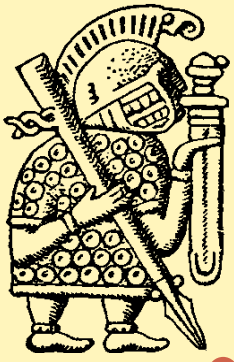

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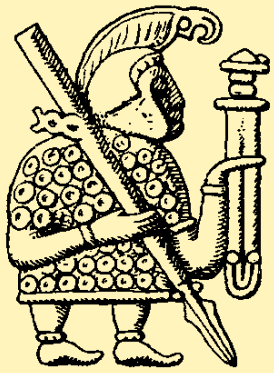

2023/2

Radiocarbon dating of purified lipid extracts from prehistoric pottery

By Madison Holly Llewellin and Sven Isaksson

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In this paper we present the results of an investigation where we try to date pottery from an Early to Middle Iron Age site in eastern middle Sweden. An overarching aim is to contribute to the discussion of the timing of a change in land-use and settlement structure that occurs in the middle of the first millennium CE. Selecting a well preserved site for excavation, we performed both plant macrofossil analyses of soil-samples and lipid residue analyses on pottery. Both archaeobotanical materials as well as lipid extracts were subjected to radiocarbon dating. The results show that the radiocarbon dating of purified lipid extracts from prehistoric pottery is possible.

Keywords: Radiocarbon dating, pottery, lipid residues, Middle Iron Age, Eastern middle Sweden

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Introduction

With radiocarbon analysis a radiocarbon event is dated. The radiocarbon event is defined as the event when a carbon accumulating material is cut off from its radiocarbon source (Nelson 1998). When sampling for radiocarbon dating in archaeology it is a major challenge to select a sample that represents a radiocarbon event that is as close as possible in time to the archaeological event, i.e. the event that the archaeologist wants to date, which is rarely identical to the radiocarbon event. Often we try to sample short-lived materials such as carbonized plant seeds, hoping that the year of growth is close to, say, the use of a building that we want to date. Furthermore, small objects such as carbonized seeds are quite mobile in the soil due to so-called bioturbation. To complicate things even more, archaeological sites located in the Boreal forest run a significant risk of having been subject to forest fire since their inception. In this paper we present a case study dealing with these kinds of problems applying a novel approach by dating purified lipid residues extracted from prehistoric pottery by radiocarbon analysis.

Background

During the Early Iron Age (500 BCE–550 CE), a region with extensive systems of interconnected stone fences and settlements was established in the provinces of Uppland and Västmanland in

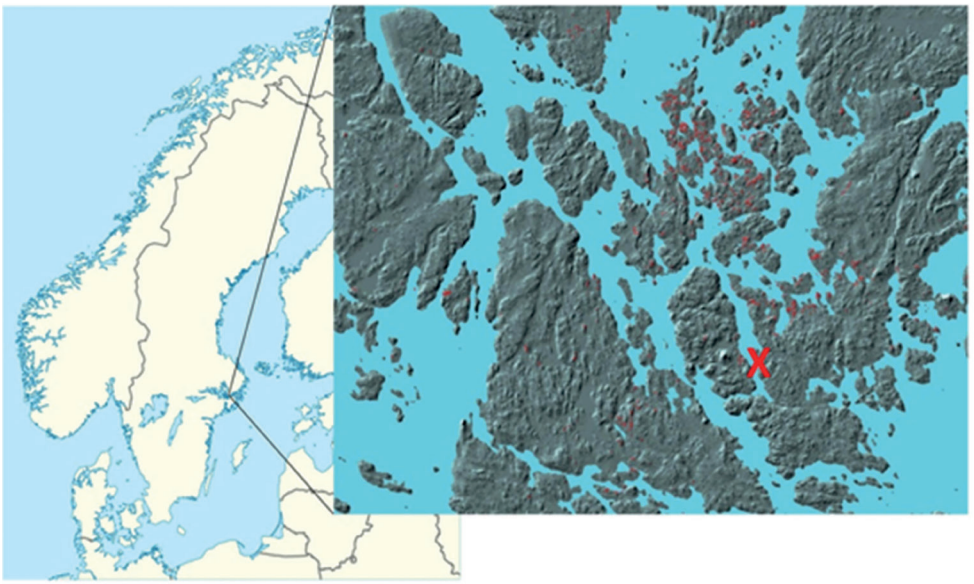

Fig. 1. Map of Scandinavia and the location of the area of investigation, with sea level set to a situation corresponding to the beginning of the Early Iron Age. The location of the site of investigation, Påtåker, is marked by a red X. Map produced by Andreas Viberg based on geodata (Lantmäteriet).

eastern middle Sweden. The region can be subdivided into several geographically defined districts of connected settlements and stone fence systems (Essen & Ramström 2007, p. 73). One of these has its centre in what today is south central Uppland. During the Early Iron Age, coves of the Baltic Sea broke into the landscape and the area was an agricultural, rural milieu set in a coastal archipelagic environment (Essen & Ramström 2007, pp. 73–75) (fig. 1).

A substantial section of the southern part of this stone fence district was subject to extensive archaeological investigations and excavations in connection with infrastructural development in the area between 2003 and 2004 (cf. Johansson & Lindgren 2005; Olausson 2008). During these investigations, efforts to date the stone fences were made at several sites and the results show that they have consistently been built in the decades around year 0 CE (Johansson & Lindgren 2005, p. 15). The investigated remains of this system of stone fences and stone fence-connected settlements give the impression of having been constructed as a finalised structure from start, with little to no evidence of rebuilding or restructuring during its time of use. The main reason for the construction of the stone fence system was the management of livestock movement in the landscape, being closely integrated with arable farming through the practice of manuring (Essen & Ramström 2007, p. 71). The stone fences have functioned as dividers of different land-uses but also as social markers of ownership and occupation (cf. Cassel 1998; Isaksson et al. 2019). Detailed investigations also indicate that there was a hierarchy among settlements within the stone fence system (Hamilton & Vinberg 2011; Jansson 2020).

This stone fence system was abandoned fairly simultaneously during the Migration Period (400–550 CE), whereby the stone fences lost all their functions (Pedersen & Widgren 1999, p. 321; Johansson & Lindgren 2005, p. 16). Both the locations of settlements and of cemeteries were rearranged within the landscape during this transition. At some sites farms are completely abandoned (Isaksson et al. 2019), at other sites there is evidence of continuity but

with structural rearrangements. The reasons behind this change have been the subject of much debate. Similar changes on the island of Gotland in the Baltic Sea have been associated with the introduction of a two-field crop rotation system (Carlsson 1979) but there is no evidence for such a system in Uppland until the 13th C. CE (Ericsson & Strucke 2008, p. 60). Another suggested contributor is the Post-glacial land uplift causing earlier waterways with access to the Baltic Sea to become non-traversable by boat. This is based on the observation that the farm sites that were really abandoned were those that had lost their access to the waterways, underlining the importance of transportation (Jansson 2020), but also the direct access to marine resources. Contributions to abandonment could come from a land-use locked in the landscape by the immovable stone fence system (Pedersen & Widgren 1999, p. 294) and a strong dependence on animal manure making the agricultural system equally inflexible and therefore vulnerable to sudden changes. One such change that could have contributed is the so called 536 CE dust veil event (Gräslund & Price 2012), caused by a number of volcanic eruptions resulting in several years of cold summers (Larsen et al. 2008; Büntgen et al. 2016; Helama et al. 2018). This event is acknowledged to have had a long-term impact throughout Eurasia by causing crop failure and concurrent famine and societal crisis, which in some regions led to human migrations (McCormick et al. 2012; Büntgen et al. 2016).

These fundamental economic changes did not come alone, there is also ample evidence for massive social and cultural changes; it was a change of epochs. The establishment of a new political order and a Late Iron Age elite in Scandinavia during the 6th C. CE has been attested by several scholars (e.g. Hershend 1992; Callmer & Rosengren 1997; Arrhenius 2001; Andrén 2014). This coincides in time with a religious shift and the materializations of the Old Norse mythology (cf. Andrén et al. 2006; 2014), used by the rising elite as their ideological base (e.g. Herschend 1992; Hedeager 1997; Isaksson 2000). Andrén (2014, p. 171) relates this transformation to what he refers to as a "cluster of change" occurring in the Migration Period. Material evidence for these changes in the archaeological record of eastern central Sweden are the emergence of monumental terraces for prominent hall-buildings (Herschend 1997; Ljungkvist & Frölund 2015), the large burial mounds (Bratt 2008) and the commencement of the boat-grave cemeteries (e.g. Arwidsson 1980; Ambrosiani 1983; Arrhenius 1983). This is the start of what later becomes the Viking Age and eventually medieval Sweden (cf. Isaksson 2000, p. 62).

In order to be able to begin to untangle the causes and effects in this complex shift, the precise dating of the last use of the abandoned settlements in the Migration period is crucial. This was the task that we set out to try and resolve.

Site selection and investigation

The central parts of the stone fence district in southern central Uppland have a long chronological continuity and archaeological remains from the Late Iron Age and historical times cover and obscure the earlier ones. This also applies to the stone fence sites investigated in 2003–2004, many of which now lay embedded in the historical cultural landscape and with continued use from the Early Iron Age and onwards (Essen & Ramström 2007, p. 75). For our purpose we needed samples from a more undisturbed area on the rim of the interconnected stone fence system. There is such an area within the Rösjö Forest Nature Reserve in eastern Sollentuna, southern Uppland. The area is located on the southwestern edge of the stone fence district and has been subject to an earlier archaeological investigation and detailed survey carried out by the Stockholm County Museum (Grönwall & Höglin 2006). This investigation concludes that the area in question, with its unmixed Early Iron Age character, has high scientific potential and is likely to contain the answers to many of the issues concerning the rise and fall of the stone fence landscape (Grönwall & Höglin 2006, p. 18). These were precisely the kind of sites we were looking for.

Of the several sites within the area, the site Påtåker (L2015:4456, Sollentuna RAÄ 62) is one of the most suitable since it has been left more or less undisturbed after being abandoned. The visible remains today consist of a number

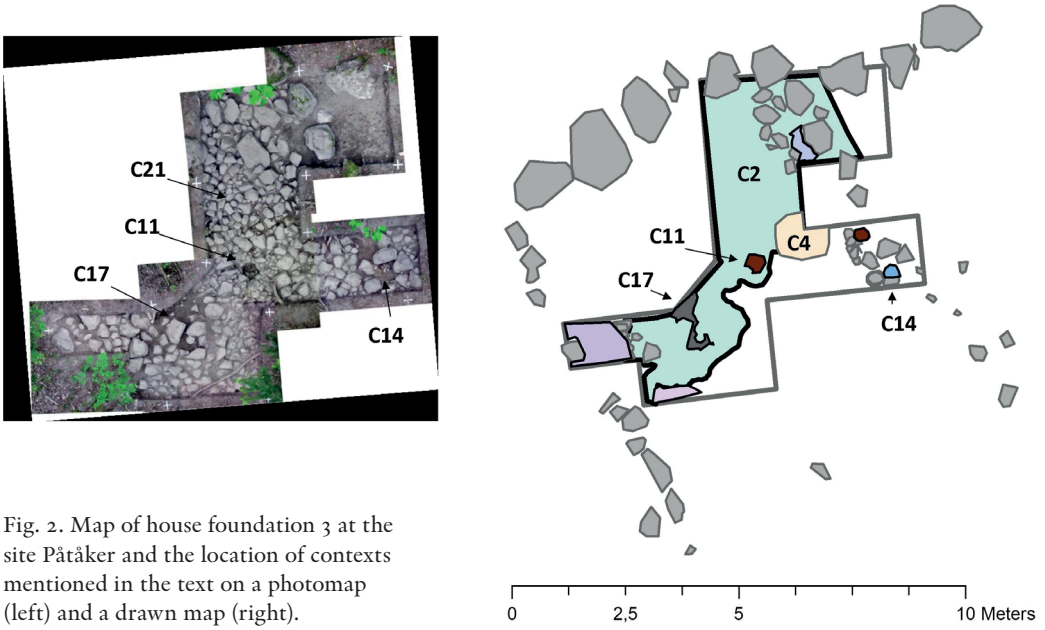

Fig. 2. Map of house foundation 3 at the site Påtåker and the location of contexts mentioned in the text on a photomap (left) and a drawn map (right).

of stone fences, a couple of stone settings, two house foundations and a terrace (Isaksson & Fjellström 2016; Isaksson et al. 2019). During the Early Iron Age this site was located directly by waterways that first provided access to the Baltic Sea, which was lost due to the Post-glacial land uplift around 200 CE (Karlsson & Risberg 2006, p. 111).

Research excavations were carried out at the site by the Archaeological Research Laboratory of Stockholm University in 2016–2017, and the two house foundations and the terrace have been partially excavated (Isaksson & Fjellström 2016; Isaksson et al. 2019). On the two house foundations culture layers were found, as well as traces of buildings in the form of features, e.g. post-holes, stone-foundations, wall-ditches, etc. (fig. 2). Finds from the culture layers and features consist of primarily of burnt clay, burnt and unburnt bones, ceramics, various iron objects and slag. The finds belong typologically to the Iron Age. The house-remains on the two foundations are consistent with traces of buildings from the Early Iron Age of the region (Isaksson et al. 2019, p. 20).

The raised terrace, however, is somewhat of an outsider in this context. As already stated above, the construction of these kinds of monumental terraces for prominent hall-buildings starts in the decades around 600 CE (Ljungkvist & Frölund 2015, p. 10) and does not really belong in the Early Iron Age. The investigation of the terrace shows that it is man-made but there are no traces of any proper occupation layer or of any features from a building. There are pairwise depressions visible on the surface of the terrace that were suspected to indicate the location of pits for roof-supporting posts, but the ones that have been excavated have been refilled more or less naturally. More importantly, there is no evidence of any post ever to have been placed in that pit. It is as if someone started the project of constructing a terrace but then for some reason abandoned the project, and maybe the whole settlement.

Dating materials

The site is located approximately 18.5 m.a.s.l. which, compared to the Holocene post-glacial shore displacement of eastern middle Sweden,

corresponds to c. 1100–1000 BCE (Karlsson & Risberg 2006; Åkerlund et al. 1995; Risberg et al. 1991). That provides us with a very convenient *terminus post quem*; the site simply cannot have been occupied before that.

In order to get the precision and accuracy in the dating we needed a datable material formed at a very precise radiocarbon event very close to the archaeological event we wanted to date. Plant macrofossils can be such a material if found in a closed archaeological context. An observation made during fieldwork was the low but virtual omnipresent occurrence of small pieces of carbonized plant materials in the soil in general. This was observed both during the excavation and during soil probing in the surrounding area, initiating the suspicion that the area had been subject to forest fire since its abandonment. The frequency of forest fires has both natural and cultural bases (Granström & Niklasson 2008). The most common natural cause is ignition by lightning, which has been estimated to occur with a frequency of once every 5th–20th year per 100 km^2 in the Boreal forest (Granström & Niklasson 2008, p. 2354). Also, throughout history people have burned the forest to keep the land in good condition for grazing. It has been observed that when land-use changed from grazing to forestry, the frequency of forest fires decreased abruptly in the mid-19th C CE. From investigations of vegetation history in sediments of the lake Fjäturen, located c. 500 m north of the site at Påtåker, there is evidence of a steady increase in charcoal particles from c. 770–400 BCE, interpreted as the start of human fire activities in the area (Karlsson & Risberg 2006, p. 85). Over the c. 3 000 years since the site at Påtåker came out of the water it is highly likely that it has been exposed to several forest fires, natural or man-made, which made us concerned regarding the sampling of carbonized plant materials for radiocarbon dating.

In spite of this several contexts were sampled for plant macrofossil analysis when the site was investigated. These yielded a quite poor material, with no carbonized grains that could be identified to species. This, in combination with the suspicion of historic forest fires in the area, made us look for complementary materials to analyse as well. Pottery is a fairly frequent find category from the excavation. However, there were no organic surface residues or food crusts on any of the potsherds found at this site.

All potsherds were sampled for organic residue analysis in the field, never handled with bare hands, wrapped in aluminium foil before placed in plastic bags and put in a freezer the same day in wait for analysis. The sampled pottery was registered in detail in the laboratory, sherds from thirty different vessels identified, sampled and analysed for adsorbed food lipid residues (Wehmer 2016; Jansson 2017).

Compound specific radiocarbon dating of singular fatty acids from archaeological pottery has been proven to be a fruitful approach (Hedges et al. 1992; Stott et al. 2001; 2003; Berstan et al. 2008; Casanova et al. 2017; 2018; 2020). This earlier research has shown that the radiocarbon dating of food lipid residues from prehistoric pottery is possible. But the compound specific approach is quite laborious. The isolation of singular lipid components for radiocarbon dating is necessary if the lipid residue is contaminated by post-excavational handling (e.g. fingerprints, packing materials, glues, lacquers, etc.). That is not the case with our samples and we decided to investigate if any of the potsherds contained any food lipid residues suitable for direct radiocarbon dating.

Method

All thirty potsherd samples were analysed for food lipid residues using a standard ultrasonic aided solvent extraction, followed by the derivatization of extractives into trimethylsilyl ethers/esters and the subsequent analysis by gas chromatography mass spectrometry (cf. e.g. Isaksson 2018). The lipid residue yield was recorded and extracted components identified, these molecular results are presented in table 1. For the attempt to radiocarbon date food lipid residues priority was given to samples with high lipid yields. There was a distinct difference in lipid content between the potsherds from the two house foundations, with higher yields from foundation 3, and we chose to concentrate our sampling to this foundation. Four potsherds (F33, F72, F80 and F83) containing remarkably

Sample	SiteArea	Cont.	Type of sherd	Lipid(mg/g)	FA	C18:0/ C16:0	ia BR
F5	3	2	Body	0.7	12 (18) 30	1.09	x
F14	3	2	Body	0.1	14 (18) 28	2.44	x
F15	3	1	Body	0.03	14 (18) 24	1.2	x
F20	3	1	Body	0.3	14 (18) 28	1.5	x
F28	3	2	Rim	0.03	12 (18) 26	1.2	x
F33	3	4	Shoulder	1.8	12 (18) 28	5.6	x
F36	3	2	Rim	0.2	12 (18) 22	2.01	x
F52	3	2	Body	0.3	12 (18) 32	2.59	x
F65	3	2	Rim	tr	tr	tr	n
F66	3	18	Body	0.2	12 (18) 26	1.7	x
F72	3	2	Body	1.2	12 (18) 20	1.59	x
F73	3	2/17	Body	0.5	12 (18) 35	2.66	x
F75	3	2/17	Rim	0.5	12 (18) 28	2.03	x
F80	3	2/21	Body	3.5	12 (18) 35	1.74	x
F82	3	2/21	-	tr	tr	tr	n
F83	3	2/21	Body	1.5	10 (18) 35	1.51	x
F94	1	31/34	Rim	0.06	12(18)20	1.01	x
F100	1	33	Body	0.17	14(18)28	0.51	x
F119	1	40	Body	0.07	14(18)18	0.94	x
F114	1	36	Body	0.05	15(16.28)30	0.7	x
F127	1	36	Rim	0.09	14(18)30	1.12	x
F136	1	36	Body	0.08	14(18.24)30	1.16	x
F138	1	36	Rim	0.07	14(16)24	0.89	n
F140	1	36	Rim	0.06	14(18)30	1.42	x
F148	1	31	Body	0.06	16, 18	0.86	n
F152	1	36	Rim	0.06	14(16)28	0.76	x
F158	1	51	Body/Shoulder	n	n	n	n
F169	1	31	Body	0.07	16, 18	0.49	n
F173	1	40	Body/Shoulder	0.05	16, 18	1.29	n
F185	1	59	Body	0.05	16, 18	0.62	x

Tab 1. The result of the molecular analyses of lipid residues in potsherds from Påtåker. Lipid ressidue concentrations are given in milligrams of extractives per gram of ceramic sample. **FA** indicates the distribution of free (straight-chained and saturated) fatty acids, described in the format k(m)n, where k is the number of carbon atoms in the carbon chain of the shortest fatty acid, n of the longest and m the carbon chain length of the dominant fatty acid. Since terrestrial animals produce more stearic acid (C18:0) in comparison to most aquatic animal and plants a high **C18:0/C16:0** ratio indicates that the fatty acid composition is dominated by terrestrial animals. **ia BR** indicates which iso- and anteiso-branched fatty acids are present in the sample. **DA** indicates the dicarboxylic acids (carbon chain-lengths) present in samples. **TAG** indicates the range of acyl carbon lengths of intact tryacylglycerides detected, where a wide distribution (40-54) indicate dairy lipids. **IPFA** indicates which isoprenoid fatty acids are present in the sample; Phy = Phytic acid, Pri =

	DA	TAG	IPFA	APFA	LCK	Chole.	Phytost.	LCAL	Terp.
	n	40-52	Phy	n	29-35	X	tr	22-30	DT
	n	44-50	Phy	n	tr	tr	n	22-32	DT
	n	n	n	n	n	n	n	22-30	DT
	tr 9	40-52	Phy	18	n	n	tr	22-30	DT
	n	n	n	n	n	n	x	22-28	tr DT
	8.9.10.11	n	Phy	18	29-35	X	n	22-32	DT
	n	tr	n	n	n	n	n	22-32	DT
	9	tr	Phy	18	31-35	n	x	22-30	DT
	n	n	n	n	n	n	tr	tr	tr DT
	9	46-52	n	n	n	n	n	22-30	N
	7.8.9.10	n	Phy. Pri	n	n	X	x	22-28	DT
	9. 10	40-54	Phy	n	33	X	n	22-30	tr DT
	9.1	n	Phy. Pri	18	29-35	X	n	22-30	DT
	n	40-54	Phy	18	n	X	x	22-32	DT
	n	n	n	n	n	n	n	tr	n
	9	40-54	Phy	n	n	X	tr	22-30	n
	n	n	n	n	n	n	x	n	tr DT
	9	n	Phy	n	n	x	x	22-28	DT
	n	n	n	n	n	n	n	n	tr TT
	n	n	n	n	n	n	tr	22-26	tr TT
	n	n	n	n	n	n	n	24-32	tr DT. tr TT
	n	n	n	n	n	n	n	n	TT
	n	n	n	n	n	n	n	n	TT
	n	n	n	n	n	n	tr	22-30	TT
	n	n	n	n	n	n	n	n	tr TT
	n	n	n	n	n	n	tr	22–32	tr TT
	n	n	n	n	n	n	n	n	n
	n	n	n	n	n	n	n	n	tr TT
	n	n	n	n	n	n	n	n	n
	n	n	n	n	n	n	tr	n	n

pristanic acid. APFA indicates the carbon chain lengths of ω-(o-alkylphenyl)fatty acids found. **LCK** indicates between which carbon chain lengths long chain ketones are found. **Chole.** indicates in which samples cholesterol, or its decomposition products, that has been detected and **Phytost.** indicates in which samples plant sterols, or their decomposition products, that has been detected. **LCAL** indicates the range of carbon lengths of long-chain alkanols detected. **Terp.** indicates in which samples terpenoids are detected; DT are diterpenoids (mainly dehydroabietic acid and related compounds), TT are triterpenoids. An "x" indicates that the substance could be detected, an "n" indicates that the substance could not be detected, and a "tr" that there are possible traces.

high concentrations of absorbed lipid residues were chosen for radiocarbon analysis.

The samples are dominated by lipid residues of terrestrial origin (tab. 1), no sample contains a full range of markers for aquatic animal residues. But there are observable variations which for the testing of this approach is advantageous. Sample F80 and F83 show no evidence of aquatic lipids, both samples instead showing evidence of dairy lipid residues in the form of a wide distribution of intact triacylglycerides. Sample F33 and F72 contain a broad range of short-chain dicarboxylic acids that could indicate contribution from aquatic oils. Sample F72 also contains two isoprenoid fatty acids which indicate a contribution from aquatic animal residues. These two samples were chosen in order to allow the detection of any reservoir effects.

One of the potsherds (F72) was found in the deposited culture layer (context 2) on foundation 3 (fig. 2), another (F33) was found in a sandy layer (context 4) that is superimposed by context 2 and partially covers the stone set foundation (context 21), and two (F80 and F83) were found in the interface (context 2/21) between the deposited culture layer (context 2) and the set stone foundation (context 21). This provided a stratigraphically sound sample of this house foundation.

Unidentified charred seeds from three contexts from foundation 3 (fig. 2) were also selected for radiocarbon analysis. These were recovered through floatation and sieving of macrofossil samples from the fill of a roof supporting post (context 11), the fill of a wall-post (context 14) and from the fill of a waste-pit (context 17).

For the extraction of lipid residues from the potsherds for radiocarbon dating a modified version of the so called Folch's method (Folch et al. 1957; Eggers et al. 2016) was used, adapted for the isolation of lipid residues from the clay matrix of potsherds and their simultaneous purification from exogenous contaminations. Based on the lipid residue analysis (cf. tab. 1) an amount of ceramic powder estimated to provide enough lipid residues for radiocarbon analysis was homogenized through sonication in a mixture of chloroform and methanol (2:1, v:v). The non-extractable residue and the liquid phase were separated through centrifugation (3000 rpm, 30 min.) and the liquid phase was collected. This process was repeated three times and the extracts were combined. The collected liquid phase contains a large range of organic compound classes, both apolar compounds (e.g., lipids, terpenes, etc.) and polar components (e.g., sugars, dicarboxylic acids, etc.) (cf. Medeiros & Simoneit 2007) due to the usage of methanol as a polar component in the extraction mixture. However, the inclusion of this polar solvent is important for the efficiency of this method as it improves the solubilisation of lipid molecules (Eggers et al. 2016:2). But this has the obvious drawback that it also extracts polar components, which in the present samples would be a problematic mixture of compounds from the burial environment (cf. Medeiros & Simoneit 2007) and from food processed in the vessels. The exchange of apolar lipids between the burial environment and buried potsherds has been shown to be negligible (Heron et al. 1991) but polar components can be transported through the percolation of groundwater. To purify the lipid residue extracts deionized water was added to the chloroform/methanol mixture, and shaken vigorously. Allowed to settle, a new aqueous phase was formed. In this upper water and methanol phase hydrophilic components and salts are enriched, while the lower chloroform phase retains the lipids (Eggers et al. 2016, p. 2). The aqueous phase was discarded and the washing was repeated two more times with new pure water. The purified lipid extracts were blown down to a few hundred microliters under a gentle stream of nitrogen gas. The highly concentrated extracts were then pipetted directly into pre-weighed tin foil capsules for radiocarbon dating and very carefully blown down to dryness. The potential problems resulting from the incomplete removal of carbon containing organic solvents prior to radiocarbon analysis has been recognized for applications of compound specific radiocarbon dating (e.g. Casanova et al. 2018, p. 11028) and is equally relevant for this approach. In order to reduce this problem and maximize the removal of solvents the tin foil capsules were heated to 70 °C, i.e. c. 10 °C above the boiling point of chloroform, for one hour. The tin foil capsules were then allowed to cool and were weighed to check the lipid residue

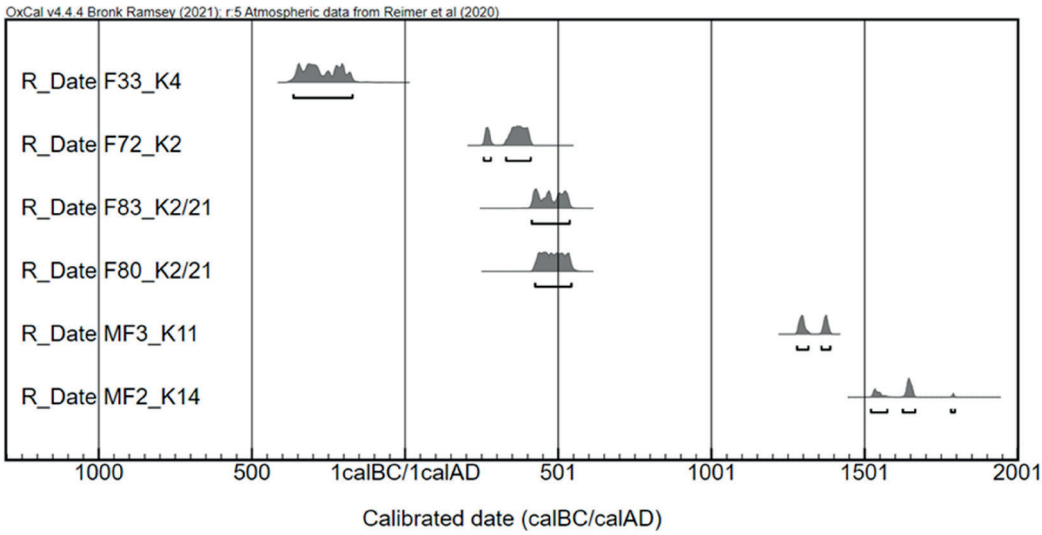

Fig. 3. Probability distributions of the calibrated radiocarbon dates (tab. 2 & 3).

yields before being sent to radiocarbon analysis at the Centrum voor Isotopenonderzoek (CIO), University of Groningen, the Netherlands. The extracted lipid samples in tin cups were not chemically pre-treated by the CIO laboratory, but only combusted, graphitized and measured by Accelerator Mass Spectrometry (AMS) using a Mini Carbon Dating System (MICADAS). The charred plant remains went through standard pre-treatment procedures (Acid/Alkali/Acid or Acid wash; www.rug.nl/research/ centre-for-isotope-research/research/radio-carbon-dating) for the radiocarbon analysis of such materials at the CIO laboratory.

Results

The results of the radiocarbon analysis and of the bulk $\delta^{13}C$-analysis of all samples are presented in table 2. The radiocarbon dates of all samples were calibrated to calendar years using the online OxCal 4.4 software, with the exception of one of the carbonized seed samples (MF1) since it produced a too young radiocarbon age. The results are summarized in table 2 and figure 3.

Discussion

All three of the carbonized seed samples came out with dates much later (i.e. dated to historical times) than expected in comparison to the archaeological evidence of the site (i.e. Early to Middle Iron Age). It is important to not dismiss radiocarbon dates solely on the grounds that they do not fit with the expected dates. On historical maps, stretching back to the mid-19th century CE, there is no evidence of any buildings at the site, even though there are written records of a croft in the vicinity from 1666 CE. The location of this croft is known, and it is located c. 200 m north of the archaeological site, still occupied by buildings. Though we cannot wholly exclude activities in historical times at the archaeological site it is fairly unlikely that the features from which these soil samples were collected are the remains of historical buildings. The architecture they derive from is prehistoric, typical for the Early to Middle Iron Age of the region. The charred seeds are certainly small enough to allow them to be transported in the sandy soil, e.g. earth-worms tunnelling in the soil may definitively transport seeds between strata and contexts. The radiocarbon event, i.e. when these seeds got cut off from the carbon cycle, is not equivalent to the archaeological event, i.e. when the buildings were in use. The potsherds analysed in this study were all large enough to be much less mobile in the soil, and

Sample	Context	Sample type	Pretreatment	Project ID	GrM	^{14}C Age(yr BP)	± 1 δ	
F33	4	Lipid residue	Mod. Folch's	ml068	19643	2196	28	
F72	2	Lipid residue	Mod. Folch's	ml067	19912	1705	21	
F80	2/21	Lipid residue	Mod. Folch's	ml066	19911	1587	21	
F83	2/21	Lipid residue	Mod. Folch's	ml065	19910	1614	24	
MF1	17	Carbonized seed	AAA	ml069	20206	>1950AD	-	
MF2	14	Carbonized seed	A	ml070	20207	269	22	
MF3	11	Carbonized seed	A	ml071	20208	670	22	

the results of the radiocarbon analyses look very promising.

However, as erroneous as dismissing radiocarbon dates solely on the grounds that they don't fit one's expectations is also to accept radiocarbon dates solely based on the fact that they do so. The δ^{13}C-value of the extracted lipids should be in accordance with the origin indicated by the lipid residue analyses. As already stated sample F33, to some extent, and, in particular, F72 showed evidence of aquatic contribution from the molecular analysis and do also show higher δ^{13}C-values compared to samples F80 and F83 which in the molecular analysis showed contribution from milk lipids instead. Even though the δ^{13}C-values of F33 and F72 are not as high as the generally accepted limit for lipids of marine origin (i.e. > -25 ‰) an aquatic reservoir effect could be expected regarding these samples. Sample F72 is contextually close (found in the fill of context 2) to samples F80 and F83 (found in the interface between context 2 and context 21). Sample F72 is about a hundred ^{14}C years older than F80 and F83. This is an age difference that fits reasonably well with reported reservoir effects for the Baltic Sea (Glykou et al. 2021, p. 5). Sample F33 is considerably older than what could be expected from a reservoir effect from the Baltic Sea alone, producing a radiocarbon date a few decades later than the earlier observed start of human fire activities in the area (c. 770–400 BCE; Karlsson & Risberg 2006, p. 85) and a century and a half before the establishment of the stone fence system (c. 0 CE; Johansson & Lindgren 2005, p. 15). Sample F33 is found in context 4; a sandy fill that superimposes context 21 and is superimposed by context 2. This sandy fill, used to partially cover the stones of context 21, must have been placed there in connection with the construction of foundation 3. This fill most probably originates nearby and the radiocarbon date of F33 could show that there were activities at the site prior to the construction of foundation 3.

The carbon content of a lipid residue sample should not deviate considerably from the theoretical carbon content range of lipids. The carbon contents of some potentially relevant compound classes are presented in table 3. Two of the lipid residue samples (F72 & F83) have a carbon content that is within the range of pure lipids, the other two are too low (F33 & F80) (cf. tab. 2 & 3). This could be the result of an unsuccessful removal of polar components in the cleaning process. It could also be the result of an unsuccessful removal of solvents, i.e. methanol and chloroform. Methanol is today usually produced through hydrogenation of carbon dioxide while chloroform is produced through the chlorination of methane. Since chloroform is a petroleum-derived product and therefore contains no radiocarbon a few per mill of carbon from the solvent could result in a date several decades older than the true age (cf. Casanova et al. 2018, p. 11028). However, the lower carbon content could also be the result of remaining colloidal mineral particles from the powdered ceramic, in which case no differing carbon would be added.

Plastics from packing materials, etc., have varying carbon content, from c. 40% in chlorinated polymers to between 60 and 90% in the most common plastics (Smeaton 2021). Since

	%C	$\delta^{13}C$(‰; IRMS)	±1 δ	Calibrated range BCE/CE
	64.8	-28.62	0.21	365–172 BCE
	74.4	-28.77	0.15	258–411 CE
	54.0	-31.21	0.15	415–539 CE
	73.9	-31.06	0.15	425–544 CE
	60.0	-27.15	0.17	>1950 CE
	45.8	-27.44	0.17	1522–1795 CE
	55.0	-27.58	0.17	1280–1389 CE

Tab. 2. The result of the radiocarbon analysis. Calibration of the radiocarbon dates was made using the OxCal 4.4 software (Bronk Ramsey 2021) and atmospheric data from Reimer et al. (2020). The calibrated range presented is with 95.4% probability. Pre-treatment; "Mod. Folch's" is the method presented in this paper, "AAA" is the Acid/Alkali/Acid wash and "A" is the Acid wash.

Compound (class)	Theoretical carbon content (weight %)
Poly Ethylene (PE)	c. 92
Resins etc. (di- triterpenoids)	79–85
Phtalates (plasticizers)	74–75
Lipids	72–79
Lignin, cutin & suberin	62–72
Humus	c. 60
Carbohydrates	40–44
Proteins, peptides, amino acids	30–64
Polyvinyl chloride(PVC)	c. 38
Methanol	37.5
Chloroform	10.1

Tab. 3. The carbon content range of different organic compound classes, sorted by descending carbon content. The theoretical carbon content has been calculated from the molecular formulae of the most common compounds in each class.

plastics are commonly made from petroleum they would contaminate a sample with very old carbon resulting in too old radiocarbon dates. The current samples have not been in direct contact with plastics and there was no evidence (e.g. traces of plasticizers) of plastic observed during the molecular analyses. Colloidal particles of plastics in the solvents could pass undetected if too large to be detected by the GCMS.

The purified lipid extract approach to direct dating of pottery can be used on potsherds with high lipid yields, preferably from singular biospheres (i.e. atmospheric or aquatic), and with a minimum of post-excavational contamination. The single compound approach (e.g. Casanova et al. 2018; 2020) requires even higher yields since only two fatty acids (palmitic (C16:0) and stearic acid (C18:0)) are dated instead of the entire lipid extract. The single compound radiocarbon dating approach should be preferred for samples that are contaminated by e.g. plasticizers from packing materials. Also, the single compound radiocarbon dating approach yields two dates per sample, of the palmitic and stearic acid respectively, which is used as a quality control since the results are only accepted when the two dates agree. However, since the purified lipid extract approach does not involve any derivatization of the lipids, no mass-balance correction for the methyl group added in the derivatization of fatty acids is needed, as is the case with the compound specific approach. Also, the problems of contamination from column bleed and cross-contamination during the preparative Gas

Chromatography applied in the compound specific approach do not apply for the purified lipid extract approach. There is, however, room for improvement of the purified lipid extract approach when it comes to the purification process and especially in the development of procedures to establish sample pureness after extraction but before radiocarbon analysis. In single compound dating Nuclear Magnetic Resonance (NMR) is used to control the purity (Casanova et al. 2017) which works fine since the sample contains singular components. This might not be as applicable to the purified lipid extract approach since each sample contains multiple lipid compound classes. To test the reliability of the procedures, samples of lipids of known age (old or recent) should be analysed in parallel with unknown samples, as in-house standards and method blanks.

Of the four lipid residue samples, sample 83 had a carbon content in range and showed evidence of purely terrestrial carbon both in the molecular and the stable carbon isotope analysis. This potsherd, found in the interface between the stone-set foundation and the accumulated culture layer of house 3, comes from a ceramic vessel that was used to process food (including milk fats) which with 94.5% probability left the carbon cycle (i.e. was harvested) in the calibrated range 415–539 CE. In the already mentioned investigations of vegetation history recorded in sediments of Lake Fjäturen (Karlsson & Risberg 2006) the oldest traces of a grazing landscape date from around 700 BCE and the oldest traces of cultivation are from around 300 BCE. The traces of plants associated with grazing are patchy all the way into the present, the cultivation traces on the other hand cease around 500 CE and then return in the early Middle Ages. The survey also shows that Lake Fjäturen was separated from the sea around 200 CE (Karlsson & Risberg 2006, p. 111), meaning that the lakes at the site got cut off from the Baltic Sea for good.

That the water system at which the site Påtåker is located loses its connection to the sea at around 200 CE is not the direct cause of the site's abandonment since it is still in use 415–539 CE. This loss was, perhaps, not directly ruinous but may have had a protracted effect. Given the range of the lipid residue date it is very tempting to connect it to the 536 CE climatic event. However, contemporaneity does not imply causality. Also, this the youngest radiocarbon date of the lipid residues in the potsherds provides a *terminus post quem* for the site to that time range.

Milk lipid residues are found in five out of thirty analysed vessels from the site and aquatic animal residues are found in three (tab. 1). Previous analyses of soil samples from fossilized fields at the site have shown that these areas were fertilized (Isaksson & Fjellström 2016). This is evidence of dairying and the use of manure from cattle to uphold the small fields, supporting the importance of cattle for the system. There is also evidence for the processing of fish, and another importance of aquatic resources rather than as waterways alone. The pottery-use pattern of the site is fit for fishing milk farmers, locked in an inflexible land-use system situated in a shallowing archipelago. The disconnection of the close-by lakes from the sea may have affected the fishing at least qualitatively and maybe also quantitatively. A timeworn practice of inflexible land-use will have leached the soil of the fields making the system heavily reliable on manuring. This will have caused a strong reliance on the access to animal fodder and land for grazing. A few years of restricted vegetal primary production due to cold summers caused by the 536 CE climatic event would have had a serious effect on the access to animal feed, which would cause a decline in livestock, which would result in a shortage of manure for the fields. If the fields don't get enough manure the crop will fail, and the system is in a negative spiral that would be very hard to reverse without major change. And a major change came, in both land-use and social structure.

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