

Provenance analysis of human cremations by $^{87}\text{Sr}/^{86}\text{Sr}$ isotopic ratios: migration into an Iron Age mining region in North-Rhine Westphalia

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Abstract

The Siegerland (North-Rhine Westphalia, FRG) is famous for its early mining industry and ore exploitation. The archaeological context of cremated burials as well as grave goods indicate parallels to today's Wetterau (Hesse), suggesting migration into the Siegerland. After morphological examination of the cremations augmented by a histological age-at-death determination, provenance analysis by use of stable strontium isotope analysis was carried out. 60 individuals from the burial mound at Netphen-Deuz in the Siegerland were available for anthropological examination. The $^{87}\text{Sr}/^{86}\text{Sr}$ isotopic ratio was measured in 29 dentine and 15 bone samples. At least 19 individuals exhibited a non-local isotopic signal which was compatible with a provenance from the Wetterau region. Since $^{87}\text{Sr}/^{86}\text{Sr}$ isotopic ratios in the bioapatite are thermally stable, provenance analysis of cremated finds is thus possible, whereby a testable archaeological hypothesis is prerequisite. Histological examination of cremated bones proved indispensable for the age-at-death estimation.

Introduction

The Siegerland in North-Rhine Westphalia (FRG) is characterized by a low mountain range landscape that was not particularly suitable for agriculture in prehistoric times. Its economic importance rather lies in its rich ore deposits, which include the largest siderite deposits worldwide, and other minerals that contain copper or silver (Kirnbauer and Hucko, 2011). Since the second half of the 4th century BC, a large-scale mining landscape seems to have developed suddenly and, according to pres-

ent knowledge, without any traces of previous such developments (Baales *et al.*, 2014). After a phase of particular proliferation, the early mining industry disappeared again suddenly around the turn to the Common Era and was not resumed before the early Middle Ages. It is therefore highly likely that both people and technology had arrived in the previously only poorly populated region for the purpose of metallurgy, that constituted a powerful *pull factor* for human migration (Zeiler *et al.*, 2017).

The burial site of Netphen-Deuz has been excavated between 1987 and 1996 but was not completely evaluated before now. The small number of burials (n=67) that had accumulated in the course of about 800 years suggests that the associated settlement must have been quite small. Since an unknown number of burials is most probably lost due to erosion and modern agricultural activities, the actual number of inhumations could have been larger. Only 22 out of the 67 burials could be classified chronologically based on the grave goods. The site had obviously been in use from the younger Iron Age until the Middle and Late Latène Period whereby the majority of the cremations are dated into the latter time. Find context and particular grave goods in some of the burials at the necropole exhibit obvious relations to a region located about 100 km further south-east, the Wetterau in today's Hesse (Figure 1). It is therefore plausible to hypothesize that several individuals buried at Netphen-Deuz primarily originated from there.

In the course of the recent evaluation of the site, 60 available cremations were subjected to an anthropological macro- and microscopical investigation. Since an explicit hypothesis was formulated based on the archaeological context and finds, stable strontium isotopic ratios were measured in the cremations. Dental enamel is usually no more preserved in cremated finds. Therefore, compact bone, and wherever available dentine from the same individual was analyzed. $^{87}\text{Sr}/^{86}\text{Sr}$ isotopic ratios in bioapatite are measured since many years for the scope of provenance analysis. Strontium is incorporated by food and drinking water, whereby the bioavailable strontium isotopic ratio in a region is frequently related to the respective such ratios in the soil and drinking water (Bentley, 2006; Slovak and Paytan, 2012). Since strontium is always associated with calcium in nature, dietary behavior is also very important for the mixture of source strontium isotopes in the consumers' tissue. $^{87}\text{Sr}/^{86}\text{Sr}$ ratios in the bioapatite are thermally stable (Harbeck *et al.*, 2011) and not altered by cremation processes although even prehistoric cremation pyres can reach tempera-

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tures up to 1000°C (McKinley, 2016). Recently, it has been shown experimentally by Snoeck *et al.* (2015) and later by an analysis of archaeological finds (Snoeck *et al.*, 2016) that cremated bones are a suitable substrate for provenance analysis by use of stable strontium isotopic ratios.

In this paper, we present the results of a systematic isotopic investigation of the cremations from Netphen-Deuz in an attempt to answer the following questions: Is there archaeometric evidence for a substantial migration into the Siegerland? Are $^{87}\text{Sr}/^{86}\text{Sr}$ ratios capable of supporting the hypothesis of immigration from the Wetterau region? If both answers would be *yes*, then not only material culture was introduced into the Siegerland during the Iron Age, but it had been accompanied by people that populated the region.

Materials and Methods

60 individuals from the Netphen-Deuz necropolis that was in use from the early to the late Iron Age (ca. 800-15 BC) were investigated. The burial type, urns and grave goods exhibit parallels to the Wetterau region in Hesse that is located at a distance of about 100 km (Zeiler and Nikulski, 2015).

All cremations were first investigated osteologically according to standard anthropological criteria (Grupe *et al.* 2015; McKinley, 2000). Typical for the Iron Age, preservation of the cremations was poor. While the weight of a modern cremation varies around approximately 2 kg (Warren and Maples, 1997), average weight of the cremations of this study was 360.3 g only. Also fragment size was very small: with a variability between <15mm and 35 mm, 65.6% of all cremations exhibited a fragment size between 15 and 25 mm. Colour of the finds was indicative of a heat exposure $\geq 800^\circ\text{C}$. Due to this poor preservation, macroscopical age-at-death assessment was frequently restricted to very gross age categories such as *minimum adult*. Therefore, compact bone of all individuals was examined histologically in addition (*e.g.* Bell, 2012).

For this, one piece of compact femur or humerus per individual was embedded into epoxy resin (Biodur E12, Gunter von Hagens, Heidelberg) that was mixed with hardener (Biodur E1) in a ratio of 100:28. Embedded specimens were kept for 48 hours in a desiccator at a pressure of 200 mbar. On the third day, the samples were exposed to atmospheric pressure at room temperature for another 24 hours, and finally dried for 24 hours at 35°C . After complete solidification of the resin, at least five cross sections per specimen with a thickness between 70 and 100 μm were prepared with a microtome saw (Leica 1600). The thin sections were air dried for 24 hours and fixed onto glass slides. Prior to microscopy, the sections were ground with silicon-carbide-sandpaper (Struers, grain 220) and polished with AP-A-suspension (Struers comp.). After drying for 24 hours, the sections were finally covered with glass, inspected with a light microscope Axioskop 2 plus (Zeiss) under both bright field and polarized light, and documented with Axiocam MRC (Zeiss) at a 50- and 100fold magnification. The images were saved digitally by use of AxioVision (4.8.1) program and edited with Photoshop elements 12. Age-at-death was quantified by use of the regressions published for cremated femoral bone by Hummel and Schutkowski (1993),

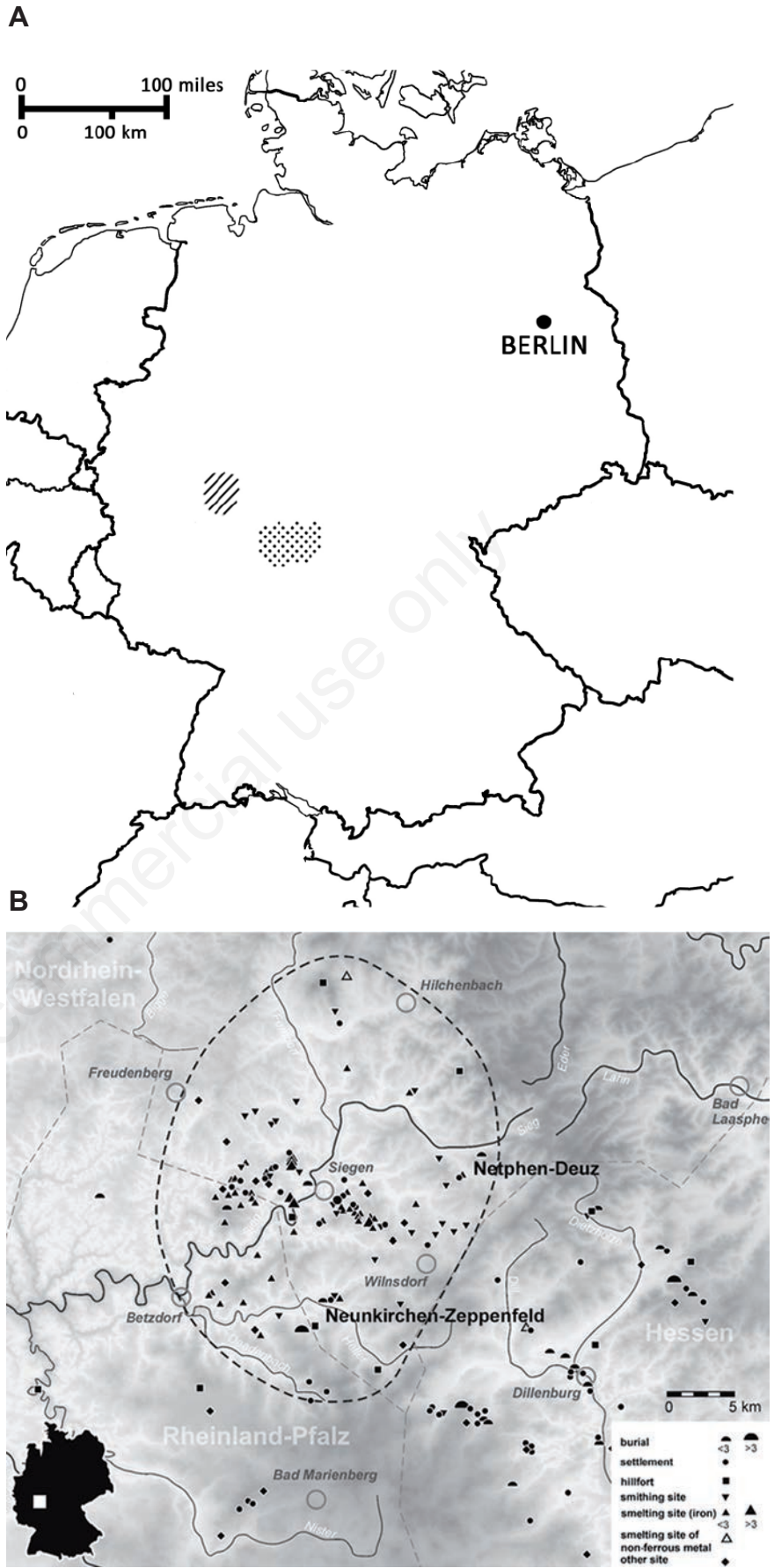


Figure 1. Location of the Siegerland (striped) and the Wetterau (dotted) regions in Germany (A), and location of the burial site of Netphen-Deuz (B).

Table 1. Morphological and histological diagnoses and $^{87}\text{Sr}/^{86}\text{Sr}$ isotopic data for the cremated finds investigated in this study.

Burial	Morphological age	Histological age (femur)	Histological age (humerus)	Sex	$^{87}\text{Sr}/^{86}\text{Sr}$	2 SE (M) [%]	Sample
Deuz 1	min. adult	66.2 ± 10.9	-	indet	0.7099	0.002	femur
Deuz 2	min. juvenile	39.9 ± 10.9	-	indet	0.7127	0.0021	femur
Deuz 3	min. adult	43.5 ± 10.9	25.4 ± 8.8	female	0.70898	0.0034	dentine incisor
Deuz 4	max. adult	39.9 ± 10.9	-	female	-	-	
Deuz 5	minimum adult	39.9 ± 10.9	-	male	-	-	
Deuz 6	20-40 years	46.5 ± 10.9	-	male	0.70931	0.0059	dentine
Deuz 7	20-40 years	76.3 ± 10.9	-	female	0.70932	0.0018	dentine incisor
Deuz 8	max. 40	28.0 ± 10.9	-	indet	0.71154	0.0021	femur
Deuz 9	n.d.	31.6 ± 10.9	-	male	-	-	
Deuz 10	30-40 years	32.7 ± 10.9	-	female	0.70828	0.0026	dentine premolar
Deuz 11	20-60 years	31.0 ± 10.9	-	indet	0.71232	0.0029	long bone
Deuz 12	min. adult	25.0 ± 10.9	-	male	0.71004	0.0013	dentine
Deuz 13	n.d.	35.7 ± 10.9	-	indet	-	-	
Deuz 14	n.d.	34.5 ± 10.9	-	indet	-	-	
Deuz 15	n.d.	13.1 ± 10.9	-	indet	-	-	
Deuz 16	n.d.	47.7 ± 10.9	-	female	0.71033	0.0027	dentine incisor
Deuz 17	max. adult	33.3 ± 10.9	-	indet	0.71175	0.0022	femur
Deuz 18	n.d.	23.2 ± 10.9	-	female	0.71033	0.0021	dentine
Deuz 19	n.d.	52.4 ± 10.9	-	indet	0.71349	0.0023	femur
Deuz 21	min. adult	51.2 ± 10.9	-	indet	0.70991	0.0029	dentine incisor
Deuz 22	n.d.	19.6 ± 10.9	-	indet	0.71383	0.0033	dentine canine
Deuz 24	20-60 years	26.2 ± 10.9	-	indet	-	-	
Deuz 25	n.d.	29.2 ± 10.9	-	indet	-	-	
Deuz 26	n.d.	19.6 ± 10.9	40.3 ± 8.8	female	0.71286	0.0021	humerus
Deuz 27	20-60 years	22.6 ± 10.9	-	indet	0.71322	0.002	tibia
Deuz 28	min. adult	31.6 ± 10.9	-	indet	0.71091	0.0023	dentine incisor
Deuz 29	n.d.	23.8 ± 10.9	-	indet	0.71056	0.0021	dentine premolar
Deuz 30	20-60 years	32.7 ± 10.9	-	male	0.7098	0.0022	dentine molar
Deuz 31	n.d.	26.8 ± 10.9	-	indet	0.713	0.002	long bone
Deuz 32	n.d.	35.1 ± 10.9	-	indet	-	-	
Deuz 33	n.d.	30.4 ± 10.9	-	indet	0.71258	0.0043	femur
Deuz 34	n.d.	41.1 ± 10.9	-	indet	0.70797	0.0021	dentine premolar
Deuz 35	30-40 years	31.0 ± 10.9	-	indet	0.71339	0.0019	femur
Deuz 36	40-70 years	60.2 ± 10.9	-	male	0.71069 0.70797	0.0014 0.0024	femur dentine premolar
Deuz 37	n.d.	36.3 ± 10.9	-	indet	-	-	
Deuz 38	min. adult	38.7 ± 10.9	40.3 ± 8.8	male	0.71345	0.0033	dentine
Deuz 39	20-60 years	25.0 ± 10.9	-	female	0.71221	0.002	dentine
Deuz 40	40-60 years	51.2 ± 10.9	-	female	0.7146	0.0026	humerus
Deuz 41	20-60 years	59.6 ± 10.9	45.6 ± 8.8	male	0.70928	0.0023	dentine premolar
Deuz 42	min. adult	31.0 ± 10.9	24.5 ± 8.8	female	0.71326	0.0018	dentine molar
Deuz 43	n.d.	17.8 ± 10.9	-	indet	-	-	
Deuz 45	40-70 years	42.3 ± 10.9	-	indet	-	-	
Deuz 46	n.d.	30.4 ± 10.9	-	indet	-	-	
Deuz 47	20-70 years	52.4 ± 10.9	-	male	0.71203	0.0014	dentine incisor
Deuz 48	40-70 years	16.6 ± 10.9	-	indet	0.70908	0.0014	dentine
Deuz 50	n.d.	36.9 ± 10.9	-	indet	-	-	
Deuz 51	n.d.	48.9 ± 10.9	-	indet	-	-	
Deuz 52	min. adult	68.5 ± 10.9	-	male	0.71345	0.0029	dentine incisor
Deuz 53	n.d.	33.3 ± 10.9	-	indet	-	-	
Deuz 54	20-40 years	31.6 ± 10.9	-	female	-	-	
Deuz 55	min. adult	29.8 ± 10.9	-	female	-	-	
Deuz 56	20-40 years	28.0 ± 10.9	-	female	0.71354	0.0024	phalanx

Continued on the next page.

and by Yoshino *et al.* (1994) for uncremated humeri. Regressions established on uncremated bones need an adaptation of the visual field when applied to cremations (Nováček, 2012). An image visualizing the counting method is provided here (Figure 2).

29 dentine and 15 bone samples were selected for the strontium isotope analyses and processed according to Toncala *et al.* (2017). The samples were first washed ultrasonically three times with distilled water for 5 minutes each and air dried. Next, they were etched ultrasonically for 5 minutes with concentrated HCOOH and washed with distilled water until a pH between 5 and 6 was reached. Afterwards, the samples were weighed and heated in a muffle furnace for 12 hours at 800°C. After cooling to room temperature, the samples were weighed again for the calculation of the apatite yield. Next, the bones and teeth were ground to a fine, homogenous powder. 15 to 30 mg powder were weighed into Teflon cubes and dissolved in 1 mL concentrated nitric acid (65% suprapure). The solution was heated in closed cubes on a hotplate for 24 hours at 100°C for pressure digestion, and the liquid was evaporated afterwards. Prior to the column separation, the samples were solubilized in 1 mL 10N nitric acid (ultrapure) and heated on the hotplate at 100°C for 20 minutes. Sr-Resin (Eichrom®, SR-B25-S) was put into the columns that been previously filled with 1 mL doubly distilled strontium water (Fa. Roth). The columns were then cleaned with 1 mL 10N HNO₃ (ultrapure) to remove divalent ions. After that, the columns were flushed with 1 mL muriatic acid to remove lead, followed by a washing step with 1 mL doubly distilled strontium water. Finally, 100 µL of 10N nitric acid was added. 300 µL of the samples was applied onto the cleaned and conditioned columns and rinsed with 10N nitric acid in four steps: 100 µL, 200 µL, 600 µL, and finally 200 µL. Strontium was collected by adding 1000 µL 0.05N nitric acid (ultrapure), and

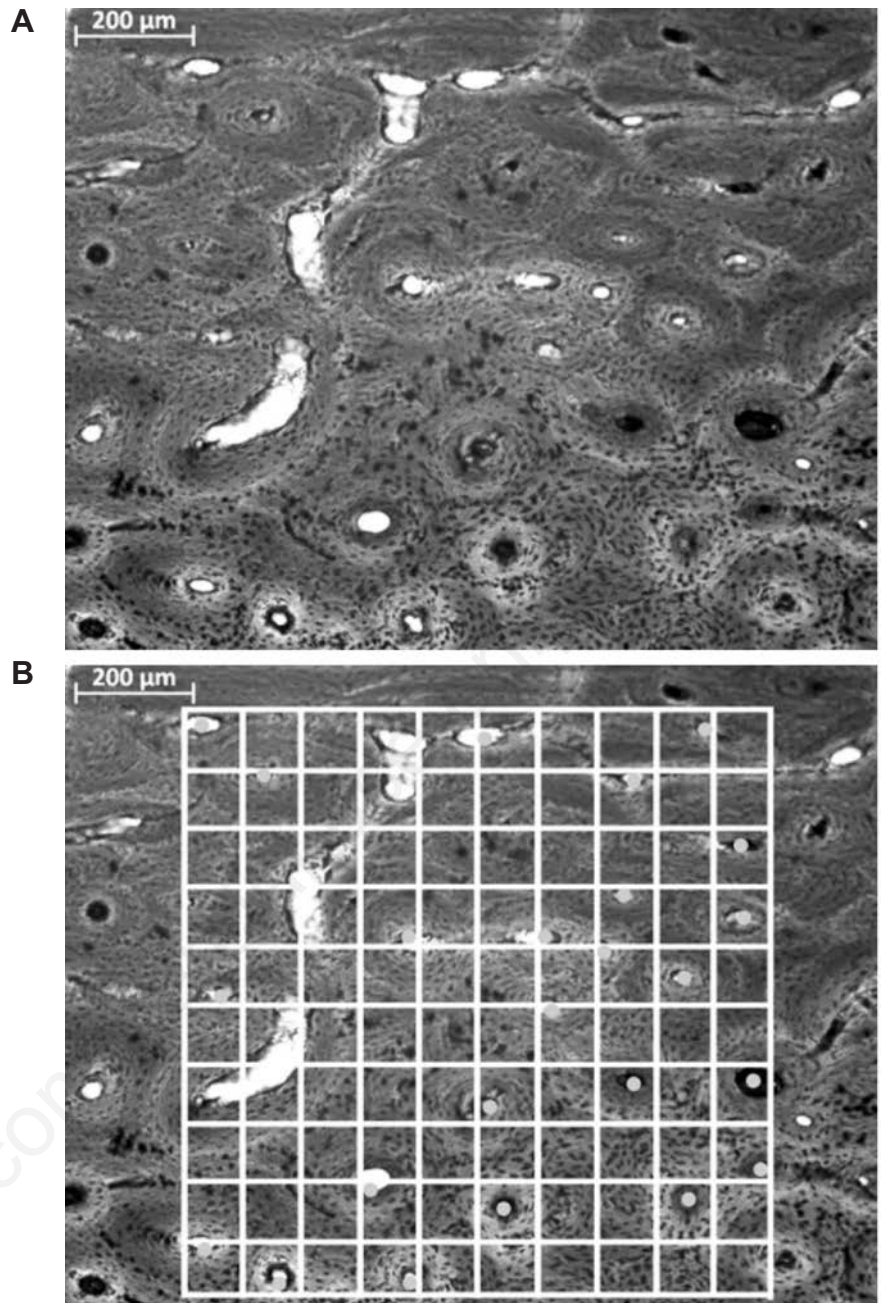


Figure 2. Example of histological sections (burial 53). A) 100 µm, 10× objective, without evaluation; B) 100 µm, 10× objective, with evaluation after Hummel and Schutkowski (1993). Images by author Sidney Sebald.

Table 1. Continued from previous page.

Burial	Morphological age	Histological age (femur)	Histological age (humerus)	Sex	⁸⁷ Sr/ ⁸⁶ Sr	2 SE (M) [%]	Sample
Deuz 57	20-60 years	45.9 ± 10.9	52.6 ± 8.8	indet	0.71189	0.0016	dentine premolar
Deuz 58	min. adult	53.0 ± 10.9	-	male	0.71263	0.0024	dentine incisor
Deuz 59	n.d.	29.8 ± 10.9	-	indet	-	-	
Deuz 61	min. adult	32.2 ± 10.9	-	indet	0.71317	0.0017	dentine premolar
Deuz 62	min. adult	44.1 ± 10.9	-	indet	0.71349	0.0038	dentine premolar
Deuz 63	min. adult	28.0 ± 10.9	32.4 ± 8.8	indet	0.71374	0.0033	humerus
Deuz 64	40-60 years	26.2 ± 10.9	25.4 ± 8.8	female	-	-	
Deuz 65	max. 60 years	36.9 ± 10.9	-	female	0.71315	0.0018	dentine molar
Deuz 66	-	-	-	indet	0.71404	0.0013	dentine

the samples were evaporated on a hotplate at 100°C. Stable strontium isotopic ratios were analyzed at the RieskraterMuseum Nördlingen with a Thermal Ionisation Mass Spectrometer Finnigan MAT 261.5 on single tungsten filaments. Standard reference material SRM 987 served for quality control. Measurement precision was ± 0.00001 , the measurement accuracy (=double standard error [%]) was ≤ 0.0043 . The data were

evaluated by univariate statistics and the Isoplot 4.1 software.

Results

Morphological, histological, and strontium isotopic data for all individuals are listed in Table 1.

By use of osteological features alone,

poor preservation of the cremations permitted for an age-at-death assessment for 36.1% of all individuals only, and another 31.1% could only be grossly categorized by a minimum or maximum age-at-death. Neither children nor elderly persons ≥ 60 years were detected. As depictable from Table 1, a histological age-at-death assessment was not only capable of refining the gross osteological age categories and the

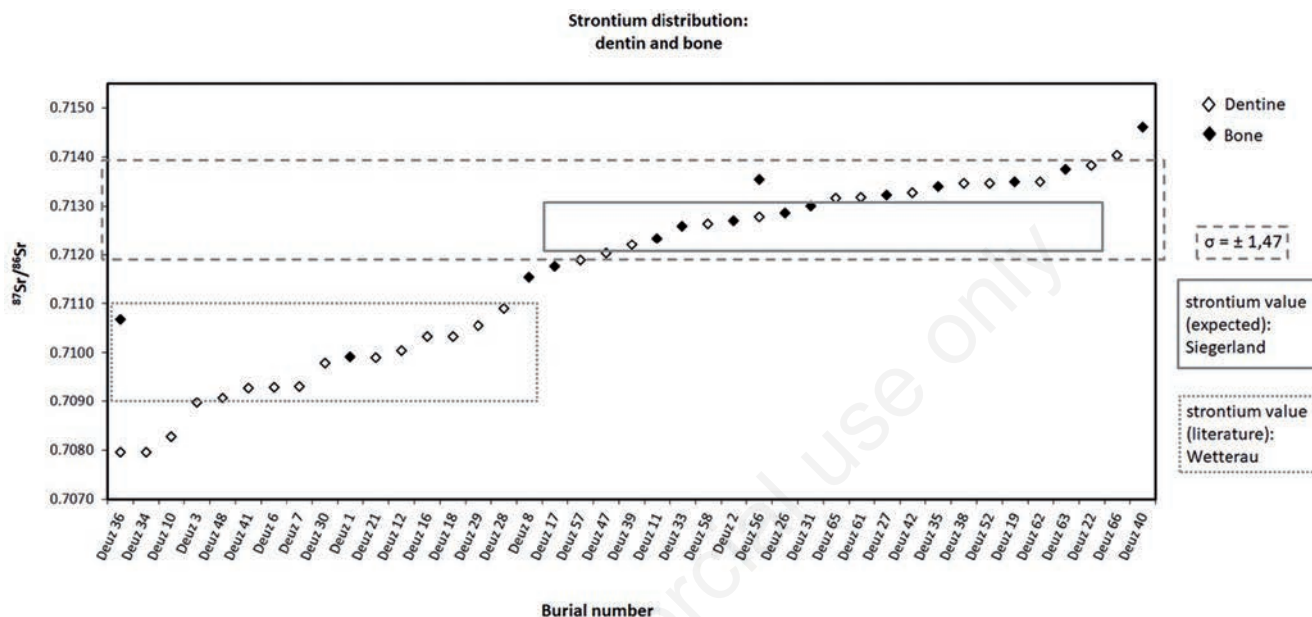


Figure 3. $^{87}\text{Sr}/^{86}\text{Sr}$ isotopic ratios of the cremated finds in ascending order.

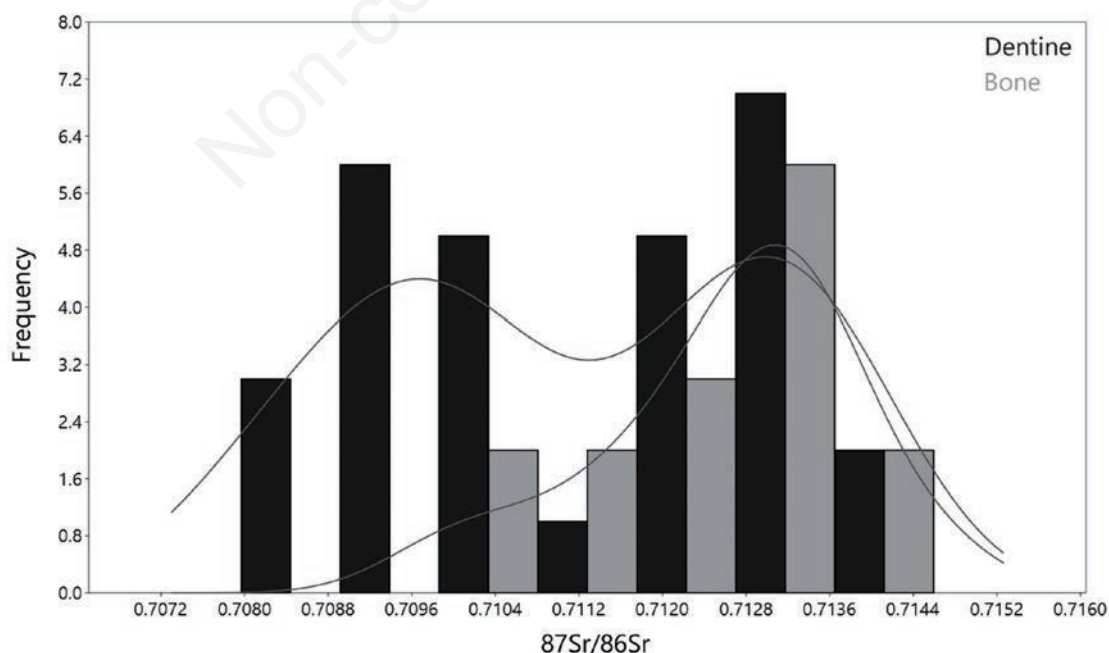


Figure 4. Kernel-Density of the distribution of the isotopic ratios exhibit two peaks.

identification of more juveniles and old-aged individuals, but permitted an age-at-death estimate for all 60 individuals.

$^{87}\text{Sr}/^{86}\text{Sr}$ isotopic ratios varied considerably between 0.70797 and 0.7146 (difference 0.00663; Table 1) and are sorted by ascending order in Figure 3. Visually, two plateaus emerge from the diagram that are confirmed by the Kernel-Density distribution of the isotopic ratios (Figure 4). The first one includes isotopic ratios between 0.7098 and 0.7118, which are in total agreement with published data from the Wetterau in Hesse, a possible region of origin according to the archaeological context ($^{87}\text{Sr}/^{86}\text{Sr}=0.709\text{-}0.711$) (Bentley and Knipper, 2005; Knipper *et al.*, 2014). The second plateau comprises isotopic ratios between 0.7122 and 0.7136. Entered into the Isoplot software with a standard deviation of 1.47, the majority of the isotopic data vary between 0.71186 and 0.71390, matching the expected isotopic data for Devonian bedrock that dominates the region of the burial site. Special emphasis is on the individuals Deuz 36 and Deuz 56, where both bone and dentin were analyzed. While the strontium isotopic ratios from Deuz 56 differ by 0.00077 ($^{87}\text{Sr}/^{86}\text{Sr}_{\text{Bone}} = 0.71354$; $^{87}\text{Sr}/^{86}\text{Sr}_{\text{Dentin}} = 0.71277$) only, the values of burial 36 differ significantly by 0.00272 ($^{87}\text{Sr}/^{86}\text{Sr}_{\text{Bone}} = 0.71069$; $^{87}\text{Sr}/^{86}\text{Sr}_{\text{Dentin}} = 0.70797$). According to the dentine isotopic ratios, this individual and two others (Deuz 34 and 10) spent their childhood neither in the region of burial, nor in the Wetterau.

Discussion and Conclusions

Basically, the same criteria for a morphological age determination are applied to uncremated and cremated skeletons. A high degree of fragmentation and poor representation of the skeletal parts in cremations however poses specific problems because diagnostic anatomical parts are frequently missing. Therefore, about one third of the cremations could not be classified at all osteologically and another third only into gross categories only. By application of histological criteria, all individuals could be aged. The majority had died of young and old adult age, but several individuals that died aged 60 years or even beyond were now detectable. This rises the average life expectancy at the site which is yet insufficiently known for the Iron Age as a whole (Schaal *et al.*, 2015) Whether the lack of children is due to an early deterioration of the fragile bones (Bello *et al.*, 2006) or whether these subadults were buried separated from the adults cannot be assessed by hindsight.

The strontium isotope analysis confirms the hypothesis of a specific migration pattern of some individuals to their place of death. No data on bioavailable $^{87}\text{Sr}/^{86}\text{Sr}$ isotopic ratios in the Siegerland are available yet, therefore, there is no proof that individuals with isotopic signatures between 0.71186 and 0.71390 were native to the site. Although these data are in agreement with the geological background, the influence of diet on a consumers' strontium isotopic ratio can be considerable. Frequently, the majority of individuals on a burial site that share similar $^{87}\text{Sr}/^{86}\text{Sr}$ isotopic ratios are considered local (*e.g.* Bentley *et al.*, 2002) what is at the same time the most parsimonious interpretation (*Occam's razor*; Beckmann, 1998). $^{87}\text{Sr}/^{86}\text{Sr}$ isotopic ratios in archaeological skeletons from the Wetterau region in today's Hesse have been reported by Bentley and Knipper (2005) and Knipper *et al.* (2014) and are in total agreement with the respective such ratios (0.709-0.711) measured in 14 cremations, almost one fourth of all individuals (Figure 3). It should be emphasized that due to the rather slow bone turnover (Martin *et al.*, 1998), Sr in bone accumulates in the course of many years in the adult. Individuals exhibiting a significantly different bone $^{87}\text{Sr}/^{86}\text{Sr}$ isotopic ratios than the soil at the site of recovery therefore must have been late immigrants to the site. The actual number of immigrated people is therefore even underestimated. The isotopic analyses confirms the hypothesis of the most probable place of origin of these non-locals that is based on the archaeological context and finds, and at the same time confirms the considerable *pull factor* of the early mining industry in the Siegerland.

How powerful this pull factor should have been is demonstrated by the case of the male Deuz 36. He had died aged 60 or even beyond, and had a dentine $^{87}\text{Sr}/^{86}\text{Sr}$ isotopic ratio of 0.70797. Therefore, he was most probably not native to the Wetterau either. A plausible explanation could be that before he died and was recovered at his place of burial, he had spent several years of his life in a region that matches the isotopic signatures of the Wetterau, according to his significantly different bone isotopic signature. Alternatively, this individual could have migrated directly to the Siegerland some years before his death without even passing the Wetterau, and the bone isotopic ratio is a mixture that plots among the Wetterau samples simply by chance. Since burial 36 belongs to early phases of the site and grave goods exhibit relations to Northern Hesse (Zeiler *et al.*, 2017), the first interpretation seems to be the more plausible one.

This male, and the individual Deuz 1

that had also died at a quite old age, were obviously such late immigrants to the sites because their bone isotopic composition was not adjusted to the local values (Figure 3). It is noteworthy that none of the immigrants to the site were dated younger than the Hallstatt Period. In contrast, three of oldest burials on the necropole (nos. 1, 29, and 48) were immigrants as well. To date, these cremations are the oldest evidence for a first population of the Siegerland in the course of the Iron Age and it seems as if a previously rather deserted area became populated. Our results suggest that overall mobility in the Iron Age could have been higher as previously suggested, a fact that could better explain the archaeologically visible culture transfer at this time.

Because of the usual high fragmentation and poor representation of a skeleton after cremation, osteological analysis is often tedious and cremations are frequently put in second place by both archaeologists and anthropologists. In Central Europe, cremating the dead was the major if not exclusive burial rite from approximately 1300 BC until 400 AD, a time spanning about 1500 years (Grube *et al.*, 2015). This is an intolerable gap in terms of a major scientific goal in physical anthropology, namely the reconstruction of population dynamics and their causes in time and space. Although provenance analysis by a single stable isotopic ratio only has its limits without doubt, more efforts – indispensably including microanatomy – should be dedicated to cremated finds.

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