PATTERNS OF CHEMICAL CHANGE IN FOSSIL BONES AND VARIOUS STATES OF BONE PRESERVATION ASSOCIATED WITH SOIL CONDITIONS

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Summary

The preservation of human and animal bone from four prehistoric sites of different age was investigated in relation to soil conditions. The surface, density, hardness, and organic content of the bones are correlated and can thus serve to describe the state of preservation of the bone. Correlations between osseous deterioration, soil acidity (as measured by pH) and the calcium content of the soil were found to be significant. Elements associated with soil contamination (iron, aluminum, manganese) are found in significantly higher proportions in poorly preserved bones.

Analysis of trace elements from the bone mineral used for diet reconstruction in the same bone samples showed that various amounts of magnesium are lost through leaching. Therefore, magnesium is excluded from use for diet reconstruction. The zinc concentrations are not altered. The elements barium and strontium appear not to be so sensitive to diagenesis. Zinc, barium, and strontium may serve as useful prehistoric dietary indicators.

Résumé

Modification chimique des os fossiles et variation des stades de préservation osseuse en relation avec les conditions du sol.

L'état de conservation d'os humains et animaux provenant de quatre sites préhistoriques d'âge différent a été analysé en relation avec les caractéristiques du sol. La surface, la densité, la dureté et le contenu organique des os sont en corrélation et peuvent ainsi servir à la description de l'état de conservation des os préhistoriques. Les corrélations entre la dégradation osseuse, l'acidité du sol (mesurée par pH) et la teneur en calcium du sol sont aussi significatives. Les éléments fer, aluminium et manganèse, qui indiquent une contamination des os par le sol, se trouvent en concentrations très significatives dans des os mal conservés.

L'analyse d'éléments-traces dans la phase minérale des os, employée pour la reconstitution de l'alimentation préhistorique, a montré que du magnésium est lessivé en proportion différente du tissu osseux, dépendant de la condition de conservation des os. C'est la raison pour laquelle cet élément est exclu pour la reconstitution alimentaire. Les concentrations de zinc ne sont pas altérées. Le baryum et le strontium ne semblent pas être sensibles aux processus diagénétiques. Zinc, baryum et strontium peuvent donc servir d'indicateurs de l'alimentation préhistorique.

Zusammenfassung

Chemische Veränderungen und verschiedene Erhaltungszustände von Knochen in Abhängigkeit von den Bodenbedingungen.

Die Erhaltung von Menschen- und Tierknochen aus vier prähistorischen Fundstellen unterschiedlicher Zeitstellung wurde in Abhängigkeit von den Eigenschaften des Hüllsediments untersucht. Die Messungen zeigten, daß Oberflächenbeschaffenheit, Dichte, Härte und organischer Gehalt der Knochen miteinander korrelieren und diese Eigenschaften sinnvoll zur Beschreibung des Erhaltungszustandes archäologischer Knochen dienen können. Ebenfalls signifikant sind Korrelationen zwischen dem Abbau des Knochengewebes, der Bodenacidität (gemessen als pH) und dem Calciumgehalt des Hüllsediments. Die Elemente Eisen, Alumunium und Mangan, die eine Kontamination von Knochen durch das Hüllsediment anzeigen, wurden in signifikant höheren Konzentrationen in schlecht erhaltenen Knochen gefunden.

Die Analyse von Spurenelementen im Knochenmineral, die zur Rekonstruktion prähistorischer Nahrung verwendet werden, zeigte, daß Magnesium abhängig von der Knochenerhaltung unterschiedlich stark aus dem Knochengewebe ausgewaschen wird. Dieses Element sollte deshalb für Ernährungsrekonstruktionen nicht hinzugezogen werden. Die Zinkkonzentrationen zeigten keine Veränderungen durch die Bodenlagerung. Auch Barium und Strontium scheinen nicht sehr sensibel auf diagenetische Vorgänge zu reagieren, weshalb sie neben Zink als sinnvolle Indikatoren prähistorischer Nahrung dienen können.

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Key Words

Animal and human bone, Soil, Preservation, Diagenesis, Major, minor and trace element analysis.

Mots clés

Os animal et humain, Sol, Conservation, Diagenèse, Analyse d'éléments principaux, d'eléments rares et d'éléments-traces.

Schlüsselworte

Tier- und Menschenknochen, Sediment, Erhaltung, Diagenese, Haupt-, Nebenbestand- und Spurenelementanalyse.

Introduction

The chemical composition of human and faunal bone excavated from archaeological sites can provide archaeologists, archaeozoologists, and anthropologists with a wide range of information; for instance, radiocarbon dating and diet and climate reconstruction. However, post-mortem contamination from the surrounding soil matrix complicates many of these applications. The preservation of bones on archaeological sites varies considerably according to soil conditions. In general, bone is preserved well in soils with neutral or slightly alkaline pH and poorly in acidic soils. Although an empirically demonstrable relationship between bone preservation and pH should surprise no one, few if any researchers have attempted to quantify this for predictive purposes or as a basis for the above mentioned investigations. Within the scope of a Master Thesis (Stephan, 1992), the bone preservation depending on the conditions of the associated soil was investigated and the suitability of certain trace elements in bone for dietary reconstructions tested. The exercise is part of a wider attempt to understand the relationship between preservation and burial environment in detail and works towards a quantification of that relationship by using statistical methods and comparing physical properties and major and trace elements of archaeological bone and the surrounding soil.

Sampling sites and strategy

For the investigations bone and soil samples were obtained from four different sites. The oldest one, Stuttgart-Bad Cannstatt "Bunker"/ Southwest Germany (Wagner, 1984), is dated to the Mindel-Riss-interglacial period at about 200,000 years b.p. The samples of the second site, Geißenklösterle/Southwest Germany (Hahn *et al.*, 1985; Hahn, 1988), originate from Aurignacian and Gravettian layers, which are dated between 30,000 and 36,000 years bp and to about 23,000 years bp. The third site, Moringen/Großenrode II/North Germany (Heege and Uldin, 1991), is a Neolithic collective burial dated to about 3000 years bc From the youngest site, Troy/Northwest Turkey (Korfmann, 1991), samples were obtained from Troy I layers (Early Bronze Age 3000-2800 bc), Troy II-V layers (Middle Bronze Age from the middle of the 3rd millennium to the middle of the 2nd millennium bc) and from Hellenistic and Roman remains of the lower city (Troy VII-IX, from 800/700 bc to about the 4th century ad). Except the bone samples from Großenrode II, which originate from human skeletons, all other samples are animal bones.

Material

In all sites the bone samples were recovered together with the adhering soil. In Troy the Hellenistic-Roman bone samples consist of long bones from cattle, which were not in situ but which contain sufficient amounts of soil in the medulary cavity. To have sufficient bone material for the investigations and to avoid interferences, only bone samples that were more than 5 cm long and unaltered by fire were chosen. Besides this, only bone diaphysis from subadult or adult individuals were used. This choice was based on severel reasons. First, cancellous bone is much more difficult to clean. Second, bones from infantile or juvenile individuals are more porous and therefore more influenced during the deposition in the soil. Third, there are only slight variations of the trace element concentration in the diaphysis and the element concentration of the cortical bone gives a good representation of the element concentration of the whole skeleton (Herrmann *et al.*, 1990: 235). For the evaluation, it was necessary to compare the results of the archaeological bones with those of recent unaltered bone. Because of the few data of recent bone in literature, fresh long bones from cattle and pig from Tübingen/Southwest Germany were investigated in the same manner as the archaeological material.

Analytical methods

Bones

After the archaeozoological or anthropological⁽¹⁾ determination, the colour of the bone surface and of the

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Table 1: State of preservation categories.

Category	State of preservation
1	**very poor preservation**; fragile bone; original bone surface completely destroyed and cracked
2	**poor preservation**; original bone surface mainly destroyed; surface with a lot of small hollows and fissures
3	**medium preservation**; original bone surface at several places destroyed
4	**good preservation**; surface mainly intact; only few and shallow destructions
5	**very good preservation**; strong bone; surface intact; no evidence of postmortem destruction of osseous material

inner cortical bone tissue was determined using Munsell Soil Color Charts. Then the outer and inner surfaces and the edges of the bone samples were viewed with a stereo microscope. On the basis of this description, the specimens were scored for preservation according to the five categories shown in Table 1. The main reason for the classification was the preservation of the upper cortical layers(2).

After this classification, density (kg/dm^3) and hardness (N/mm^2) were measured(3) and subsamples taken, which include a representative portion of the cross-section, i.e., the periosteal through endosteal portions. Therefore, all subsequently measured element concentrations are mean values of the concentrations of coloured and of noncoloured regions of the bone tissue. The organic matter was determined from weight loss between 100° C and 500° C. In the remaining bone ash, i.e., the inorganic bone phase, mainly consisting of hydroxyapatite, major and trace elements were determined. The calcium (Ca) content was analysed titrimetically, the phosphorus (P) concentration colorimetrically. The trace elements aluminium (Al), barium (Ba), iron (Fe), magnesium (Mg), manganese (Mn), strontium (Sr) and zinc (Zn) were determined by atomic absorption spectrophotometry.

Soil

For all soil investigation the mixed fine fraction < 2 mm was used. The amount of organic matter and the P content were determined using the methods for bones described above. The total element composition was measured by X-ray fluorescence spectrometry. For the evaluation, the elements measured in the bones with the addition of silicon (Si) as a major constituent of soil were chosen.

Statistics

If the samples sizes were sufficient the metrical data within one site and of different sites were compared by using the non-parametric Wilcoxon significance tests. Correlation (PC statistic shareware) and factor analysis (SPSS/PC+) were carried out to refine the relationships of bone properties and between bone properties and soil conditions. More details of the experimental procedure were described elsewhere (Stephan, 1992: 20-37).

Results and discussion

The physical properties, density and hardness, show strong relationships with the organic content of the bones, i.e., the lower the organic content the lower density and hardness of the bone tissue (tab. 2). For the preservation categories correlations cannot be calculated because they are nominal data. However, a 'normal' comparison of the states of preservation with the bone properties mentioned above shows the poorer the visual estimation of the preservation, the stronger the decomposition of the organic phase and the lower the density and hardness.

In addition to this, the properties of the surrounding soil are determinative for the bone preservation. The correlation coefficients in table 2 indicate the higher the pH, water content, and Ca concentrations and the lower Si concentrations of the soil, the harder the bones. The pH values and Ca concentrations have the strongest influence(4). This is because in an alkaline environment the decomposition of the collagen is slower and the main constituents of the bone mineral are harder to dissolve than in acidic soil. Qualita-

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(2) The categories were not based on fragmentation because animal bone samples were as normally for archaeological animal remains, mostly fragments of complete skeletal elements.

(3) For the hardness the ball-thrust hardness (German DIN-Norm No. 53456) was chosen. The hardness of the outer bone surface was measured perpendicular to the collagen fibers.

(4) Like the positive correlation coefficient of $r = 0.6293^*$ shows soil pH and Ca content have a strong relationship caused by the dependence of the pH values on the Ca content and the solubility of Ca compounds in the soil (Schachtschabel *et al.*, 1989: 14, 108, 116, 117). Besides this relationship the concentrations of Ca and Si in the soil show a strong negative correlation ($r = -0.9830$). The reasons are the composition of the original rock and leaching of calcium compounds during the weathering carbonate rocks.

Table 2: Correlation matrix of physical properties and Al, Fe and Mn concentrations of the archaeological bone samples and soil conditions.

	Correlation coefficient (r)		
	Density	Hardness	Organic content
bones: N = 26			
Density	1.0000		
Hardness	+.4660*	1.0000	
Organic content	+.3439+	+.7886*	1.0000
Al	-.1991	-.6415+	-.5741*
Fe	+.3139	-.0486	-.5708*
Mn	+.5476*	+.2641	+.2126
Mg	-.1573	+.4123+	+.5241*
soil: N = 18			
pH	-.0759	+.5561*	
water content	+.5921*	+.6998*	
Si	-.2273	-.6746*	
Ca	+.1505	+.5505*	
P	+.5609*	+.6512*	
+: $p > 0.05$; *: $p < .01$			

tive relationships between the state of bone preservation and the pH of the soil were shown by different authors. Correlation and regression analysis were carried out by Gordon and Buikstra (1981) who showed a more or less strong relationship between bone preservation and soil pH depending on the individual bone age.

Figure 1 shows the data of each site. The bones from Stuttgart-Bad Canntatt are poorly preserved(5) and the organic bone component is almost completely decomposed, although the soil pH of 7.7 is high and the soil contains a high concentration of Ca(6). Therefore the soil conditions can´t be the only reason for the poor bone preservation. Additionally, the quite long time of 200,000 years the bones were interred seems to be relevant. The Geißenklösterle bone samples are well preserved, based on the slightly alkaline soil (pH 7.7) and its high Ca concentration. Quite astonishing is the better preservation of the older Aurignacian bones than the younger Gravettian bones. The older bones were classified in a better state of preservation and are twice as hard. The reasons seem to be the significant differences between the soils. The soil of the Aurignacian layers is wetter than the Gravettian soil because of climatic differences, which were reported by Campen (1990).

The preservation of the bones from Großenrode is poor. The hardness is about 10% of the hardness of recent bones and about only 50% of the organic content of the bones is preserved. The reason is the slight acidic, Ca poor and Si rich (35%) soil which developed from Si rich loess.

The state of preservation of the Troy bones is according to the alkaline, Ca rich soil quite good. The younger Hellenistic-Roman bones are not as well preserved, less hard and contain less organic matter than the older Bronze Age samples. This is caused by differences in soil conditions. The Hellenistic-Roman soil samples originate from the medulary cavity and from humic areas. Contrary to this, the Bronze Age soil samples consist of settlement debris with a lot of mud brick material and contain more water and organic material than the younger soil.

More information about the bone preservation contain the Al, Fe and Mn concentrations (tab. 2). The Al contamination of the bone tissue increases with increasing decom-

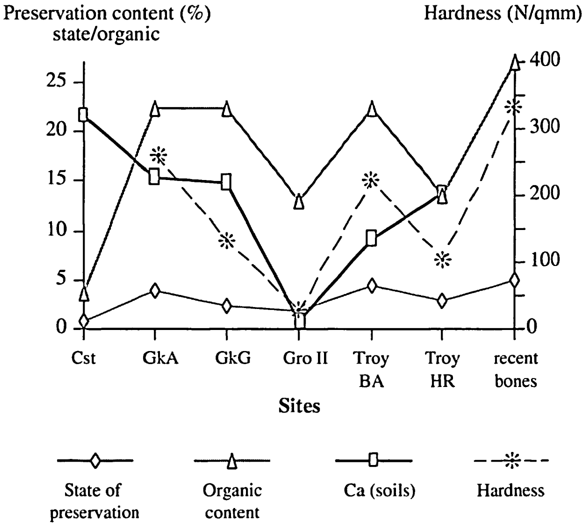

Fig. 1: Mechanical and chemical properties of bone samples (N = 43) and soil conditions (N = 26). Abbreviations of the sites: Cst: Stuttgart-Bad Cannstatt "Bunker"; GkA: Geißenklösterle Aurignacian; GkG: Geißenklösterle Gravettian; Gro II: Moringen/Großenrode II; Troy BA: Troy Bronze Age; Troy HR: Troy Hellenistic to Roman.

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(5) Because of this very poor preservation, measurements of the density and hardness were not possible.

(6) This soil composition is caused by the sediment development on calcareous tuff which consists mainly of calcium carbonate.

position of the bone cortex and decreasing bone hardness and organic content - and according to the correlations between bone preservation and soil pH - with increasing soil acidity (tab. 3). The probable reason is the higher solubility of Al compounds in acidic soils (Schachtschabel *et al.*, 1989: 42-44). The Al contamination of bone tissue appears to be unrelated to the Al content of the surrounding soil, because correlations between Al in the bone and soil samples are missing. According to these results, the poorly preserved Cannstatt, Großenrode and younger Gravettian Geißenklösterle bones contain higher amounts of Al than the Troy and the Aurignacian Geißenklösterle samples and recent bones (fig. 2). The Großenrode bone samples contain quite more Al than Cannstatt and Geißenklösterle Gravettian because of the more acidic surrounding soil.

The Fe content in archaeological bone depends not so much on the bone preservation, although it is negatively correlated with the organic content of the bones (tab. 2). The extent of the Fe invasion depends on the Fe concentration in the soil (tab. 3), i.e., the more Fe in the soil, the more Fe invaded into the bone tissue. Large amounts of Fe minerals were able to move from the surrounding soil into the tissue of the Cannstatt bone samples, because of the high destruction of the outer cortical layers and the high Fe concentration in the soil (fig. 2). The heavy destruction of the upper cortical layers and the decomposition of the main part of the organic matter favoured the invasion of Fe into the bone tissue of the Großenrode samples.

The behaviour of Mn is correlated with the bone density, but not so much with the bone hardness and the organic content (tab. 2). Contrary to Fe, its concentration in archaeological bone seems independent from the Mn concentration in the soil (tab. 3). The reason for the high Mn concentrations in bone could be the accumulation and mobilisation of the biophile element Mn by microbial activity associated with the presence of phosphates and organic substances in the soil (Keeley *et al.*, 1977; Schachtschabel *et al.*, 1989: 276-279). According to the high organic content of the soil, the Mn concentrations in the Geißenklösterle bone samples are significantly higher than in the bones from the other sites (fig. 2). The Troy bones are according to their good preservation only slightly contaminated with the three soil associated elements.

Table 3: Correlation matrix of organic content, Al, Fe and Mn of the bone samples and soil conditions.

	Correlation coefficient (r)		
n = 18	bones Al	Fe	Mn
soil			
pH (KCl-solution)	**-.6537***	-.1090	+.2594
Al	-.0216	-.1361	+.1564
Fe	+.0787	**+.4123+**	-.2472
Mn	+.2470	-.0975	-.2879

+: $p < 0.05$; *: $p < 0.01$

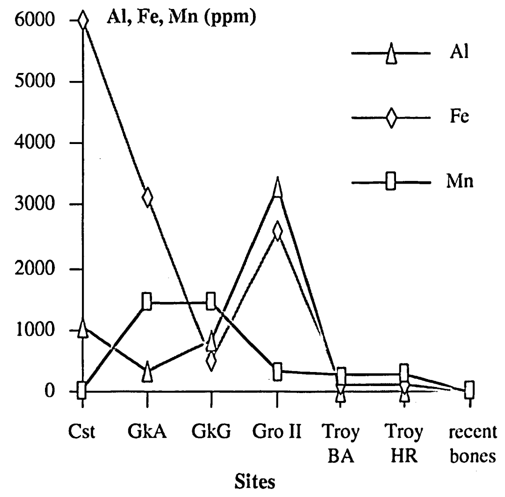

Fig. 2 : Trace elements Al, Fe and Mn in bone samples (N = 43). Abbreviations of the sites: Cst: Stuttgart-Bad Cannstatt "Bunker"; GkA: Geißenklösterle Aurignacian; GkG: Geißenklösterle Gravettian; Gro II: Moringen/ Großenrode II; Troy BA: Troy Bronze Age; Troy HR: Troy Hellenistic to Roman.

From the present study and investigations of Lambert *et al.* (1985) and other authors, it is obvious that Al, Fe, and Mn are in significant higher concentrations in soils than in recent and archaeological bones. These elements are able to invade into interred bones in clay minerals and as oxides and sulfides (Parker and Toots, 1980; Williams and Potts, 1988). Investigations on the trace element distribution indicate a homogeneous distribution of Al, Fe, and Mn within the inner cortical layers and an enrichment at the cortical surface (Lambert *et al.*, 1985; Parker and Toots, 1980). On the bone samples of the present study, different depths of Fe and Mn invasion are visible in the colouring of the bone tissue, which is caused by brown, yellow and red coloured Fe minerals and the black colours of Mn oxides(7). Because the upper cortical layers are coloured, these elements influenced only the upper layers of the Cannstatt, Großenrode and Troy bones. On the other hand, the whole bone tissue of the Geißenklösterle samples is brown, grey and yellow coloured. Therefore, all cortical layers are permeated by Fe and Mn.

These results show that the preservation of all Troy bones is much better and the contamination with Al and Fe much less than that of the Großenrode material, which has the same age as the Troy I samples. Despite the low fragmentation and the short length of time interred, the Troy bones are slightly better preserved and significantly less contaminated with Al, Fe and Mn than the much older Paleolithic Geißenklösterle material. Besides the discussed arguments climatic differences and different mechanical stress could cause differences in the state of bone preservation. Due to their old age the Cannstatt bones are much more poorly preserved than all other samples. The only slightly different Ca and P concentrations of all archaeological bone samples compared to recent bones show that the inorganic part of the bones, the hydroxyapatite, is not very much influenced by diagenesis. Despite the above mentioned decomposition of the organic part, the investigated bone samples should, therefore, offer a good basis for further investigations like diet reconstruction. For diet reconstruction the trace elements Mg, Ba, Sr, and Zn in the hydroxyapatite are used. The investigations of these elements give the following results.

Because correlations are missing, the Zn concentrations in the bone samples seem not to be influenced by diagenesis like Grupe (1986) and other authors report, so that Zn may serve as a useful dietary indicator. It has to be mentioned that Zn is an essential element for mammals and its concentration depends on the general physiological state of the body and diet influences play a minor part.

The significant lower Mg values in all investigated archaeological bone samples show, compared to recent bones, that Mg was leached out of the bone tissue. The positive correlations between Mg, the organic content, and the hardness indicate that the Mg loss increases with the increasing decomposition of the organic bone phase and the decreasing bone hardness (tab. 2 and fig. 3). The Mg leaching is additionally influenced by the soil pH. The positive correlation (r = + 0.5423*) and factor attachment indicate a higher loss at lower pH values. Other authors reported leaching (Parker and Toots, 1980) as well as enrichment (Klepinger *et al.*, 1986) and no diagenetic influences (Lambert *et al.*, 1985, 1989) of Mg in interred bones. However, all reported values are lower than that of recent bones and the range of the data is quite high. Therefore, the reconstruction of prehistoric diet using Mg seems not possible or useful.

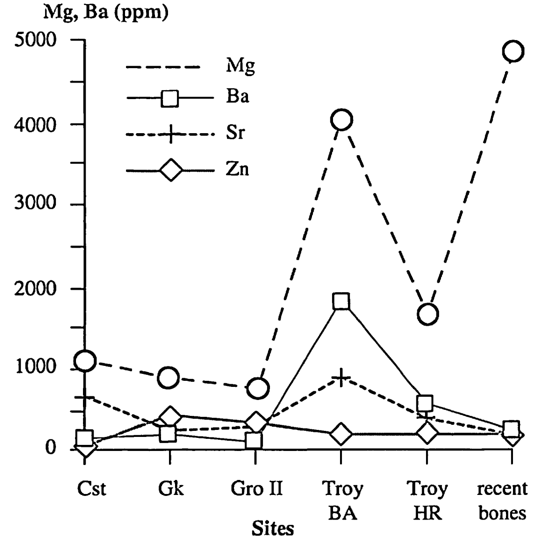

Fig. 3: Trace elements Mg, Ba, Sr and Zn in bone samples (N = 43). Abbreviations of the sites: Cst: Stuttgart-Bad Cannstatt "Bunker"; GkA: Geißenklösterle Aurignacian; GkG: Geißenklösterle Gravettian; Gro II: Moringen/Großenrode II; Troy BA: Troy Bronze Age; Troy HR: Troy Hellenistic to Roman.

For the behaviour of Ba and Sr, the investigations gave no common results. In the Cannstatt, Geißenklösterle and Großenrode bone samples alterations of the Ba and Sr concentrations cannot be detected, i.e., no correlations between them and the physical and chemical bone properties and soil conditions exist. The elements seem not to be influenced by diagenesis, as it is normally suggested, because of their structural relationship to the hydroxyapatite (Grupe, 1986). The Troy bones contain high amounts of Ba and Sr. Remarkable are the high element concentrations in the Bronze Age bones (fig. 3). The reason may be due to high element contents in the diet of the animals how Burton and Price (1990), Grupe (1986), and others have suggested. It is also astonishing that the Ba concen-

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(7) Black, grey and brown colour of interred bone tissue could be caused by humic substances too (Schachtschabel *et al.*, 1989, 219-220). The main colours of the investigated bone samples are, however, reddish and yellowish brown and so they are probably not so much influenced by humic substances.

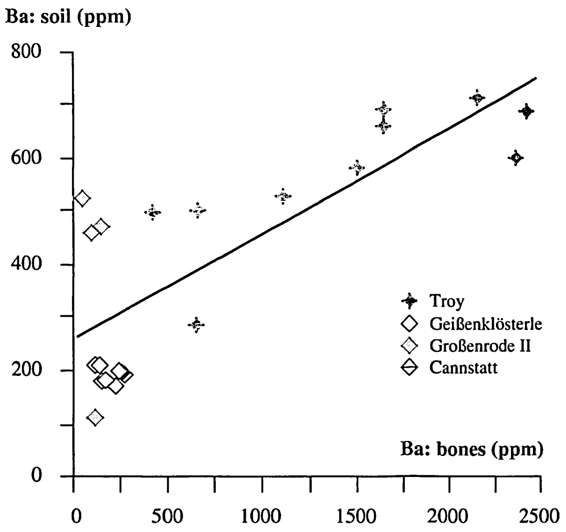

Fig. 4: Correlation diagram of barium in soil and bone samples (N = 26).

r = 0.7829; N = 24; $p < 0{,}0001$; Ba(soil) = 0.1944 (Ba bones) + 254.8; SE = 129,6

trations in these samples exceed the Sr concentrations. The Ba concentration in bone tissue is normally much lower than the Sr concentration, because of the chemical properties, as it is the case for the three other sites. According to the exceeding Ba concentrations, a diagenetic influence of the Bronze Age bone material seems more convincing. The strong correlation shown in figure 4 seems to support this explanation. On the other hand, Ba and Sr concentrations are always much higher in the bones than in the surrounding soil. An invasion of these elements are therefore not obvious, although several authors described contaminations of the upper cortical layers of interred bones with Ba and Sr (Francalacci, 1989; Lambert *et al.*, 1985; Runia, 1987). Based on these investigations it was not possible to decide whether the high Ba concentrations are caused by high Ba amounts in the nutrition or by contamination. This problem requires further investigations(8).

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(8) In all sites, no differences between species were established. Theoretically there has to be differences in the concentrations of the elements used for diet reconstruction. But these could not be mentioned because of the small sample size.

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