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The Last Domicile of the Iceman from Hauslabjoch: A Geochemical Approach Using Sr, C and O Isotopes and Trace Element Signatures

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We present Sr, C and O isotope ratios and quantitative ratios of trace element ratios in the rib and the femur of the Tyrolean Iceman "Ötzi". The results from the rib and the femur are compared with those obtained from pre-industrial historic human skull bone fragments collected in the late 19th century from charnel houses located on different geological backgrounds throughout North and South Tyrol. Comparison of strontium isotope data from the bone fragments locates the Iceman's domicile, during the last 5 to 10 years of his life, to the crystalline part of the Central Alps. Statistical analysis of the trace element ratios, together with oxygen isotope results, suggests that the Iceman probably spent most of his last years in southern elevated part of the Ötztal. However, the results from Iceman only represent those from a single individual and are not a representative average of a group. It cannot therefore be established with absolute certainty that the original trace element signatures were conserved, especially during the first stage of mummification. Our conclusions should therefore be regarded as tentative, but nevertheless the best possible indication his domicile, and a stimulus for further research. © 2001 Academic Press

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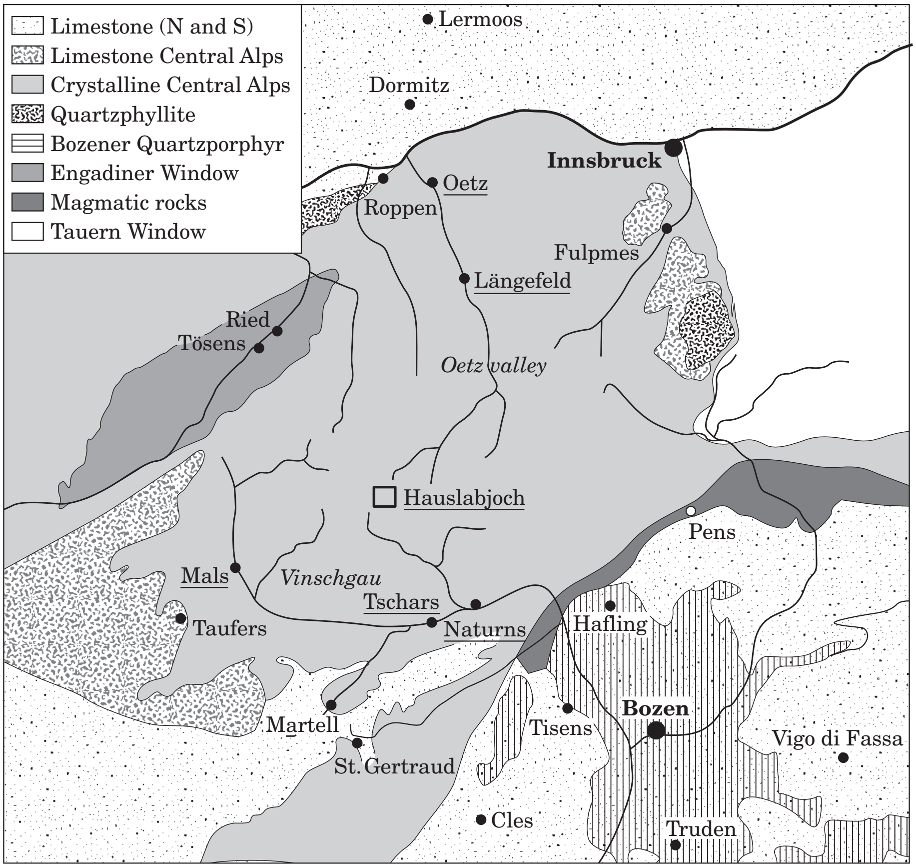

Figure 1. The major geological units in Tyrol and sample locations (except Innsbruck and Bozen, which are shown for geographical reference). Underlined locations are used in the evaluation of trace element ratios.

Introduction

After the initial discovery of the Iceman in September 1991 the age of the mummy (between 3350 and 3100 BC) was not determined, by ^{14}C dating, until 1995 (Prinoth-Forwagner & Niklaus, 1995). Even now, almost ten years later, there is still a great popular interest in tracing the domicile of the Iceman and any other fact that might shed some light on his way of life. In this paper we will show that isotope and trace element geochemistry can contribute to resolving important archaeological questions.

His isolated last resting place at the Hauslabjoch Pass on the Central-European continental divide (see Figure 1) is directly between the Ötztal (a valley in North-Tyrol) and the Vinschgau (a valley in South-Tyrol). Analysis of his bone morphology has established that the Iceman had a similar physiology to his contemporaries. However, his origins could not be located exclusively to either North or South Tyrol (Bernhard, 1995). Analysis of DNA has shown that the Iceman was a Central-North-European (Handt *et al.*, 1994). By analysing the food residue in the Iceman's colon Oeggl (1998) was able to demonstrate convincingly that the Iceman must have spent the spring and early summer of his last months south of his last resting place.

Trace elements and elemental isotope ratios vary between geological boundaries, are transferred from rocks to groundwater, soil and plants, and thus can be incorporated into human tissue (Price, Gruppe & Schröter, 1994). Compact bone of healthy individuals undergoes constant remodelling, therefore the trace element and isotopic composition of bone is representative of the food supply five to ten years before death (Price *et al.*, 1995).

In this paper we attempt to locate the domicile of the Iceman during his last years by using geochemical signatures in bone. Bone material from dating Tyrol from AD 1200 to 1800 was used for comparison purposes.

Bone material from Tyrol

The Alpine valleys of Tyrol are deep and narrow. This not only constrains migration of the indigenous population, as documented by genetic and linguistic analysis (Stenico *et al.*, 1996), but also means that land is at a premium. Consequently, local cemeteries are small and corpses are exhumed after relatively short periods (no more than 100 years) to make room for the newly deceased. Exhumed bones are stored in ossuaries that, because of the nature of mountain air, are ideal for conserving their quality.

The Natural History Museum in Vienna houses a fine collection of skulls from North and South Tyrol. They were collected at the end of the 19th century from various charnel houses or church ossuaries (Tappeiner, 1883). The well-preserved skulls analysed in this study are, on average, 200 to 400 years old (Westerhoff, 1989). The minimum age of 200 years is important as industrialization caused the release of many trace elements into the environment. Thus, more recent trace element signatures may not be representative of geochemical origin (Hoogewerff, 1998).

Sampling

Some authors have discussed the inhomogeneous distribution of trace elements in bone in the different parts of the human body (Sandford, 1993). The Tappeiner collection only contains skulls. Regrettably, but understandably, sampling a matching part from the skull of the Iceman was not possible. One sample from his left rib and one from his femur were taken instead. By focusing solely on isotope and trace element ratios, potential problems caused by inhomogeneous distribution of elements in the bone matrix are largely eliminated.

To obtain a representative sample set from Tyrol at least five samples were taken from each of two villages on every major geological unit in the Tyrol. A slightly higher density of samples was taken in the Vinschgau and the Ötztal. Most ossuaries and cemeteries were visited to obtain soil samples for estimating the risk of local contamination. Local water was sampled to check the relation between the drinking water composition and geology (see Figure 2).

Methods

Extreme care was taken not to contaminate the samples during preparation. Samples were digested in a UV digestion apparatus. Triplicate samples of NIST1400 and NIST1486 standard bone powders were treated in the same manner. Two fragments (about 10 mg) from each Iceman sample were dissolved, without rinsing, in Milli-Q water. Two additional fragments (also about 10 mg) were rinsed with Milli-Q water before dissolving in order to check for contamination during sampling or, in the case of the exposed femur, contamination during removal of the body from the glacier in 1991.

Trace elements were measured on a PE-SCIEX ELAN 5000A ICP-MS. Selected samples were double-checked with Total Reflection X-Ray Fluorescence Analysis (ATOMICA EXTRA IIA). The Sr isotopes from the skulls and the bone material from the Iceman were measured by thermal ionization multi-collector mass spectrometry using two similar instruments: Finnigan MAT 262, NIST987-Sr n=12: $^{87}\mathrm{Sr}/^{86}\mathrm{Sr}=0{\cdot}710245 \pm 10$ 2σ (Vroon, 1992) and Finnigan MAT 262, NIST987-Sr n=6: $^{87}\mathrm{Sr}/^{86}\mathrm{Sr}=0{\cdot}710233 \pm 8$ 2σ (Miller & Thöni, 1995). Carbon and oxygen isotopes were analysed using a Finnigan MAT 251 multicollector gas isotope instrument in combination with the Finnigan Carbo-Kiel system (0·1 mg bone powder per analysis).

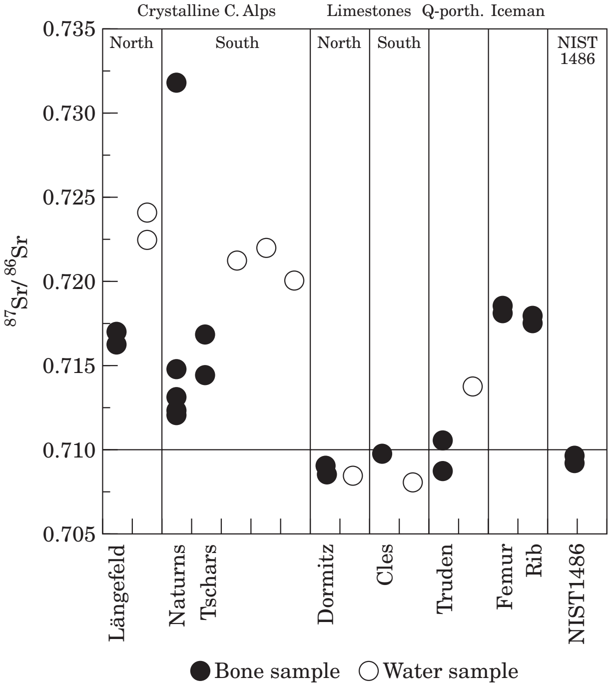

Figure 2. Strontium isotope values in bone and water samples. Note the clear distinction between samples coming from the crystalline Central Alps and the Limestones. Recent stream water results demonstrate the influence of geological background on the isotopic composition.

Results and Discussion

Results obtained on standards are presented in Table 1, to establish the quality of the analysis. The Sr, C and O isotope results are given in Table 2.

Contamination and diagenesis

The use of trace elements to reconstruct past diets or migration patterns is notoriously difficult. The major uncertainties are possible contamination of the bone by the soil or groundwater, or redistribution of elements within the body by microbiological activity (Sandford, 1993; Price, 1989).

Analysis of the skin of the Iceman suggests submersion of the Iceman in (cold) water for several months during which transformation of fat into adipocere was the first stage of mummification, and subsequent desiccation the second stage (Bereuter *et al.*, 1996*a*; Bereuter *et al.*, 1996*b*). Submersion and desiccation

Table 1. Results of standard measurement. NIST1400 Bone Ash and NIST1486 Bone Meal. Trace elements in mg/kg. Reproducibility determined by triplicate analysis of separately digested samples

Element or isotope ratio	NIST1400 Measured	NIST1400 Certified	NIST1486 Measured	NIST1486 Certified	Reproducibility (%) 8 triplicate samples
Ni	17	—	10	—	9
Zn	168	181	132	147	3
Ga	1·2	—	0·7	—	13
As	0·6	0·4	<0·01	0·006	18
Sr	248	249	265	264	4
Mo	0·26	—	0·32	—	9
Cd	0·034	0·030	<0·01	0·003	12
Ba	232	—	285	—	6
Pb	7	9	1·1	1·3	4
Cu	2·2	2·3	0·81	0·8	4
^{87}Sr/^{86}Sr	—	—	0·709303 ± 7 (*N*=2)	—	—
δ^{13}C ‰	− 13·81 (*N*=2)	—	− 7·23 (*N*=2)	—	—
δ^{18}O ‰ PDB	− 11·49 (*N*=2)	—	− 11·70 (*N*=2)	—	—

Table 2. Sr, C and O isotope results of the Iceman samples and selected skull bones. Numbers in parenthesis are results of duplicates

Sample	Location	Geology	^{87}Sr/^{86}Sr Amsterdam	^{87}Sr/^{86}Sr Vienna	δ^{13}C (‰)	δ^{18}O (‰) PDB
Ötzi femur not rinsed	Hauslabjoch	—	0·717654 ± 6	—	—	—
Ötzi femur rinsed	Hauslabjoch	—	0·717970 ± 6	—	− 14·63 (− 14·31)	− 7·06 (− 7·27)
Ötzi rib not rinsed	Hauslabjoch	—	0·718625 ± 7	—	—	—
Ötzi rib rinsed	Hauslabjoch	—	0·718636 ± 6	—	− 13·90 (− 13·83)	− 7·23 (− 7·42)
4554	Langenfeld	Gneiss	0·717110 ± 9	—	− 14·89	− 5·77
4555	Lagenfeld	Gneiss	0·716364 ± 8	—	− 16·19	− 6·52
4361	Naturns	Gneiss	0·713115 ± 6	—	− 11·65	− 4·60
4363	Naturns	Gneiss	0·712344 ± 6	0·712338 ± 7	− 13·19	− 5·11
4364	Naturns	Gneiss	0·714836 ± 6	—	− 13·75	− 8·80
4366	Naturns	Gneiss	0·712076 ± 9	—	− 13·19	− 8·23
4369	Naturns	Gneiss	0·731949 ± 7	0·731899 ± 6	− 12·64	− 5·53
4388	Tschars	Gneiss	0·716810 ± 6	—	− 14·49	− 4·85
4402	Tschars	Gneiss	0·714426 ± 5	—	− 15·13	− 5·80
4617	Truden	Limestone	—	0·708857 ± 5	− 13·83	− 5·30
4489	Dormitz	Limestone	—	0·708995 ± 7	− 12·50	− 3·05
4490	Dormitz	Limestone	—	0·709117 ± 7	− 13·42	− 5·43
4766	Cles	Limestone	—	0·709805 ± 7	− 14·63	− 6·15
4614	Truden	Q porphyr	—	0·710618 ± 6	− 14·90	− 4·60

could both have led to open system behaviour of the corpse. Enrichment or depletion of certain elements in the bone mass could alter trace element abundances, their ratios or their isotopic signal. Analysis of ice found at the Iceman's last resting place exhibits the typically low concentration of trace elements normally found in glacier ice. The Sr concentration in the ice is less than 0·01 ppm whereas the normal concentration in human bone is between 50 and 500 ppm. The ratio between the concentrations in bone and ice is 1000 or more for other elements analysed in this study. Flowing water could enhance exchange with the bone mass, however the "Iceman" was submerged in ice for 5000 years so it is unlikely that large volumes of water have exchanged with the corpse.

In order to preserve the trace element chemistry of the skulls it was important that all the bones were exhumed within less than 100 years after burial (Tappeiner, 1883). Subsequent storage in the dry and, from our observations, well-ventilated ossuaries resulted in good preservation of the trace element chemistry. Of the ten specimens used for histological comparisons, only one (4489) had been attacked by microorganisms, while another (4617) showed some sediment infiltration.

The levels of trace elements in the bone samples studied are comparable to those in fresh human bone and this is a first indication that no extreme bulk contamination occurred during burial. A much lower Pb concentration in the Iceman's bones relative to the Mediaeval skulls can be explained by the technogenic use of Pb since Roman times. This has been documented in historical bone material before (Gruppe, 1991).

The lanthanide elements should also comprise good indicators of open system contamination, either by bulk contamination or by solution exchange, as their concentration is less than 0·2 ppm in live bone (Yoshinaga *et al*., 1995) and is considerably higher in soil (10–150 ppm). Cerium should be an especially good indicator as Ce exchanges readily with Ca in the apatite lattice (Deer, Howie & Suzman, 1983). The unrinsed femur sample has a Ce concentration of 20 ppm, whereas the rinsed fragment contained only 0·4 ppm Ce. The rib sample has a stable Ce concentration around 0·09 ± 0·01 ppm (unrinsed and rinsed fragment). It therefore appears that the rib was well protected from contamination by the surrounding tissue. The skull samples (all except four which contained up to 0·5 ppm Ce) had low (<0·15 ppm) biogenic Ce levels.

The TR-XRF analysis of different 5 mg fragments of the rib gave CaO/P_2O_5 ratios between 1·09 and 1·22 (± 0·01). These values are significantly different from ideal hydroxy-apatite ($CaO/P_2O_5=1{\cdot}32$). This represents a slight under-saturation in Ca, which is a good indication for past biological, rather than recent geological control of apatite composition (Radosevich, 1993).

The Iceman's bone fragments were examined by X-ray diffractometry and clean hydroxy-apatite was observed, possibly containing small amounts of organic tissue. In two of the skull samples small quartz peaks were present, indicating sediment infiltration. Analysis of the soil samples from the different cemeteries and from the location where Ötzi was found did not show any suspiciously extreme compositions.

Identifying the Iceman's domicile during the last five to ten years

The $^{87}Sr/^{86}Sr$ ratio in marine limestone reflects the composition of the seawater where it was deposited and is closely confined to the range 0·707–0·708 (Koepnick, Deniso & Dahl, 1988; Jones *et al*., 1994). The central crystalline Alps have a significantly higher $^{87}Sr/^{86}Sr$ ratio because of higher, sustained Rb/Sr ratios in the continental crust. Stream water samples demonstrate that water chemistry is related to the geology. In Figure 2 the Sr isotopic composition in streams mirrors the difference in $^{87}Sr/^{86}Sr$ ratios between crystalline and limestone source rock.

Strontium chemically resembles Ca and it can be assumed that Sr follows Ca absorption in the food chain (Larsen, 1997). Therefore it is to be expected that the Sr isotopic composition in human bone resembles the $^{87}Sr/^{86}Sr$ of the local geology through absorption from water or from locally grown food. To test this hypothesis we analysed the Sr isotopic composition of nine skulls from the crystalline central Alps, four from the limestone areas and one from the Bozener Quartz Porphyr. The results in Table 2 show the expected differences in $^{87}Sr/^{86}Sr$ from different geological settings. $^{87}Sr/^{86}Sr$ ratios in the range 0·7088–0·7098 can be observed in skulls from the limestone areas and this range of values is slightly higher than those for Jurassic and Cretaceous seawater (Koepnick *et al*., 1988; Jones *et al*., 1994). This can be explained by diagenesis of the limestones (Jones *et al*., 1994) and/or preferential leaching from the soils (Blum & Erel, 1997). As a result, bioavailable Sr may have an $^{87}Sr/^{86}Sr$ ratio that deviates slightly from that of the bulk source rock (effect less than 0·001). In the gneiss area the skull $^{87}Sr/^{86}Sr$ ratios range from 0·7120 to 0·7320. The water samples in the gneiss area show similar values. The one value from the Bozener Quartz Porphyr is in agreement with those expected in people living on volcanic rocks in this area, i.e. an $^{87}Sr/^{86}Sr$ ratio intermediate between mantle and crustal values.

The small but significant difference of $^{87}Sr/^{86}Sr$ between the femur and rib could be due to contamination. As the unrinsed femur has a lower $^{87}Sr/^{86}Sr$ than the rinsed femur bone it can be assumed that the loosely bound Sr represents a contaminant with a lower $^{87}Sr/^{86}Sr$. The fact that both fragments of the rib have almost identical $^{87}Sr/^{86}Sr$ and a higher $^{87}Sr/^{86}Sr$ than the femur suggests that these represent the uncontaminated value best used for comparison. The Iceman clearly spent most of his life in the crystalline areas as his bones (both the rib and the femur) have $^{87}Sr/^{86}Sr$ values well above those found in skulls from the limestone areas.

Different parts of the skeleton may have different turnover rates for Sr and a difference in $^{87}Sr/^{86}Sr$ could therefore be a result of migration. If the second value from the rinsed femur is an uncontaminated value, this suggests the exciting possibility that the slightly different values between the rib and the femur could indicate the Iceman had been travelling.

Although the number of samples is limited, it should be noted that the $^{87}Sr/^{86}Sr$ in the skulls from the Ötztal seem to be slightly higher than in those from the Vinschgau, except for the extreme sample 4369 from Naturns. This sample has also an extreme As/Sr ratio, which might indicate occupational exposure to As from ore refining.

Carbon and oxygen stable isotopes

Carbon and oxygen isotopes were measured on whole bone from all skulls (not all are shown in Table 2) and also from the rib and femur samples. The carbon isotopic composition does not display significant variation, suggesting that the dietary habits of the individuals in this research, including the Iceman, are not very different. Oeggl (1998) also presented evidence that the Iceman had a well-balanced last meal.

In principle, the $\delta^{18}O$ of whole bone is directly related to the $\delta^{18}O$ of drinking water and foodstuffs and is therefore influenced by the elevation of the water source or food (Fricke, Clyde & O'Neil, 1998). Elevated grounds will be replenished from higher

Table 3. Trace element ratios selected for their ability to discriminate between the Ötztal and the Vinschgau. Note that most of the ratios of the rinsed femur are similar to those of the rib. P *values in the last 2 columns demonstrate the high probability that the rib of the Iceman belongs to the Ötztal population of bone ratios*

Ratio	N-Gneiss average ± Std Dev.	S-Gneiss average ± Std Dev.	Rib average of duplicate ± Std Dev.	Femur rinsed	Femur not rinsed	*P* value Ötztal vs rib av.	*P* value Vinschgau vs rib av.
Sr/Ga	125 ± 30	216 ± 90	109 ± 2	102	82	0·65	0·29
Ba/As	44 ± 40	15 ± 17	101 ± 29	43	16	0·19	<0·001
Sr/Ni	8·2 ± 2·2	14 ± 6	8·2 ± 1·3	7·1	3·6	0·99	0·35
Mo/As	1·4 ± 1·4	0·6 ± 0·4	2·4 ± 0·5	0·8	0·7	0·50	<0·001
Cd/As	1·0 ± 1·0	0·15 ± 0·12	0·82 ± 0·18	0·14	0·6	0·91	<0·001
Mo/Ga	0·4 ± 0·2	1·8 ± 2·1	0·39 ± 0·01	0·3	0·5	0·91	0·52
Sr/Zn	0·5 ± 0·2	1·0 ± 0·4	0·19 ± 0·04	0·5	0·3	0·12	0·06
As/Ga	0·5 ± 0·4	2·8 ± 3·2	0·17 ± 0·04	0·4	0·7	0·50	0·45
Ba/Sr	0·10 ± 0·05	0·07 ± 0·03	0·15 ± 0·01	0·16	0·14	0·45	0·04
As/Cu	0·09 ± 0·07	0·3 ± 0·3	0·11 ± 0·01	0·17	0·06	0·83	0·57
Mo/Ni	0·03 ± 0·02	0·12 ± 0·14	0·029 ± 0·006	0·021	0·022	0·91	0·53
As/Ni	0·03 ± 0·02	0·2 ± 0·2	0·013 ± 0·005	0·026	0·030	0·48	0·45
Mo/Sr	0·003 ± 0·002	0·009 ± 0·010	0·0036 ± 0·0002	0·003	0·006	0·97	0·61
Cd/Sr	0·002 ± 0·002	0·001 ± 0·001	0·0012 ± 0·0001	0·0005	0·005	0·62	0·69
Mo/Zn	0·002 ± 0·001	0·010 ± 0·010	0·0007 ± 0·0002	0·0014	0·0016	0·39	0·50
As/Zn	0·002 ± 0·002	0·011 ± 0·011	0·0003 ± 0·0002	0·0018	0·0022	0·33	0·36

altitudes, resulting in lower $\delta^{18}O$ in the groundwater. The southern part of the Ötztal is a highland area whereas the elevation of the Vinschgau valley is much lower. The relatively low $\delta^{18}O$ in Ötzi's bones, compared to the whole of the dataset, could be an indication that Ötzi spent most of his time in the high pastures.

Trace elements

As the Hauslabjoch pass is a major geo-morphological barrier within the crystalline domain and probably also a cultural divide, it is of interest to ascertain on which side of this barrier the Iceman spent most of his last five to ten years. We used the trace element data to obtain this more detailed geographical information. As previously argued, there is no indication of serious contamination of the Iceman's rib or skull samples. Using this assumption, we created a new dataset containing permutations of all possible trace element ratios for those elements where we were confident of the data. A *t*-test comparison of all the trace element ratios in the Northern and Southern Gneiss areas was then conducted. The 16 ratios that yielded *P*-values of less than 0·05 were selected as markers suitable for discriminating between the two areas (see Table 3). We then determined statistically to which of the two populations the Iceman's rib is most likely to belong. The results show that the two *P* value populations were significantly different with a paired *t*-test *P* value of 0·002. This indicated that, if it is assumed that the Iceman came from either the Ötztal or the Vinschgau, there is a 99% chance his last domicile was the Ötztal. More statistical tests, including non-parametric tests averaging the rib and rinsed femur samples individually or together, all give similar results. The worst *P* value found was 0·05.

Our results should be regarded as a strong stimulus for additional research, and not the definitive answer to the question of the Iceman's last domicile. We cannot be 100% certain that absolutely no mobilization of trace elements has occurred. This is particularly the case during the first stage of mummification, when the Iceman was probably submerged in glacier water. Furthermore, the bone composition of the Iceman is representative of only one individual and his bone composition may have been anomalous.

Conclusions

The Sr isotope results show that it is very likely that the Iceman had his domicile in the crystalline area either in the Vinschgau or the Ötztal. The oxygen isotopes and the trace element ratios taken together statistically favour the southern elevated parts of the Ötztal valley as the domicile of the Iceman during the last five to ten years of his life. The combination of the convincing pollen evidence that suggests a prolonged stay in the southern valley during spring (Oeggl, 1998), together with our evidence for a northern domicile suggests that the Iceman had travelled between the Ötztal and the Vinschgau before and spent the winter in the Vinschgau. The reason for the Iceman to be on the Hauslabjoch Pass has been the focus of much debate in both scientific and popular press. Our data suggest that he may have been returning home when he died on the Hauslabjoch Pass at the end of spring.

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