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Identification of activity area signatures in a reconstructed Iron Age house by combining element and lipid analyses of sediments

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ABSTRACT

One promising analytical method used in household archaeology in addition to inorganic (element) geochemical analysis is that of organic (lipid) analysis applied to anthropogenic sediments. We use both methods here to review chemical imprints that might be useful for recognizing space use and identifying daily activities in a reconstructed Iron Age village at Lejre, Denmark. As documented in many previous studies, element analysis enabled separate activity areas to be distinguished, but the results could not be used to identify the specific activities pursued in the areas. A more qualitative identification of activity areas was possible through lipid analysis, however. The carbon chain distribution, studied for Average Chain Length (ACL), Carbon chain Diversity Index (CDI) and Carbon Preference Index (CPI), enabled a similar separation to be achieved as by element analysis, so that the same areas could be discerned in addition to the reference samples. The stable was distinguished by a substantial input of coprostanol and even more so by 24-ethylcoprostanol, indicating a faecal input from herbivores. Trace levels of these markers were also identified at the entrance, where the animals had passed through. The dwelling area, consisting of two adjacent rooms, could be identified by the sterol ratio (cholesterol/[stigmasterol + β-sitosterol + campesterol]). Lipids from an archaeological context have decayed further toward simpler compounds and become more difficult to identify. Some markers have however a better potential for survival. The results emphasize the importance of further studies on ethnoarchaeological material in order to recognize past activities by element analysis. Moreover, the combination of element and lipid analyses provided a tool that enabled all the separate areas to be identified and provided positive identification of the activities concerned in all areas except the smithy.

1. Introduction

The use of soil chemistry in archaeology, in the form of measurements of both inorganic and organic contents, has steadily increased since it was introduced in the early 20th century. From the time of the pioneering work by Olof Arrhenius, in which he showed the relationship between enhanced phosphate and archaeological remains (Arrhenius, 1935), most analyses have focused on bulk variables such as total organic content (TOC) and total phosphate. Since Lutz (1951) and Cook and Heizer (1962, 1965) noted that a number of elements were enriched in anthropogenic soils, element analysis has been repeatedly used, and is now a well-established technique in archaeology (see e.g. Davies et al., 1988; Entwistle et al., 2007; Holliday and Gartner, 2007; Linderholm, 2007; Middleton, 2004; Sterckeman et al., 2006; Wells, 2004; Wilson et al., 2008). Multi-element soil analysis has been used as a means of site prospecting (e.g. Aston et al., 1998; Bintliff et al., 1990; Schlezinger and Howes, 2000), and a number of studies have concentrated on determining which elements accumulate as a consequence of human activities (e.g. Davies et al., 1988; Entwistle, 2000; Konrad et al., 1983; Linderholm and Lundberg, 1994). Other studies have been intended for use as aids to interpret space use and activities within and around archaeological structures (e.g. Cook et al., 2006; Griffith, 1981; Hjulström and Isaksson, 2007; Knudson et al., 2004; Middleton and Price, 1996; Parnell et al., 2002; Sullivan and Kealhofer, 2004; Terry et al., 2004; Wells et al., 2000; Wells, 2004) or have tried to connect the element differentiation with specific uses and origins (e.g. Barba, 2007; Fernandez et al., 2002; Isaksson et al., 2000; Middleton and Price, 1996; Parnell et al., 2002). The relationships between specific activities and the elemental signals they produce are not fully understood and need to be better established although it has been possible to identify the activity concerned sometimes (Middleton, 2004). In contrast to these methods, lipid analysis is more time-consuming but has the potential to provide a higher diagnostic means of answering specific questions related to subtle differences in soil organic matter (SOM) engendered by various overlying vegetation changes or human activities (see van Bergen et al., 1997;

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Bull et al., 1999). A notable increase has taken place in the analysis of organic material, mainly the lipid content, in the last decade. Numerous studies have been made of vegetation, diagenesis and various diagnostic markers (e.g. Allard, 2006; Andersson and Nilsson, 2001; Ekschmitt et al., 2005; Feng and Simpson, 2007; Fernandez et al., 1997; Howard et al., 1998; Jandl et al., 2005; Jansen et al., 2006; Kiikkila et al., 2006; Kögel-Knabner, 2002; Muri et al., 2004; Naafs et al., 2004; Nierop et al., 2001; Otto et al., 2005; Poirier et al., 2005; Quenea et al., 2004; Rogge et al., 2006; Ruess et al., 2002; Wiesenberg et al., 2004). Several works have also focused on problems that are more applicable to archaeology (e.g. van Bergen et al., 1997; Bethell et al., 1994; Bull et al., 1999, 2002; Evershed et al., 1997; Hjulstrom et al., 2006; Isaksson, 1998; Simpson et al., 1998). Although the combination of element analysis and lipid analysis has proved fruitful (Isaksson et al., 2000), they are seldom applied to the same material. We set out here to combine these methods in order to evaluate which activities are identified by which method and when a combination of the two is to be preferred. The material was from a reconstructed Iron Age house and village at the Lejre Experimental Centre outside Roskilde, Denmark.

2. Material and sampling

2.1. The Lethra Iron Age village at the Lejre Experimental Centre

The aim at the Lejre Experimental Centre is that all work concerning the reconstructed Iron Age village of Lethra should be as authentic as possible with regard to both building techniques and the use of the houses. The houses are inhabited from late spring to early autumn each year. The samples used here were collected from one of the reconstructed multifunctional dwelling houses, a smithy and a clay pit. The degree of authenticity in the actual activities pursued is not of interest for the present purpose, but rather it is most important that the house has been inhabited and that no modern material or chemicals have been used when constructing or maintaining it, and also that the activities in the house have been documented.

As seen in Fig. 1, the house is divided into a dwelling area, a dwelling/cooking area with a hearth, an entrance area and a stable. The only separating wall is between the entrance and the stable. The floor consists of clay tempered with grass and hay. The stable has generally been used to store personal belongings rather than to accommodate animals, but 2 goats, 2 oxen and 2 horses were stabled there in the winters of 1998 and 1999 during an experiment concerned with indoor temperatures (Severine Beck et al., 2007). Three reference samples for studying the clay floor were collected from a clay pit near the house, and an additional six samples were collected from a smithy located some 100 m further away. The clay pits that were used when the house was built have been refilled, but the clay pit that was actually sampled is located nearby. The clay mined from the clay pit had been used for various purposes, such as ceramics and for repairing of the floors. The smithy had been built quite recently and had only been used a couple of times when the samples were collected. The smithy can be divided into two areas, as seen in Fig. 1. Most of the smiths' work has taken place in the inner room on the left, where the anvil and bellows are located.

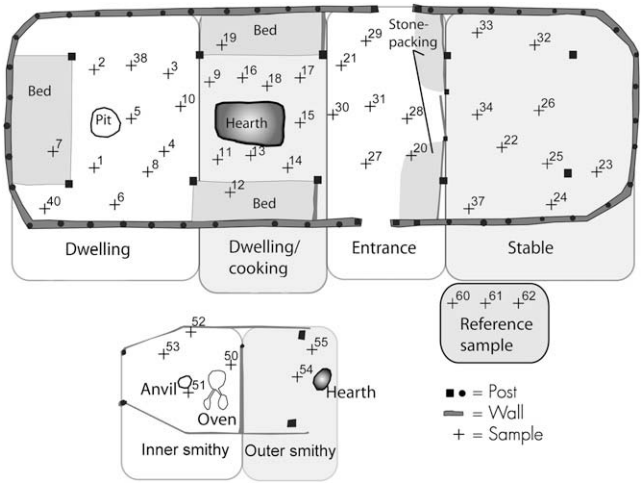

Fig. 1. Plans of the three-aisled multifunctional house and the smithy, with activity areas and sampling points. The smithy is located some 100 m from the house, and the reference samples were collected at a clay pit about 30 m from the house. The size of the multifunctional house is about 14 × 5 m.

2.2. Collection and preparation of samples

When sampling with a probe it is difficult to recognize differences in soil composition and to distinguish subtle layers and natural differences from pedogenic differences. Hence it is of great importance to identify the layer that is to be analysed during an excavation and to observe and document such differences when they appear. The present samples were collected from the top part of the floor down to a depth of ca. 2 cm, and only loose soil on the surface was removed before sampling. The characteristics of the soils from the different areas are shown in Table 1. The reference samples from the clay pit were taken from clean surfaces on the side of the pit. Although the samples were collected from the clay floor, there were considerable intrusions of hay in the samples from the stable. The soil from the smithy was somewhat darker, with a higher proportion of silt and sand. All the samples were air-dried, ground in a mortar and sieved through a 1.8 mm analytical sieve. Visible organic material such as roots cannot be assumed to be of relevance in an archaeological material and the signature of charcoal would obscure the soil composition. The frequency of charcoal is however of interest since the retention of certain elements has shown to be linked with the presence of charcoal (e.g. Davidson et al., 2007). Visible organic materials were hence documented and removed.

3. Methodology

The complex nature of the soil chemistry is nicely encapsulated by McBride (1994): "Much of soil science is empirical rather than theoretical in practice. This fact is a result of the extreme complexity and heterogeneity of soils, which are impossible to fully describe or quantify by simple chemical or physical models". Regarding the interpretation of archaeologically interesting soils

Table 1
Soil characteristics

Area	Soil	Color	Munsell
Dwelling	Gravelly clay	Brownish yellow	10YR6/6
Dwelling/cooking	Gravelly clay	Brownish yellow	10YR6/6
Entrance	Gravelly clay	Brownish yellow	10YR6/6
Stable	Coarse gravelly clay with intrusion of organic material	Brown	10YR5/3
Inner smithy	Coarse gravelly sand mixed with coarse gravelly clay	Dark gray & Brownish yellow	10YR4/1 & 10YR6/6
Outer smithy	Coarse gravelly clay I	Dark grayish brown	10YR4/2
Reference sample	Gravelly clay	Brownish yellow	10YR6/6

we can add the problem of the complexity of site use history and the effects of post-depositional soil processes. Several of the main influences are well documented, however (see Davies, 1980; Engel and Macko, 1993). Anthropogenic sediment is formed through interactions between a variety of human and natural factors. By virtue of their formative roles, these factors leave detectable traces in the sediment, including chemical residues, which can be used to identify activity areas and the activities that occurred on the sediment and contributed to its formation. It is probable that the bulk of the material that affects the soil chemistry of a household-related sediment will be derived from organic matter. Moreover, the type of organic admixture will influence the forms of soil P through biological and chemical cycling, as must also be the case for other elements (Buehler et al., 2002).

3.1. Establishing the geochemical background

One decisive step in order to be able to study the degree to which a soil has been affected by human modification is to establish its geochemical background. This is a problem that has been addressed not only in archaeology but also more generally in the environmental sciences and exploration geology. From a geochemical point of view, the term background is equivalent to the absence of an anomaly, and numerous methods have been applied in order to determine this geochemical background (Matschullat et al., 2000). In geoarchaeology, however, we tend to mean the composition of a soil that has not been affected by human influence. Many highly varied methods have been adopted for calculating the natural or unmodified chemistry of soils, ranging from the calculation of means for the lowest values (Parnell et al., 2002; Terry et al., 2004; Wells et al., 2000) to analysing reference soils (Entwistle, 2000; Middleton and Price, 1996; Middleton, 2004) and employing statistical calculations where those samples that exceed the natural distribution are considered to reflect anthropogenic influence (Cook et al., 2006). Other studies have avoided any consideration of the geochemical background and have focused on spatial patterns rather than absolute values (Entwistle and Abrahams, 1997; Linderholm and Lundberg, 1994). Theoretically, the best circumstances in which to determine the geochemical background for an anthropogenic soil would be when the layer to be analysed is made up of soil that has been brought to the site and it is possible to sample the area from which it was taken, i.e. it has remained undisturbed by human activity. This was possible at Lejre as far as the floor in the multifunctional house is concerned.

3.2. Lipid

Lipid analysis has been used in archaeology to search for human influence on soils since the early 1990s. Lipids include alkanoic acids, *n*-alkanols, *n*-alkanes, hydroxy acids, ketones, steroids, terpenoids, acyl glycerols and sterols together with phospholipids and lipopolysaccharides (see Dinel et al., 1990; Zelles et al., 1992). These compounds originate from both plants and animals, as products of deposition, decomposition and exudation, and from various pedogenic sources, including fungi, bacteria and mesofauna. The main source of lipids in soils is normally the vegetation (van Bergen et al., 1997; Oades, 1993), but human activity will affect the soil composition both directly, by adding substances, and indirectly, by affecting the vegetation. One important long-term study of the fate of lipids of different origins in different soils, based on the experiments carried out at Rothamsted, has been discussed in a number of articles (van Bergen et al., 1997, 1998; Bull et al., 1998, 2000a,b).

Lipids from decomposed plants primarily consist of long-chain ($C_n > 20$) alkanoic acids, long-chain alkanols and long-chain alkanes. Alkanoic acids may also derive from other sources. The main lipid class in the storage fat found in the adipose tissue of animals and in seeds and nuts from plants is that of the triacylglycerols (TAG), the alkanoic acids of which are mainly of short chain lengths ($C < 20$). During early diagenesis the total number of alkanoic acids decreases, the unsaturated alkanoic acids decrease in relation to the saturated ones and the proportion of acids with longer carbon chains increases (Matsuda and Koyama, 1977). Free alkanoic acids are produced by enzymatic hydrolysis of triacylglycerols (Hita et al., 1996), but abiotic hydrolysis may make a major contribution in acid environments (Morgan et al., 1973; Thornton et al., 1970). Wax esters may be hydrolysed to form free alkanoic acids and a long-chain alkanol, but it is not clear whether this is performed by micro-organisms or whether it is completely abiotic (Ficken et al., 1998; Moucawi et al., 1981). Free alkanols produced by the hydrolysis of wax esters or through the oxidation of alkanes may be converted to alkanoic acids (Ratledge, 1984). Most enzymatic oxidation of alkanes results in a homologous primary alkanol.

The indices Average Chain Length (ACL), Carbon Diversity Index (CDI) and Carbon Preference Index (CPI) describe the distribution of free carbon chains as calculated for *n*-alkanoic acids, *n*-alkanols and *n*-alkanes. ACL describes the average number of carbon atoms in a carbon chain, CDI describes whether the carbon chains in the samples are predominantly diverse or concentrated, so that a sample with both short and long carbon chains will exhibit a high CDI, while CPI indicates the degree of diagenesis of straight-chain lipids (Meyers and Ishiwatari, 1995). The CPI comes closer to 1 the farther the diagenesis has proceeded. The peak area of each carbon chain for *n*-alkanoic acids, *n*-alkanols and *n*-alkanes was integrated from chromatograms with characteristic ion fragments for each group m/z 117, m/z 103 and m/z 57, respectively.

The functions of sterols and their derivatives are widespread and diverse. They occur in all Eukarya, where they may constitute up to half of the lipids in the membranes. Animals tend to make cholesterol, while higher land plants typically produce phytosterols, mainly stigmasterol and β-sitosterol, and fungi preferentially synthesize ergosterol. These general patterns gave rise to the idea that sterol distributions could be used to distinguish biotic inputs into sediments (Huang and Meinschein, 1978, 1979). The analysis of sterols and their corresponding 5α-stanols has allowed culinary areas from archaeological contexts, ca. 1500 year old, to be identified (Isaksson, 1998). These results encouraged us to calculate some of the most common sterols in soils, i.e. cholesterol, stigmasterol, campesterol and β-sitosterol. Although these sterols are naturally present in soils, their occurrence differs as a result of human activities. Thus the intensity of each sterol was determined by integration from ion chromatograms using m/z 129 ions and the ratio cholesterol/[stigmasterol + β-sitosterol + campesterol] was calculated.

Soil lipid analyses have been used to trace and study the use of agricultural soils, e.g. manuring (van Bergen et al., 1997; Jandl et al., 2005; Wiesenberg et al., 2004). Coprostanol (5β-cholestan-3β-ol) is a product of the microbial reduction of cholesterol that is formed by microbial action in the mammalian gut (Eneroth et al., 1964), while the usual product of cholesterol reduction outside the gut, in mammalian tissues and sediments, is 5α-cholestan-3β-ol (Gaskell and Eglinton, 1976). Coprostanol is the major sterol in human faeces, and has been routinely studied as a marker of (modern) sewage pollution in marine and lacustrine sediments (Grimalt et al., 1990; Ibanêz et al., 2000; Leeming et al., 1996). Coprostanol in soil has been used to detect the presence of faecal material when resolving archaeological problems (Bethell et al., 1994). It has been shown that 5β-stanol markers and 5β-stanyl esters, both characteristic of farmyard manure, persist at places where manure has been applied at concentrations above those in non-manured and control areas at levels and time horizons that are of archaeological interest (Bull et al., 1998; Evershed et al., 1997).

3.3. GC–MS extraction and method

The soil samples consisted of about 5 g dried and sieved soil, to which 10 μg *n*-hexatricontane (C36) was added as an internal standard (IS). The soil was extracted with chloroform/methanol (2:1, v:v) followed by sonication, sedimentation and centrifugation. The extracts (2.5–3.0 ml) were transferred to vials and evaporated in a stream of nitrogen and the lipids were redissolved in chloroform by sonication. The lipid solution was treated with (50–100 μl) *N,O*-bis(trimethylsilyl)triflouracetamide (BSTFA) with 10% (v) chlorotrimethylsilane (at 65 °C for 30 min). Excess derivatizing agents were removed under a gentle stream of nitrogen and the samples were redissolved in *n*-hexane.

All the extracts were analysed by gas chromatography–mass spectrometry (GC–MS) using a Hewlett Packard (HP) 6890 GC interfaced with an HP 5973 Mass Selective Detector. Injection was carried out by the pulsed splitless technique at 350 °C with a pulse pressure of 17.6 psi. A SGE BPX5 capillary column (15 m × 0.25 mm × 0.25 μm) was used. The oven temperature was held at 50 °C for 2 min, ramped to 360 °C at 10 °C min^{-1} and held there for 15 min. Helium, held at a constant flow of 2 ml min^{-1}, was used as a carrier gas. The GC was coupled to the MS by an interface with a temperature of 325 °C. Scanning in the range *m*/*z* 50–700 provided 2.26 scans s^{-1}.

3.4. AAS extraction and method

It has been stated that a significant proportion of the anthropogenic signal is held within the more resistant soil fractions suggesting that the use of a weak acid or an exchangeable fraction extraction would fail to release a large part of the anthropogenic signal (Wilson et al., 2006). Hence we have used a strong acid. From comparing acids and temperatures on a reference soil we have found that following extraction procedure gave a good exchange and precision (Hjulström, in press). One gram of soil from each sample was digested with 10 ml Aqua Regia (nitric acid:-hydrochloric acid v/v 1:3) in a MARSX microwave oven, ramped to 175 °C over 20 min and held there for 10 min. The samples were then filtered and the filtrate was diluted to 25 ml and thereafter to a suitable dilution factor depending on the element to be analysed. The analyses were performed using a Z-5000 Polarized Zeeman Atomic Absorption Spectrophotometer to determine the concentrations of calcium (Ca), copper (Cu), iron (Fe), potassium (K), magnesium (Mg), manganese (Mn), lead (Pb) and zinc (Zn). The concentrations in the solutions were measured, recalculated to concentrations in the soil and presented as mg kg^{-1}.

4. Results

4.1. Element analysis

The results from the elemental analysis clearly demonstrate that it is possible to discriminate between areas of different use. An ANOVA analysis on the whole dataset showed statistically

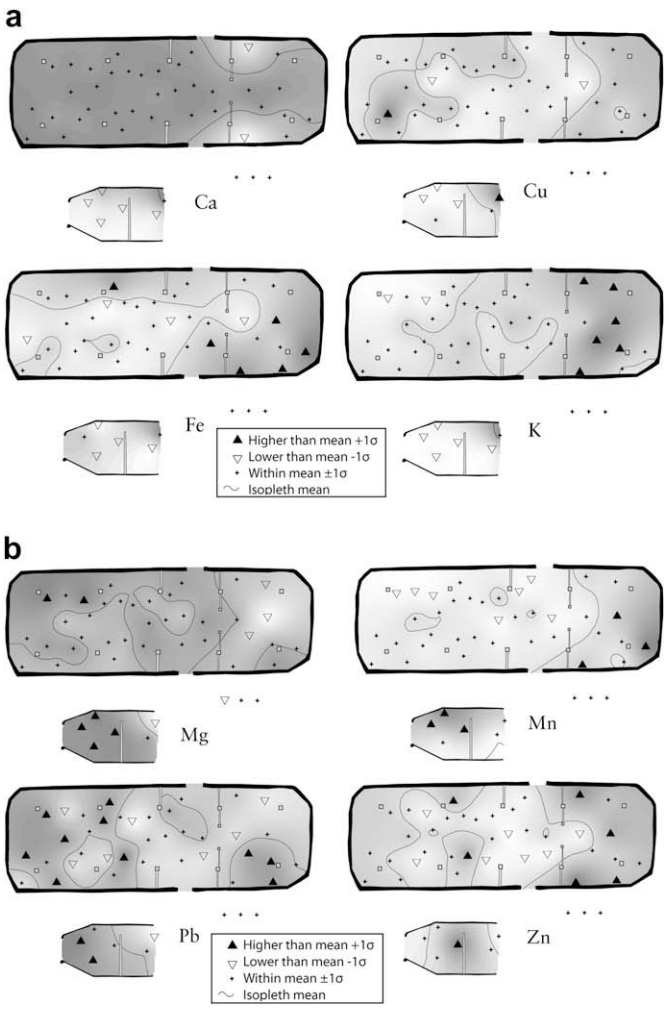

Fig. 2. Variations in Ca, Cu, Fe, K, Mg, Mn, Pb and Zn contents across the floor of the house and smithy. The isopleths are based on the absolute values. Cross = sample falling within the mean ± 1σ; upward triangle = higher than the mean + 1σ, and downward triangle = below the mean − 1σ. The lines are based on the standardizations of the samples. Solid lines denote the mean, broken lines the mean − 1σ, decreasing by 1α for each broken line, and thin lines the mean + 1σ, increasing by 1σ for each line.

significant differences (Wilks' $\Lambda = 0.00676$, Rao's $R = 5.02$, $df_1 = 48$, $df_2 = 136$, $p < 0.000$), and additional statistical calculations were also made in order to demonstrate and identify these differences. The results of the element analyses are presented in Fig. 2. The isopleth, in which white represents the lowest value and dark gray the highest, is based on the absolute element values, not taking into consideration how large a span the samples fall into for each element. A quick overview of the element values is provided in Table 2, which shows the mean value for each element and area and the total mean and standard deviation. The samples were also plotted as higher or lower than the mean ± standard deviation.

Table 2
Mean element concentrations (mg kg^{-1}) in each area and total means with standard deviations

Area	Mn	Ca	Pb	K	Mg	Cu	Fe	Zn
Dwelling	218.9	23 176	134.7	15 262	632.4	12.4	21 913	36.9
Dwelling/cooking	222.4	23 048	133.8	17 199	627.4	12.3	21 778	37.3
Entrance	209.8	22 981	132.7	17 766	630.6	12	20 040	33.6
Stable	258.1	18 047	134.5	22 906	610.7	13.2	27 278	48.1
Smithy	293.3	5965	135.2	10 367	642.8	12.3	16 944	38.4
Reference	241.62	23 615	933	20 661	589.3	13.8	26 159	33.29
Total	233 ± 48	21 861 ± 3381	134 ± 4.5	17 940 ± 3636	626 ± 19	12.5 ± 1.2	22 643 ± 4953	37.5 ± 4.6

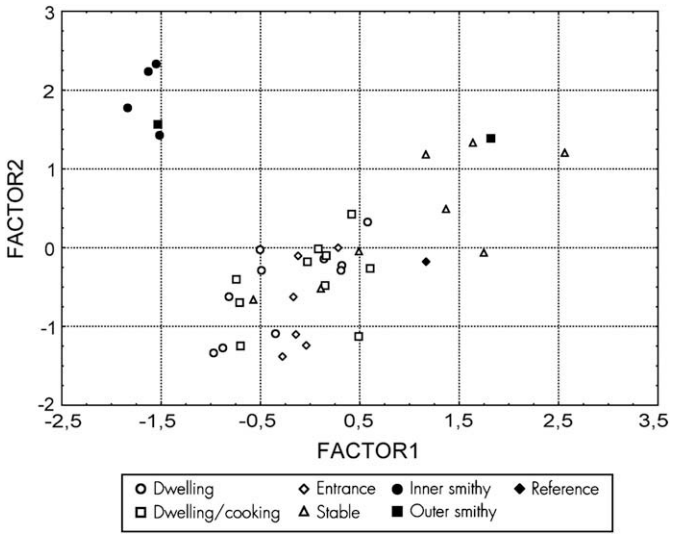

Fig. 3. Scatter plot of PCA results for all elements.

Crosses designate the samples in Fig. 2 if within the mean range, an upward triangle if above the mean range and a downward triangle if below the mean range. The lines are based on the standardization of the samples, with the thick line denoting 0.

The smithy is characterized by low Ca, Cu, Fe and K and values for Mg, Mn and Pb that are above the mean. There is also a difference between the areas separated by the wall in the smithy.

The samples from the stable are somewhat more similar to the mean values and there are often just one or two out of the nine samples that differ from the mean values. Fe, K, Mn, and Zn are nevertheless enhanced in three (Mn) to six (K) samples from the stable, while Mg values are low.

Areas characterized by movement, that is doorways and entrances, suffer more wear and tear, and they often have low concentrations of chemical residues on account of erosion (Barba, 2007; Middleton, 2004). The entrance to the multifunctional house had somewhat depleted Zn, Mn and Cu values. These samples were, more importantly, depleted also in comparison with the reference material.

A data reduction method was employed in which the element values were subjected to principal component analysis (PCA). Since

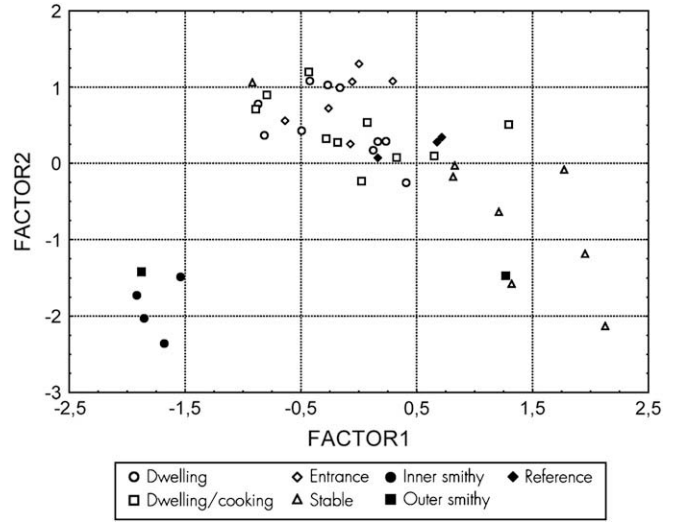

Fig. 4. Scatter plot of PCA results for all elements except Mg and with all three reference samples shown.

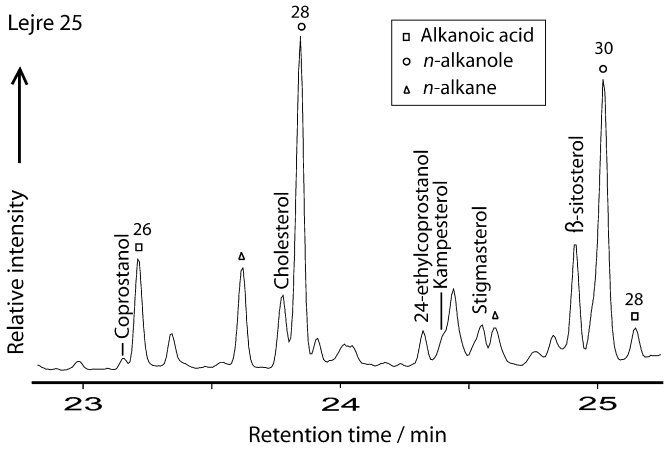

Fig. 5. Total ion chromatogram showing the sterol fraction of sample 25.

two of the reference samples lack a result for Mg, the factor analysis was performed both with and without Mg (see Figs. 3 and 4). The first three discriminant factors account for 76% of the total variance within the dataset. Factor 1 accounts for 36.5% of the total variance and has high positive loadings of Mg (0.81) and Fe (0.70) and high negative loadings of K (−0.89) and Cu (−0.71), while factor 2 accounts for 26.3% of the variance, with high positive loadings of Mn (0.79) and Zn (0.84). In the PCA analysis without Mg and with all three reference samples, factor 1 accounts for 34.7% of the variance, with high positive loadings of Ca (0.77), K (0,71) and Fe (0.65), while the dominating feature of factor 2 is a negative loading of Zn (−0.87). Both scatter plots, with and without Mg, serve well to distinguish between the smithy, the stable and the rest of the samples. The entrance has a somewhat narrower distribution when looking at factor 1 for all elements than do the dwelling and dwelling/cooking area. Otherwise these three areas cluster together and cannot be separated internally. The reference samples in the scatter plot without Mg are closely clustered.

4.2. Lipid analysis

4.2.1. Sterols and stanols

The total ion chromatogram (TIC) for the sterol fraction of sample 25 is shown in Fig. 5. Sample 25 was taken in the stable and contained both coprostanols and sterols. Some of the sterols co-eluted with other compounds, and the peak areas were integrated using the *m*/*z* 129 ion chromatogram. The sterol ratio exhibits clear

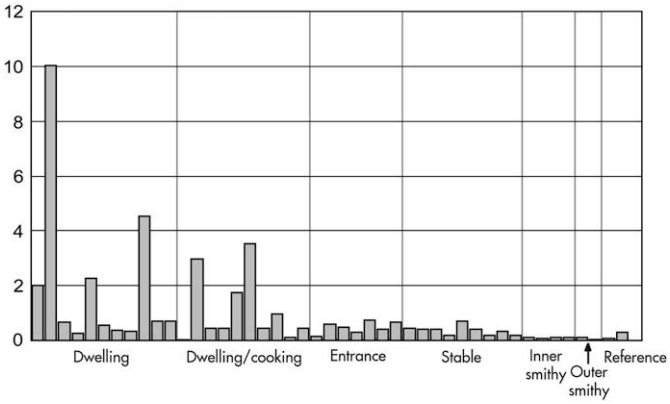

Fig. 6. Bar chart of the sterol ratio (cholesterol/[stigmasterol + campesterol + β-sitosterol]). The proportion of cholesterol in relation to other sterols is higher in areas 1 and 2. The smithy and the reference samples have a low cholesterol ratio.

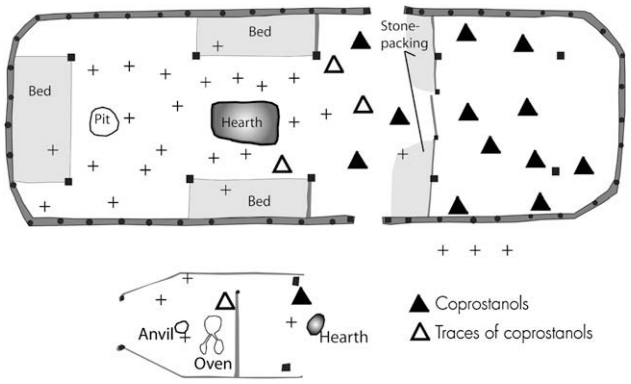

Fig. 7. Presence of coprostanol and 24-ethylcoprostanol. The ratio of coprostanol to 24-ethylcoprostanol varies from 1:2 to 1:5 in the samples.

differences between the areas, as illustrated in Fig. 6. All the samples from the smithy and the reference samples have a low sterol ratio, ranging from 0.001 to 0.1. The sterol ratio in the multifunctional house is generally higher, with only seven out of thirty-seven samples below 0.2 and only one below 0.1. No clear difference can be seen between the entrance and the stable. The dwelling area has a higher input of cholesterol, with seven out of twenty-one samples having a sterol ratio over 1, enabling the dwelling area to be pinpointed and distinguished from the entrance and stable. Some of the samples in the dwelling have a sterol ratio similar to the entrance and stable, however. What are unique for the dwelling and dwelling/cooking areas are the diversity of the sterol ratio and the relatively high sterol ratio.

Coprostanol and 24-ethylcoprostanol were detected in 17 samples. 24-Ethylcoprostanol has been used as biomarker for agricultural (non-human) faecal matter, being the principal faecal

Table 3
ACL, CDI and CPI for alkanoic acids, alkanols and *n*-alkanes in all samples

Sample		Alkanoic acid			Alkanols			Alkanes			Total (μg/g)
		ACL	CDI	CPI	ACL	CDI	CPI	ACL	CDI	CPI	
Dwelling	1	16.3	2.1		24	2.1		29.8	2.5	1.8	25
	2	16.4	1.6		23.7	2	24.9	29	2.4	1.6	50
	3	16.4	2.5		24.7	1.9		31.3	1.8	2.5	21
	4	16.5	2	11.1	24.7	1.8		30.6	2.1	1.4	14
	5	16.4	2.2		25	1.8		30.1	2.2	2.4	13
	6	16.4	1.8		25.2	1.7		28.3	3	1.5	12
	7	16.3	1.7		26.3	1.5		28.7	2.9	2.4	19
	8	16.3	2.9		24.1	1.8	84.1	28.4	2.8	1.2	34
	10	16.1	2.3		21.3	0.9	118	29.4	2.5	1.3	188
	38	16.1	1.6		23.7	1.6		30.8	2	1.3	7
	40	16.4	1.6		25.7	1.7		27.6	3.2	1.9	13
Dwelling/cooking	9	16.5	2.1		26.5	1.4		29.7	2.5	2	15
	11	16.2	2		23	2.3		30.5	2.1	1.9	72
	12	16.5	1.8		25.7	1.7		31	2	1.7	17
	13	16.3	2.2		25.1	1.6		29.9	2.3	2.6	23
	14	16.2	2.5		23.2	2.4		28.4	2.1	4.6	76
	15	16.3	2		22.9	2.2		32.1	1.5	1.3	19
	16	16.3	1.7		26.3	1.4		30	2.3	2.4	9
	17	16.2	1.8		24.1	2.1	7.8	30.3	2.3	2.2	15
	18	16.5	1.9		24.9	1.5		28.8	2.9	2.8	18
	19	16	1		25.1	1.8		32.2	1.5	1.1	5
Entrance	20	16	1.9		25.8	1.6	42.4	31.8	1.9	2	11
	21	16.7	1.9		24.4	1.8		28.6	2.7	1.8	35
	27	16.4	1.8	6.7	26.4	1.8	36.7	31.3	1.8	2.9	20
	28	16.7	3		25.3	1.8	6.2	30.6	2.3	2.4	22
	29	16.5	1.7	2.7	25.8	1.5	43	28.6	3.1	3.8	19
	30	16	2		24.9	1.7		30.7	2.1	2.1	17
	31	16.3	1.9	9.9	25.4	1.6	26.5	29.5	2.6	4.4	36
Stable	22	18.5	2.1	10.7	21.9	1.7	0	30.8	2.2	5.1	9
	23	18.3	2	13.5	26.7	1.7	21.3	29.3	1.8	48.3	102
	24	18	2.1	12.9	26.6	1.6	32.2	30.5	2	10.3	31
	25	17.8	2.3	13.4	27	1.8	26.4	30.2	2.1	11.6	54
	26	18.2	2.8	15.1	26.5	1.5	33.4	30.4	1.9	15.9	33
	32	14.3	1.9	9.8	26.1	1.3	82.7	30	1.7	–	115
	33	20.8	3.3	15.5	26.7	1.7	26.5	30.1	2	18.8	90
	34	16.5	2	0.5	27.3	1.8	45.1	30.6	2.2	2.9	6
	37	20.2	2.4	5.2	26.6	1.8	26.6	30	2.8	4.5	27
Smithy	50	21.9	3.6	10	26.5	2.2	13.4	30.1	2.4	6.5	21
	51	23.8	4.1	6.8	26.3	2.2	9.3	29.8	2.6	6.3	31
	52	22.3	3.4	14.1	26.4	1.9	14.2	30.1	2.3	8.7	28
	53	23.5	3.6	11.6	26.5	2.2	9	29.9	2.4	7.4	25
	54	24.6	3.6	8.5	26.6	2.2	12.3	30.1	2.4	8	23
	55	16.8	1.6	0.7	27	1.7	33.4	30.8	1.9	2.9	4
Reference	60	16.3	1.5	–	24.8	2.2	84.2	33.6	1.1	0.3	5
	61	15.7	1.6	0	26.4	1.6	48.2	32.2	1.5	1.2	2
	62	16.3	3.2	15.8	22.3	2.5	6.8	33.4	1.3	0.6	29

CPI is missing for several samples of alkanoic acids and alkanols due to the lack of uneven carbon chains. ACL = $\sum([C_i] \times i)/\sum[C_i]$; C_i = the relative abundance of a saturated alkanoic acid, alkanol or *n*-alkane; i = carbon number. CDI = $1/\sqrt{([C_i/100]^2)}$; C_i = the relative abundance of each carbon chain in percent. CPI for alkanoic acid and *n*-alkanols = $\sum_{even}(C_8 - C_{32})/\sum_{uneven}(C_9 - C_{33})$; CPI for *n*-alkanes CPI = $\sum_{uneven}(C_{17} - C_{35})/\sum_{even}(C_{16} - C_{34})$. C_i = the relative abundance of each carbon chain in percent.

biomarker for herbivores (Leeming et al., 1996). The samples in which coprostanol and/or 24-ethylcoprostanol were identified are shown in Fig. 7. 24-Ethylcoprostanol predominates, with the ratio between the two varying from 1:2 to 1:5 among the samples, thus providing evidence for an input of faeces from herbivores. Not surprisingly, the samples with coprostanol and 24-ethylcoprostanol are concentrated in the stable, although there is some faecal input in the entrance and the smithy. Three of the samples in the entrance contained only trace levels of the biomarkers. The animals passed the entrance area often which explain the faecal biomarkers in the area.

4.2.2. Carbon chains

The ACL, CDI and CPI values for *n*-alkanoic acids, *n*-alkanols and *n*-alkanes are shown in Table 3. The alkanoic acids had the greatest diversity in carbon chain composition, with an ACL ranging from around 16 to 24. Most samples in areas dwelling, dwelling/cooking and entrance were lacking long alkanoic carbon chains.

All the samples contained practically identical series of *n*-alkanes characterized by a monomodal distribution centred about C29 and C31 and with C25 and C27 homologues also dominant. ACL values ranged from 28.4 to 31.8 in the anthropogenic soils, while the ACL for the reference samples had the highest values, 32.2–33.5. A bimodal distribution was observed for *n*-alkanols, ranging from C16 to C32, while the bulk of the samples had a maximum at C26, followed by C28 and C24. The *n*-alkanols exhibited a greater ACL variety than the *n*-alkanes. Several samples in the areas dwelling, dwelling/cooking and entrance as well as the reference samples had a low ACL for alkanoic acids, which could be a signal that these originated from adipose tissue, although there are other sources of alkanoic acids with carbon chain lengths shorter than 20, e.g. cutin and suberin from plants (Kögel-Knabner, 2002). In this case the low ACL is more likely to be a result of the absence of long-chain fatty acids from plant wax residues, since the same pattern can be seen in the reference samples with no anthropogenic influence.

4.3. PCA and discriminant analysis for elements and lipids

The variables used for the factor analysis were all the elements, the ACLs for alkanoic acids, *n*-alkanols and *n*-alkanes and the cholesterol ratio. The same analysis was also performed without Mg in order to include all the reference samples. Factor analysis reduced the dataset with all the elements to two principal components, accounting for 52% of the total variation. By plotting the scores on the first two principal components, the various areas were found to follow the patterns seen in Figs. 8 and 9. Factor 1 is seen in Fig. 8 to be associated with high positive loadings of Ca (0.69), the ACL for alkanoic acids (0.68) and the sterol ratio (0.73) and a negative loading of Mn (−0.69) and the ACL for *n*-alkanols (−0.75). The smithy had the lowest scores on both factor 1 and factor 2, while the stable had high factor 2 values. Just as with the PCA with only element results (Figs. 3 and 4), the smithy and the stable are well separated. It can be seen in both Figs. 4 and 9, where Mg is not included, that the reference samples are all well grouped.

When the results of the lipid analyses are included in the PCA (Figs. 8 and 9), it is possible to make further divisions of the areas in the dwelling house. Looking at the scatter plot only it is not possible to separate the areas dwelling, dwelling/cooking and entrance from each other. However, from a similar result from an archaeological context with different areas identified it would be possible to discuss different use in these areas since the dwelling and the dwelling/cooking areas have a much higher diversity of the factor scores.

A discriminant function analysis, classified from the areas, was first made for the elements and lipids respectively and then combined. From the elemental analysis all elements except Mg were used as variables. For the lipid analysis ACL, CDI and CPI for alkanoic acids, *n*-alkanols and *n*-alkanes, and the sterol ratio were used as variables. The elemental analysis (Wilks' Λ 0.01121, F (42, 148) = 5.6880, $p > 0.0000$) had a correct classification for 70.5% of the samples. The lipid analysis (Wilks' Λ 0.03932, F (48, 161) = 3.1305, $p < 0.0000$) had a correct classification for 67.4% of the samples. The best classification level was reached when all parameters were combined (Wilks' Λ 0.00117, F (90, 135) = 3.5100, $p < 0.0000$), with a correct classification for 84.1% of the samples. The *p*-levels for separation between areas and correct classification are shown in Tables 4–6.

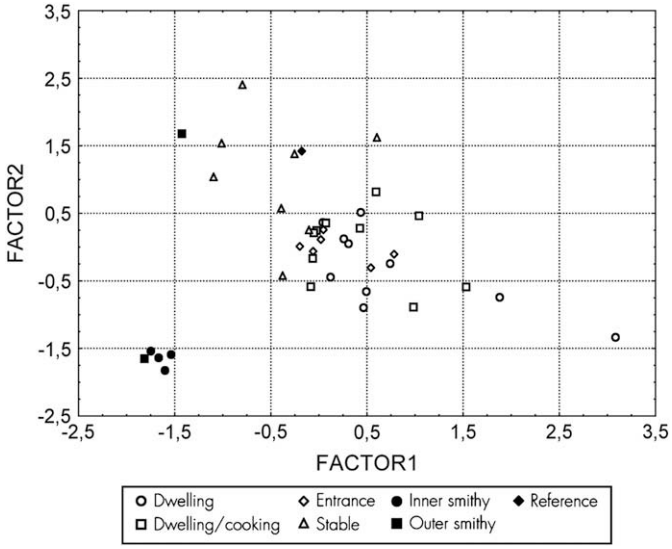

Fig. 8. Scatter plot based on the PCA for all elements and the lipid indices ACL, CDI and CPI and the sterol ratio.

5. Discussion

Space use and household-related research has for long interested archaeologists. However, for several periods and regions (e.g. the one in focus here, Iron Age Scandinavia) the visible remains are too few to discuss actual space use and it is needed to try other

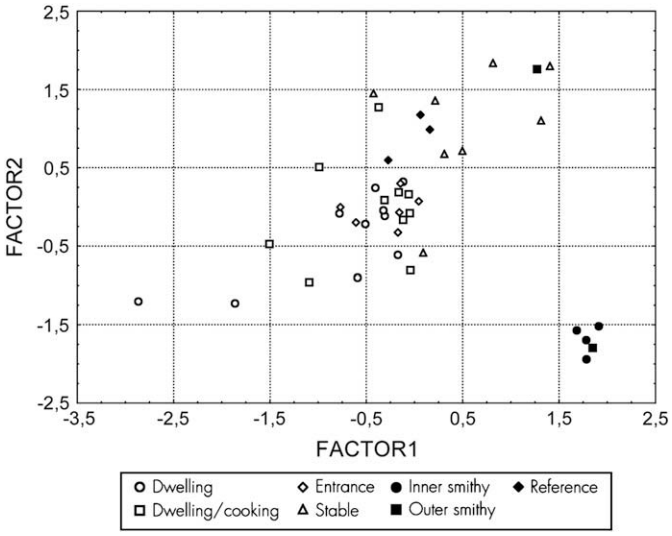

Fig. 9. Scatter plot of PCA results for all elements except Mg, the lipid indices ACL, CDI and CPI and the sterol ratio.

Table 4
p-Values and correct classification percentage from the discriminant analysis on element results

Area	*p*-levels – element					
	Dwelling	Dwelling/ cooking	Entrance	Stable	Inner smithy	Outer smithy
Dwelling/ cooking	0.699					
Entrance	0.127	0.616				
Stable	0.000	0.000	0.002			
Inner smithy	0.000	0.000	0.000	0.000		
Outer smithy	0.000	0.000	0.000	0.000	0.001	
Reference	0.350	0.590	0.712	0.054	0.000	0.000

Correct classification (%) 70.5.

Table 6
p-Values and correct classification percentage from the discriminant analysis on element and lipid results combined

Area	*p*-Levels – element and lipids combined					
	Dwelling	Dwelling/ cooking	Entrance	Stable	Inner smithy	Outer smithy
Dwelling/ cooking	0.573					
Entrance	0.059	0.114				
Stable	0.000	0.000	0.355			
Inner smithy	0.000	0.000	0.000	0.000		
Outer smithy	0.000	0.000	0.000	0.000	0.031	
Reference	0.393	0.231	0.295	0.033	0.000	0.000

Correct classification (%) 84.1.

means such as geochemistry to deduce this information. The sampled material used here comes from a modern context that is as close as we can get to a farm in Scandinavia during the Iron Age. From an Iron Age house the soil chemistry will of course be very different since the soils have been affected in various ways and the diagenesis has gone much further, and the interpretation presented here cannot be directly applied on a prehistoric house.

The reference samples have rather similar quantities for each element. All fall within the mean interval, showing that depletion in element content is also a factor to consider when studying the effect of human activities on soils. It is seldom possible to sample an undisturbed soil with no anthropogenic influence and with the same original geochemical composition as a related cultural layer. The similar values obtained for the three reference samples provide essential information, however, since we can then assume that the floor matrix was homogeneous at the moment of its construction and that it initially had a relatively consistent elemental composition over the whole surface. This supports the idea that it is more relevant to decide how to treat samples collected from one anthropogenic horizon than to classify the geochemical background. And that it is more important to study relative differences within the same horizon and soil type and to relate these to the archaeological data (see Linderholm and Lundberg, 1994; Entwistle and Abrahams, 1997).

Potassium (K) is taken up by plants in the form of K^+ ions, and this means that wood ash is rich in K. The archaeological significance of Ca regarding wood ash should be more or less the same as for K, as plants also take up large amounts of Ca. Mg is also a necessary element for plants, but is not taken up in the same magnitude as K and Ca. Mg and K have been used to identify areas with hearths (Middleton and Price, 1996).

The Ca levels were more or less the same in all areas of the multifunctional house with the exception of two samples from the stable. The Mg values for the stable are low, while K is high. A large input of K could however be expected in the stable, since manure contains more K than Ca and Mg (Lekasi et al., 2003). In a previous study of an Iron Age house in Central Sweden, the area interpreted as having been used for food storage had enhanced Ca values while the dwelling area had higher K values (Isaksson et al., 2000). Hearths have also been found to have a concentration mechanism for a range of metals including Pb, Cu and Zn (e.g. Meharg et al., 2006; Davidson et al., 2007).

This exemplifies the complexity of using element values in the soil to interpret specific activities. If we were to interpret the elements indicative of a hearth it would not have been possible to reach the correct conclusion regarding the location of the hearth in the house. When working with an archaeological material it is seldom as easy as choosing between two known probable activities but rather of a multitude of unknown activities. This means that the relationship between a specific activity and the element signal they produce needs to be better understood if used for interpreting actual activities from an archaeological material. The elemental analysis did however clearly separate several space-use areas.

The use of ACL, CDI and CPI for alkanoic acids, *n*-alkanols and *n*-alkanes will be of lesser interest when sampled from prehistoric sites since they will be influenced by later vegetation to a large degree. Although the lipids have a more rapid decay trajectory toward simpler indistinguishable compounds some markers have better potential for survival. The dwelling and dwelling/cooking and the stable were recognized by its sterol composition. Stanol and sterol ratios have shown to differ in samples from prehistoric anthropogenic layer, often connected to hearts and food preparation (Isaksson, 1998; Hjulström et al., in press). From these sites are the sterol ratios far from as obvious as shown in Fig. 6. However, the relative differences of the ratios are connected to archaeological identified areas. We have so far not been able to identify coprostanols or related compounds from an Iron Age building indicating a stable as demonstrated in this paper. Earlier studies have shown that coprostanols and related compounds have a preservation rate that makes them of interest for archaeological studies (Bethell et al., 1994; Bull et al., 1998; Evershed et al., 1997). The reason that we have not found compounds related to manuring and stabling in the prehistoric buildings we have analysed is more probably due to that the houses were not used for stabling.

Table 5
p-Values and correct classification percentage from the discriminant analysis on lipid results

Area	*p*-Levels – lipids					
	Dwelling	Dwelling/ cooking	Entrance	Stable	Inner smithy	Outer smithy
Dwelling/ cooking	0.505					
Entrance	0.156	0.272				
Stable	0.000	0.000	0.017			
Inner smithy	0.0	0.00.0	0.00.0	0.001		
Outer smithy	0.001	0.004	0.013	0.458	0.661	
Reference	0.001	0.021	0.021	0.005	0.0010	0.011

Correct classification (%) 67.4.

6. Conclusions

This study has demonstrated some advantages of combining inorganic and organic soil chemical analyses in order to locate and identify activity areas at archaeological sites. The study has also showed the relevance of considering differences in the degree of variation within different areas instead of focusing on absolute values only, in the combined organic–inorganic approach. The element analysis enabled the smithy and the stable to be distinguished as activity areas contrasted with the rest of the samples. All the reference samples exhibited similar values. When attempting an unbiased interpretation of the results from the element analyses

it was not possible to identify the activities correctly. The relation between the specific activities and the elemental indicators that they deposit needs to be better established. However, it is of importance that it was possible to separate different areas of space use with the element analysis.

The same three areas that could be distinguished by element analysis could be seen with the lipid analysis on the basis of ACL, CDI and CPI values as in the case of the element analysis. The biomarker 24-ethylcoprostanol enabled a qualitative identification of the stable. The sterol ratio was successful in identifying and characterizing the dwelling area as distinct from the rest of the house. However, no differences could be seen between the dwelling room and the dwelling/cooking room, respectively.

Sediment chemistry has for a long time shown a potential for making substantial contributions to archaeological research by providing data on the spatial organization of dwelling sites, although specific activities are difficult to identify. The relationships between certain activities and their expected elemental indicators still need to be examined further, but the combination of element analysis with lipid analysis allowed several of the general activities at the Lethra Iron Age village to be identified. This is a promising result for future geochemically based archaeological research regarding space use and activities in prehistoric houses.

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