium and oxygen isotope analyses of the double burial of Bonn-Oberkassel

Abstract

Enamel and dentine of a first molar from the female and a third molar from the male of the late Pleistocene double burial of Bonn-Oberkassel were subject to strontium and oxygen isotope analyses. The strontium isotope ratios are overall in agreement with the Rhineland as catchment area, but not exclusive to it. Despite the location of the site at the periphery of the *Siebengebirge* (literally "seven mountains"; a range of hills southeast of Bonn) preferential foraging in this volcanic region can be ruled out. The oxygen isotope data reveal a large difference between both individuals. This is likely caused by a possible breastfeeding signal in the first molar of the female, but also points to spatial or temporal differences in the habitats of both individuals during childhood. Moreover, especially the oxygen isotope data of the male indicate lower temperatures than those that are typical today. The results demonstrate the possibilities of isotope analyses in revealing hunter-gatherer mobility and late Pleistocene climate conditions, but also the importance of contemporaneous comparative data and the application of sampling techniques with higher temporal resolution.

Introduction

Isotope analyses of teeth and bones that reveal mobility patterns in human prehistory are well-established methods in modern anthropological research. Nevertheless, such applications have so far concentrated on sedentary populations (Bentley et al. 2012; Evans et al. 2012), while those for Pleistocene hunter-gatherers are scarce (Richards et al. 2008). Therefore, the oxygen and strontium isotope data of the double burial from Bonn-Oberkassel (Henke et al. 2006), which dates to about 13,400 to 14,000 years BP, are rather singular for the area in question during that time period.

The investigation presented here was guided by questions regarding the geographic origin and mobility of both individuals, while also addressing palaeoclimatic information. Is there any indication that both individuals grew up in the same habitat? How do the strontium isotope ratios relate to the baseline values near the site? Do the oxygen isotope data reflect the climatic conditions at the end of the Pleistocene?

The interpretation of isotope data of single individuals is always a challenge (Alt et al. 2012). This is especially the case for Bonn-Oberkassel where sampling was restricted to a single tooth per individual and comparative data were taken from much younger, Holocene contexts and hence from material that formed under different climatic conditions. Therefore, the interpretation presented here may end up being modified in the future when more information becomes available.

Methodological background and sample material

The interdisciplinary investigation of the double burial from Bonn-Oberkassel included oxygen and strontium isotope analysis of dental tissues. Strontium is a trace element that occurs in rocks and is released during weathering. Its isotopic composition ($^{87}Sr/^{86}Sr$) depends on the kind and age of the geological units (Bentley 2006; Knipper 2004). The element is transported via the food chain without any appreciable isotope fractionation (Capo et al. 1998) and incorporated into the mineral component (hydroxyapatite) of teeth and bones. The key material for analyses is enamel, which remains unchanged after its mineralization between birth and adolescence

(AlQahtani et al. 2010). Dentine and bone are more frequently subject to diagenetic alterations, so that their analytical results often represent a mixture of biogenic strontium and strontium which intruded from the surrounding soil (Chiaradia et al. 2003; Nehlich et al. 2009).

Strontium isotope analysis of enamel is an overall effective method for recognizing residential changes among locations with different geological characteristics. In this study we took bulk samples of the enamel and dentine of a first molar of the female and a third molar of the male that had been subject to previous aDNA analysis. Using dentine for genetic studies and enamel of the same tooth for isotope analysis has proven very effective (Brandt et al. 2010). In order to safeguard the material from the fossils, we did not sample more teeth from both individuals, even though their different intervals of enamel mineralization or the application of laser ablation strontium isotope analysis would have increased the temporal resolution of the analytical results (de Jong et al. 2010; Simonetti et al. 2008).

In Holocene contexts, oxygen isotope analysis is used alongside strontium isotope investigations to unlock information on human mobility (Evans et al. 2012). The approach is based on the geographic variation of the isotopic composition of oxygen in rainwater, which is expressed as $\delta^{18}O$. This number describes the difference of the $^{18}O/^{16}O$ ratio of the sample in relation to the isotope ratio of a standard substance (McKinney et al. 1950). Crucial factors for spatial variation include the distance from the sea, latitude, and the elevation above sea level (Longinelli and Selmo 2003). Moreover, oxygen isotope ratios are sensitive to temperatures. This causes seasonal variation of the $\delta^{18}O$ values of precipitation, but also reflects long-term palaeoclimatic alterations (Knipper 2011, with references therein; Stephan 2008). Drinking water is the main source of oxygen incorporated in the phosphate and the carbonate fraction of the hydroxyapatite (Chenery et al. 2012). Here, we investigated the phosphate fraction, which is very resistant to diagenetic alterations (Iacumin and Longinelli 2002; Luz et al. 1984). Metabolic processes and the incorporation of oxygen into biogenic tissues cause isotope fractionation. Therefore, inferring the isotope composition of the oxygen that is bound in the imbibed water using the $\delta^{18}O$ values of teeth and bones requires data conversion using species-specific equations. Over recent years several of such equations have been developed for humans (Chenery et al. 2010; Daux et al. 2008; Pollard et al. 2011) and have been applied to the data from Bonn-Oberkassel.

Laboratory methods

Sample preparation for strontium isotope analysis followed previously published procedures (Knipper et al. 2012a; Knipper et al. 2012b). All surfaces were cleaned and the enamel and dentine of the tooth fragments were separated using dental drilling equipment and homogenized in an agate mortar. After rinsing with de-ionized water, the powders were reacted with 0.1 M acetic acid buffered with Li acetate (pH ~4.5), rinsed three times, and ashed (10 h, 850°C). Strontium was separated under clean room conditions using Eichrom Sr-spec resin. The Sr concentrations of the eluants were determined with a Quadrupole ICP-MS and represent about 80 % of the original Sr concentrations of the samples. A magnetic sector field MC-ICP-MS (Multi Collector-Inductively Coupled Plasma-Mass Spectrometer) VG Axiom was used for Sr isotope analysis. The raw data were corrected according to the exponential mass fractionation law to $^{88}Sr/^{86}Sr = 8.375209$. Blank values during the whole clean lab procedure including digestion, Sr separation, and measurement were less than 10 pg Sr. The NBS 987 standard that was run along with the samples yielded $^{87}Sr/^{86}Sr$ ratios of 0.71023 ± 0.00006 (n = 7). The Eimer & Amend standard yielded 0.70801 ± 0.00005 (n = 16). All clean-lab work and analysis were done at the Curt-Engelhorn Center for Archaeometry in Mannheim, Germany.

Oxygen isotope ratios were determined for the phosphate fraction of the hydroxyapatite. First the enamel and dentine samples were pretreated with NaOCl and NaOH, followed by the silver phosphate being prepared (Knipper et al. 2014; Tütken et al. 2006). The sample powders were reacted in HF and neutralized with NH_4OH solution. The addition of $AgNO_3$ solution caused the precipitation of silver phosphate crystals (Ag_3PO_4) which were analysed in triplicates using a TC-EA at 1450°C coupled to a Thermo Scientific MAT 253 at the Department for Applied and Analytical Palaeontology at the University of Mainz. Raw data were normalized against IVA silver phosphate with $\delta^{18}O = 21.7$ ‰ (certificate no.: BN 180097). Ag_3PO_4 that was precipitated from NBS 120c prepared along with the samples gave $\delta^{18}O$ values of 22.1 ± 0.2 ‰. The in-house standards of synthetic hydroxyapatite (HAP)

Sample	Sex	Tooth	Tissue	Sr ppm	^{87}Sr/^{86}Sr	2 s	δ^{18}O (‰ vs. VSMOW)	1 s
BOB 1.1	Female	26	Enamel	38	0,70938	±0,00004	16,2	±0,2
BOB 1.2	Female	26	Dentine	205	0,70794	±0,00002	16,0	±0,1
BOB 2.1	Male	18	Enamel	55	0,7086	±0,00006	13,9	±0,3
BOB 2.2	Male	18	Dentine	160	0,70788	±0,00006	13,8	±0,4

Table 1 Strontium and oxygen isotope data of the enamel and dentine from the double burial of Bonn-Oberkassel.

yielded 17.1 ± 0.1 ‰ and Roman pig bones from the site of Dangstetten (SUS-DAN) gave 14.7 ± 0.2 ‰.

Results and discussion

Strontium isotope analysis

The upper first molar of the female yielded an ^{87}Sr/^{86}Sr ratio of 0.70938 in the enamel and 0.70794 in the dentine (Table 1). The strontium isotope ratios of the upper third molar of the male were 0.70860 in the enamel and 0.70788 in the dentine. The dentine samples have three to five times higher Sr concentrations and lower ^{87}Sr/^{86}Sr ratios than the enamel. This suggests a contribution of strontium from the burial environment to the dentine. Mantle-derived strontium with typically low ^{87}Sr/^{86}Sr values (Jung et al. 2012) was certainly omnipresent at the locality,

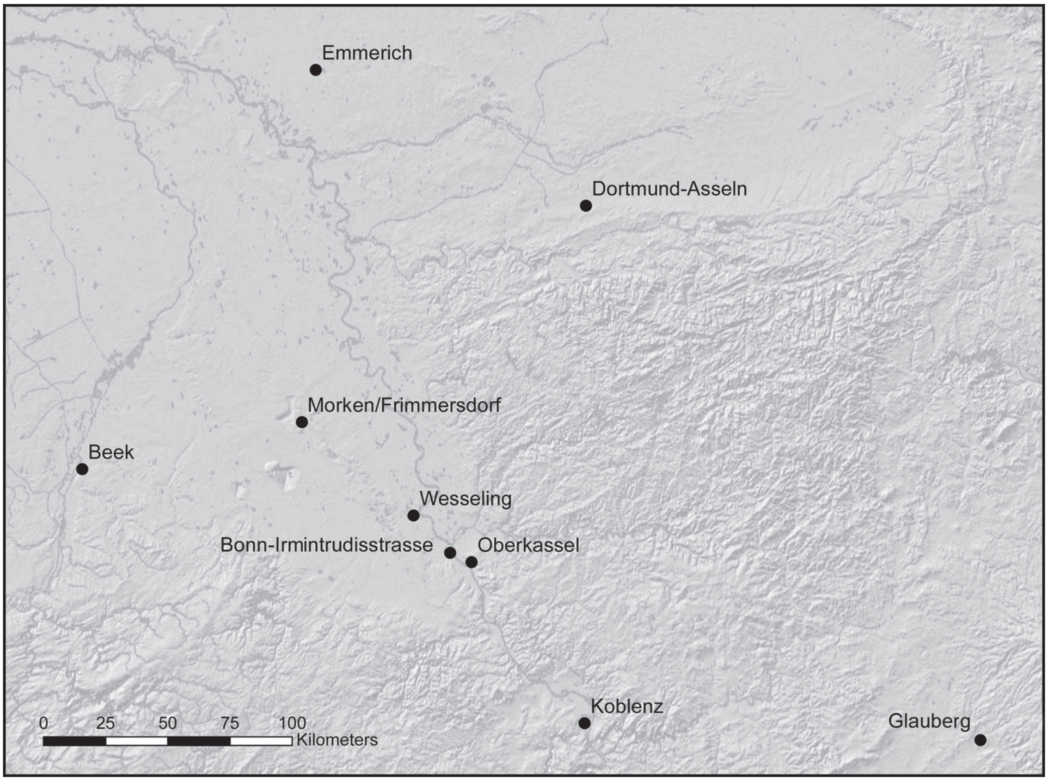

Fig. 1 Map illustrating the locations of Bonn-Oberkassel and the comparative datasets.

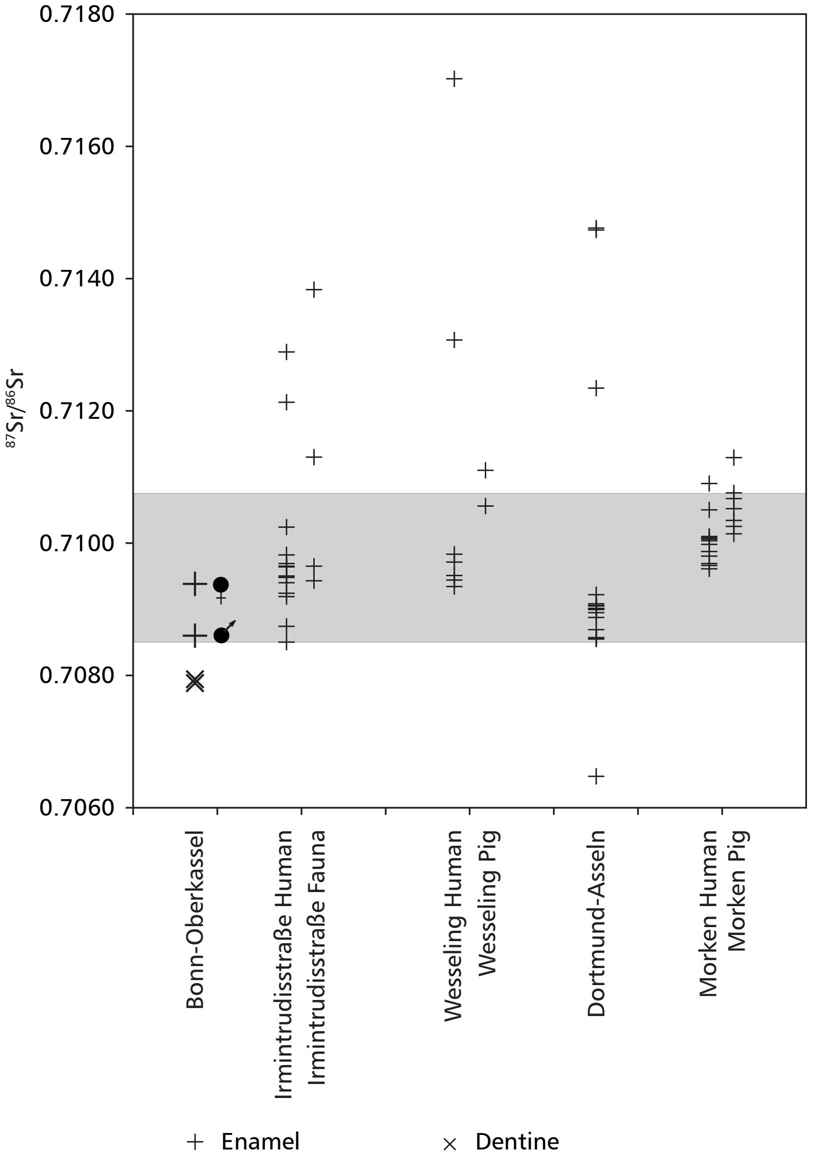

Fig. 2 Strontium isotope ratios of enamel and dentine of the double burial of Bonn-Oberkassel in comparison to the datasets of Roman burials at Bonn-Irmintrudisstrasse (Knipper et al. unpublished data) and Early Medieval cemeteries at Wesseling (Knipper et al. n.d.), Dortmund-Asseln (Sicherl 2007), and Morken/Frimmersdorf (Knipper et al. in press). The grey bar highlights the typical data range of lowland locations in the Rhineland.

which was used as a basalt quarry in modern times. Due to their diagenetic overprint, the dentine data can give an indication of the "local" baseline values at the recovery site of the double burial. Because exchange with the soil fluid is not necessarily complete, such estimations should be backed up with other complementary data.

The isotope ratios of the enamel of both individuals are more radiogenic than the biologically available strontium in areas that are shaped by Tertiary volcanism (Knipper 2011; Knipper et al. 2014; Oelze et al. 2012), which also include the *Siebengebirge*, where the site is located. Even though some influence from the basalts and trachytic tuffs that dominate the area cannot be excluded (Jung et al. 2012), the home range of both individuals was certainly not restricted to the low mountain range of the *Siebengebirge*.

The isotope ratios of the enamel of both individuals fall into the data spectra of several Holocene burial communities in the Rhineland, including a Roman cemetery at Irmintrudisstrasse at Bonn (Knipper et al. unpublished data) and Early Medieval burials from Wesseling ca. 15 km NNW (Knipper et al. unpublished data), Dortmund-Asseln ca. 95 km NE (Sicherl 2007) and Morken/Frimmersdorf, some 50 km NW of the site (Knipper et al. in press) (Fig. 1). Especially the isotope ratio of the female is well comparable with the data spectrum at Bonn-Irmintrudisstrasse, the nearest existing dataset (Fig. 2). It is in overall agreement with a possible dietary catchment in the Rhineland, but certainly not exclusive to it (Bentley and Knipper 2005). The lower $^{87}Sr/^{86}Sr$ ratio of the enamel of the male may point to additional exploitation of food sources from limestone areas and—to a very limited extent—from the Tertiary volcanic rocks near the site. The difference in Sr isotope ratios of both individuals suggests that they consumed food from slightly different habitats during tooth formation, which is to be expected for rather mobile hunter-gatherers. Overall, each of the analyzed bulk samples integrated strontium from different sources. Those can only be distinguished with a more detailed analytical strategy, ideally including laser ablation strontium isotope analysis (de Jong et al. 2010; Simonetti et al. 2008).

Oxygen isotope analysis

In contrast to the strontium isotope data, the oxygen isotope composition of the phosphate fraction ($\delta^{18}O_p$) of the enamel and the dentine of the same teeth are well comparable. The first molar of the female produced $\delta^{18}Op$ values of 16.2 ‰ in the enamel and 16.0 ‰ in the dentine, while these values for the third molar of the male were 13.9 ‰ in the enamel and 13.8 ‰ in the dentine (Table 1). The small difference between the oxygen isotope ratios of the two dental tissues argues for the integrity of the samples and the resistance of the phosphate fraction of the hydroxyapatite to diagenetic alterations.

Because of the small number of samples, the lack of contemporaneous comparative analyses and the considerable climatic alterations from the late Pleistocene to modern times, the evaluation of the analytical data is rather preliminary. An extensive database of oxygen isotope ratios of modern precipitation is provided by the International Atomic Energy Agency (IAEA), which maintains several hundred monitoring stations worldwide. The nearest stations are situated at Koblenz some 50 km SE, at Beek in the Netherlands c. 100 km W, and at Emmerich c. 140 km NW of the site (IAEA 2006) (Fig. 1). Their weighted annual average $\delta^{18}O$ values range from -7.5 ± 0.5 ‰ at Beek to -7.0 ± 0.8 ‰ at Koblenz.

In order to estimate the $\delta^{18}O_p$ values of dental tissues of individuals who imbibed drinking water originating from modern precipitation from the Rhineland, we applied the regression equations (4) and (6) proposed by Daux et al. (2008), the modified equation by Levinson et al. (1987) as published by Chenery et al. (2010) and the equation drawn from the data "superset" by Pollard et al. (2011). This results in average $\delta^{18}O_p$ values of dental tissues between 16.5 ‰ (Beek; "superset": Pollard et al. 2011) and 17.6 ‰ (Koblenz; Levinson et al. 1987; Chenery et al. 2010). The inclusion of two standard deviations from the average suggests a range from 16.0 ‰ (Koblenz and Beek; "superset": Pollard et al. 2011) to 18.4 ‰ (Koblenz; equ. (4) and (6) by Daux et al. 2008) (Fig. 3). This range is a very conservative estimate for the modern regional baseline data to be expected in the Rhineland.

A further cornerstone for comparisons is human enamel from Holocene archaeological contexts. The nearest available data come from Early Medieval (6th century A.D.) burials at Morken/Frimmersdorf (Knipper et al. n.d.) c. 50 km NW of Oberkassel and from non-normative inhumations in settlement pits near the Iron Age "princely seat" of the Glauberg (late 5th century B.C.), ca. 140 km SE of Bonn (Knipper et al. 2014) (Fig. 1). Due to the NW-SE gradient of $\delta^{18}O$ in precipitation (Rozanski 1995), Morken/Frimmersdorf is more comparable to Oberkassel, and lower baseline values are to be expected at the Glauberg. These trends are documented in the data of human enamel from both sites, which vary between 17.3 and 19.2 ‰ at Morken/Frimmersdorf (Knipper et al. n.d.) and from 15.1 to 17.1 ‰ at the Glauberg (Knipper et al. 2014) (Fig. 3). When considering these data, however, we need to appreciate that the data set from Morken/Frimmersdorf includes many deciduous teeth and first molars, which are typically enriched in ^{18}O due to early infant breast milk consumption (Wright and Schwarcz 1998). The sample set from the Glauberg comprises two non-local individuals with $\delta^{18}O$ values of 15.1 and 15.5 ‰, respectively, while the main data cluster covers a range between 16.1 and 17.1 ‰.

Figure 3 presents the oxygen isotope ratios of the dental tissues of the male and the female from Bonn-Oberkassel in relation to these comparative data. The $\delta^{18}O$ values of the male are considerably lower than both the data ranges to be expected from the consumption of drinking water from modern precipitation as well as the analytical results for Holocene burials in the greater vicinity. The $\delta^{18}O$ data of the enamel and the dentine of the female are likewise distinctly lower than the data spectrum at Morken/Frimmersdorf. There is some overlap with the oxygen isotope composition of the regional modern precipitation if the equation for the data "superset" by (Pollard et al. 2011) is applied, and with the dataset from the Glauberg, which only includes post-weaning enamel. Yet, breast milk consumption may have caused an enrichment of ^{18}O in the analyzed first molar of the female of up to 2 ‰ (Evans et al.

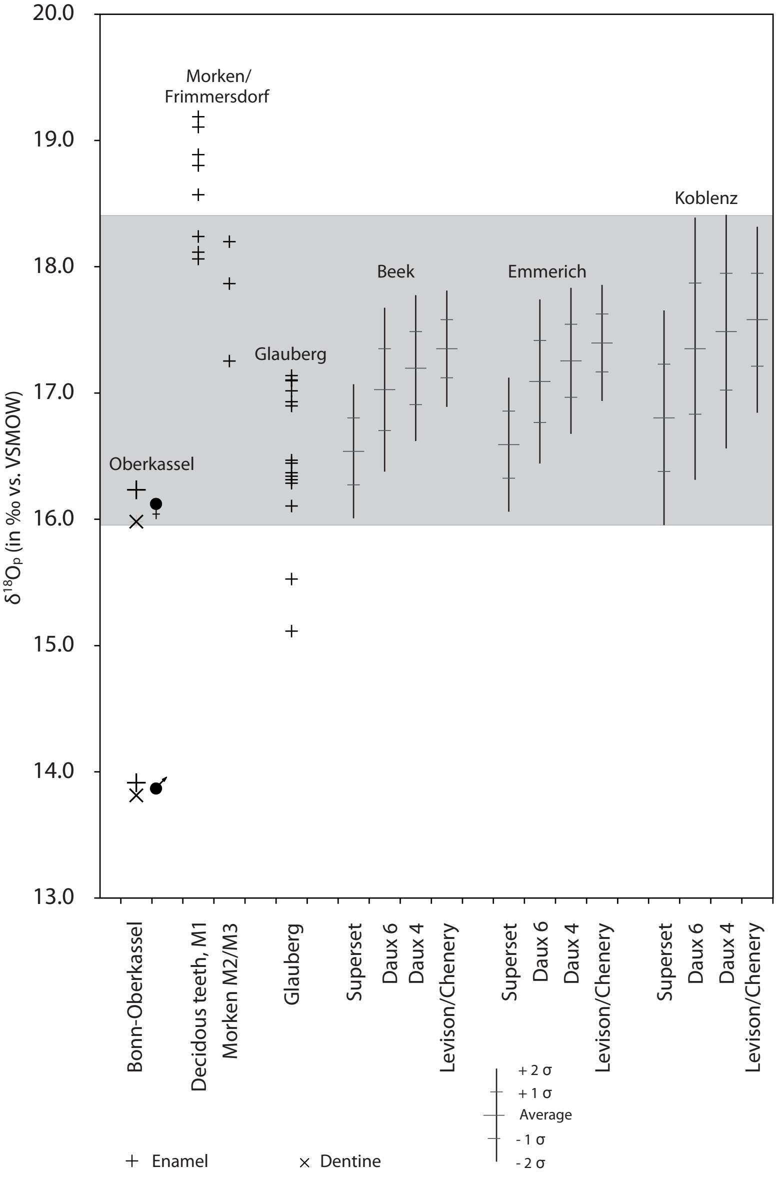

Fig. 3 Oxygen isotope ratios of enamel and dentine of the double burial of Bonn-Oberkassel in comparison to the datasets of Early Medieval burials at Morken/Frimmersdorf, Iron Age inhumations at the Glauberg (Hesse), and data ranges to be expected if drinking water is drawn from modern precipitation in the Rhineland. The latter estimates are based on data from the meteorological monitoring stations at Beek (Netherlands), Emmerich and Koblenz (IAEA 2006) and converted to $\delta^{18}O_p$ values using the equations for the data "superset" by Pollard et al. (2011), equations (4) and (6) by Daux et al. (2008), and the equation proposed by Levison et al. (1987) and modified by Chenery et al. (2010).

2012). Thus, enamel that was mineralized after weaning can also be expected to have $\delta^{18}O$ values below the modern regional ranges.

In summary, the $\delta^{18}O$ values of the dental tissues of both individuals are lower than those of the Holocene comparative data, which points to overall cooler mean annual temperatures. Yet, even when taking into account a breastfeeding effect for the female, the $\delta^{18}O$ data of both individuals display a considerable dissimilarity, which suggests that they grew up using different drinking water sources. This observation could have resulted from spatial variation, meaning that their childhood habitats included geographically different localities. Another explanation that must be considered is that the two individuals did not live contemporarily. All information that has been conveyed about the discovery in 1914 and the accompanying finds (Verworn et al. 1919) points to a contemporaneous inhumation. Nevertheless, the calibrated ^{14}C dates of the bones diverge by about 700 years even though all measurements point to the beginning of the late glacial interstadial complex (GI 1 Greenland Interstadial 1). Given the rapid temporal and spatial climatic changes at the end of the last glaciation (Coope et al. 1998; Huijzer and Vandenberghe 1998), a chronological difference between the two individuals may have contributed to the deviation of the oxygen isotope data of their teeth. Short-term climatic alterations could well have caused shifts of the oxygen isotope ratios in dental tissues of several per mil. However, as discussed elsewhere in this volume (see the contribution by Street and Jöris), a calibrated age of about 14,200 years, which corresponds to the Meiendorf interstadial, seems most likely for the burial complex. Because the analysis of different tooth types likely also added to the dissimilarities, analyzing post-weaning dental tissue of the female would be a first step in confirming or refuting the observed dissimilarity between both individuals.

Conclusions

The presented strontium and oxygen isotope analyses of the double burial are among the first such data from the central European Palaeolithic. Hence their interpretation is limited by the very small sample size and the lack of contemporaneous comparative data. The combination of the information of both isotope systems suggests that the male and the female did not consume food from exactly the same habitats during the formation of the analyzed tooth crowns. The homelands of both individuals extended certainly well beyond the site and the volcanic province of the *Siebengebirge*. The considerable offset between the oxygen isotope ratios of the samples from both individuals underlines the existence of variant catchment areas, which could either be due to spatial or to temporal differences. Because isotope data of hunter-gatherers are still very scarce, it is difficult to evaluate whether the observed variation would occur naturally among people from the same group who moved regularly or if they indicate origins in different groups. There is still much research potential in higher-resolution sampling techniques. Further studies should therefore include samples from more teeth of different formation periods and laser ablation strontium isotope analysis. Analyzing post-weaning enamel of the female would enhance the comparability of the data of both individuals and foster the palaeoclimatic information, which momentarily suggests cooler ambient temperatures than we experience today.

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