CERAMIC CHRONOLOGY IN VIEW OF ^{14}C DATINGS

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ABSTRACT

The chronology of the Pitted Ware Culture along the Swedish Baltic coast has earlier mainly been determined by land uplift data and classifications based upon pottery styles. No direct dating of the pottery itself has previously been possible and only an indirect chronology built on associated material exists. The present investigation presents a direct radiocarbon dating of four sherds from the reference sites Säter and Fagervik at the bay of Bråviken in Östergötland, representing the Säter/Fagervik II and III phases. The recently developed accelerator dating technique has enabled measurements on small amounts of carbonised material ("food crust") on the inside of the potsherds. Different chemical pretreatment procedures in order to extract the most relevant organic fraction have been studied and possibilities for future improvements are discussed. The analysis shows that the organic material is of terrestrial origin. The ages of the four investigated sherds are not possible to differentiate chronologically. They fall within the time interval 4400-4600 BP (3100-3350 cal BC) which is in accordance with expected ages for the phase II, but older than expected for phase III.

Introduction

Few artifact groups have been subjected to so much typological and chronological analysis as Neolithic pottery. On the basis of ware type, ornamentation and shape, the pottery has been attributed to certain cultures and placed in different chronological phases. The assemblages of the Pitted Ware sites along the Swedish Baltic coast consist mainly of pottery. Principally on the basis of height above sea level, they have been divided in three chronological periods - the Säter or Fagervik II, III and IV phases. These phases are also associated with particular pottery styles of the same names, but the styles have never been properly defined and their chronological standing is far from settled.

The Middle Neolithic Säter/Fagervik chronology

The chronology of the Pitted Ware culture which grew out of the excavations of the three Säter sites in Östergötland at the beginning of the 20th century is still more or less current. At that time geologists and archaeologists were beginning to seriously consider shoreline displacement as important for the choice of settlement site location. According to this well-known theory, the coastal settlements of hunters followed the shoreline, as it moved down to lower and lower levels during the Litorina period. Unfortunately the investigations of the Säter sites have only been published in short papers (Almgren 1906a;23ff, 1906b;118,Nerman 1911;6 ff, 1927, Nihlén 1927;143 ff, Engström 1934;192ff). The sites lie at a distance of not more than 300 m from each other, and they do not reach below 27.5, 26 and 24 m a.s.l respectively. The pottery from the different sites exhibits certain differences of ware, shape and ornamentation, and the terms Säter II, III and IV were adopted to designate different variants, "styles",of pitted ware. Shoreline displacement was regarded as proof of the chronological differentiation of the styles. Thus, a relative chronology for the Pitted Ware Culture came into being, a chronology which was also thought to be applicable outside Östergötland. In general terms, the pottery of the three Säter phases can be described as follows:

Fig 1. The location of the Säter and Fagervik sites.

By establishing the lowest level for the most common pottery types at the rich Fagervik site in Östergötland, Axel Bagge claimed to observe five settlement phases, Fagervik I-V (Bagge 1951). Each phase is characterised by its special pottery style. The pottery of Fagervik II, III and IV largely corresponds to that of the equivalent Säter styles. The phases Fagervik I and V have been added. Fagervik I, characterised by pottery reminiscent of funnel-beaker pottery, represents a transitional phase between the Funnel Beaker Culture and Pitted Ware Culture, while Fagervik V, with its battle-axe pottery, belongs to the Battle-Axe Culture. Bagge's methods have been called into question, primarily by Malmer, who however, together with many others, sees the chronological scheme as a more or less true reflection of the actual development (Malmer 1962;721ff, Meinander 1964;20ff, Löfstrand 1974;4ff).

However, it is important to realise that the Säter/Fagervik styles do not originate in the classification of distinctive types, but rather that they are the result of dividing up the pottery according to the level at which it was found. The stylistic grouping of the pottery is therefore questionable. Many motifs are found on more than one settlement level and in more than one period, which makes it difficult to decide whether the levels contain chronologically mixed material or the motifs span over several periods.

Before the introduction of radiocarbon dating into archaeology during the 1950s', the Pitted Ware Culture was thought to have resulted from immigration, the culture lasting for only a few hundred years, roughly 2300-1900BC (Stenberger 1964;94ff). The study of radiocarbon dates, now available for a number of sites along the Swedish east coast, shows that they span a much longer period and, above all, that they go back further. Several datings suggest that settlements existed as early as 2600-2700 BC (Welinder 1973, 1978;106, Wyszomirska 1984;56 f, Burenhult 1982;163, Segerberg & Possnert 1990, unless otherwise stated, dates are uncalibrated). The Pitted Ware settlements seem, therefore, to have been contemporaneous with settlements of other cultural groups, and they have gradually come to be interpreted as the hunting sites of a cultural complex

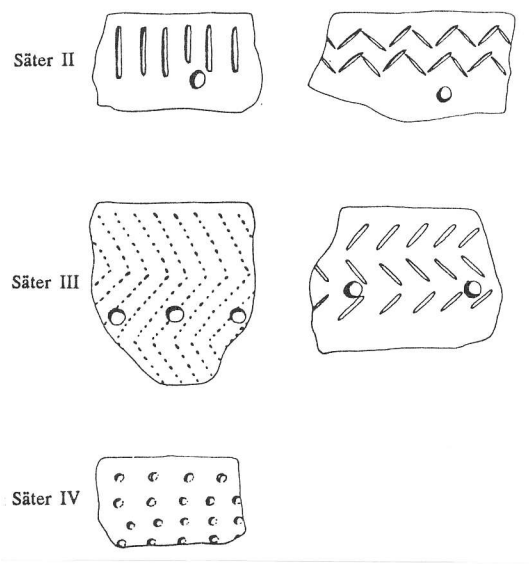

Fig 2. Pottery motives from the Säter sites.
Säter II, Alternately inclined lines and impressions, zigzags etc on the upper part of the vessel. Deep pits. The clay is tempered with granite. **Säter III**, Incised lines or fine combimpressions alternately inclined. Deep pits. The clay is tempered with granite or limestone. **Säter IV**, Shallow pits or combimpressions covering the whole surface. No pits The clay is tempered with limestone.

encompassing both agriculture and hunting (Skaarup 1973;143ff, Welinder 1976, 1978, Burenhult 1982;158 ff). However, it should be pointed out that the association between the existing radiocarbon dates and the features meant to be dated is seldom discussed in the literature. The above dates for the Pitted Ware Culture as a whole should be seen as the result of a number of analysed samples collected more or less at random from a large number of excavations yielding pitted ware sites. Samples have been submitted to different radiocarbon laboratories and have often undergone more or less uncritical analysis, where for example, the chances of receiving a correct date from the analysed material are not adequately clarified.

With a small number of exceptions, it would appear to be evident that virtually no radiocarbon dates can be associated with the pottery itself or even with particular settlement phases. There is seldom any observable archaeological stratigraphy in the cultural deposits of the sites. This means that radiocarbon datings do not result in a direct dating of the actual pottery.

Thus, no absolute dates from the different Säter/-Fagervik stages exist, although some suggestions have been presented (Welinder 1973;114 ff). If the styles ever really existed, we can have very little idea of whether they were chronologically separated. It is also impossible to know whether the styles overlapped. Hence dates which depend entirely on the traditional pottery "styles" should be avoided. The typological analysis of the pottery should be reconsidered, and efforts must be made to find a method of dating samples of the pottery itself.

Radiocarbon dating of pottery

By radiocarbon analysis of carbonised material, usually interpreted as food remains, which is sometimes encountered on the inside of potsherds, the chronological questions can be tackled in a new way. This material ought to be of about the same age as the pot. With such an analysis it is possible to date the pottery itself and in this way test the ceramic chronology.

Sporadic analyses of this kind have previously been conducted, for example on Ertebölle pottery which sometimes has relatively thick crusts (Andersen & Malmros 1985, Arrhenius & Lidén 1989). As food remains on Neolithic pottery are not generally quantitatively adequate for conventional radiocarbon analysis, it was not until the advent of the new AMS (accelerator mass spectrometry) technique that dating of these sherds became possible.

Thus, together with the Archaeological Research Laboratory in Stockholm, the Department of Archaeology and the Tandem Accelerator Laboratory in Uppsala launched a joint project to study the chronology of the Pitted Ware Culture using scientific methods-radiocarbon analysis. An examination of some site assemblages in the Museum of National Antiquities in Stockholm showed that the Säter assemblages in particular contained a relatively large number of sherds with thick food crusts. To start with, four sherds were selected, three from the Säter sites and one from the Fagervik site, for an initial pilot study. The goal was to develop methods for the pretreatment of this type of material, since the demands on pretreatment of the food crust are higher than those placed on, for example seeds, shells and charcoal. For this reason, it was important in this first study to use samples of the highest possible quality, ie the food crust should preferably form an unambitious thick layer on the sherd. The selection of samples was therefore primarily dictated by "crust-quality" and in the second place by the ware and ornamentation of the ceramics. One of the four selected sherds has a type of ware and patterns characteristic of the Säter II style (fig 3) and the other three have a type of ware and patterns characteristic of the Säter III style (figs 4-6).

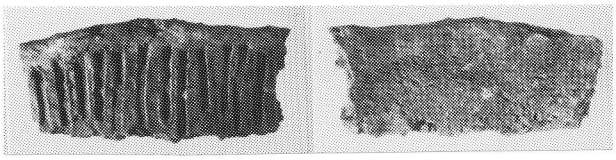

Fig 3. Sample sherd Ög 258 Ua 1729, Ua 1734. Säter II site, Kvarsebo parish, Östergötland, SHM 15101.Rim sherd. Granite temper. Ornamentation of deeply incised vertical lines below the rim. The sherd can be classified as Säter II pottery. 1:2.

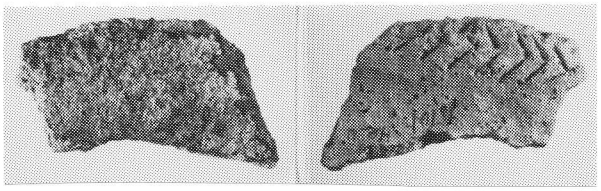

Fig 4. Sample sherd Ög 247. Ua 1727, Ua 1732. Säter II site, Kvarsebo parish, Östergötland, SHM 15101.Shoulder. Limestone temper. Ornamentation of line-drawn herring-bone pattern on lower portion. The sherd can be classified as Säter III pottery. 1:2.

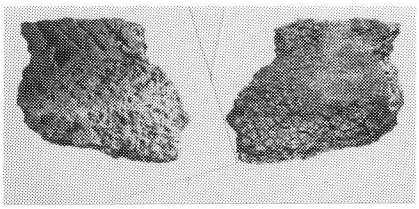

Fig 5. Sample sherd Ög 254. Ua 1728, Ua 1733. Säter III site, Kvarsebo parish, Östergötland, SHM 12931.Shoulder. Limestone temper. Ornamentation of line-drawn herring-bone pattern and pits. The sherd can be classified as Säter III pottery. 1:2.

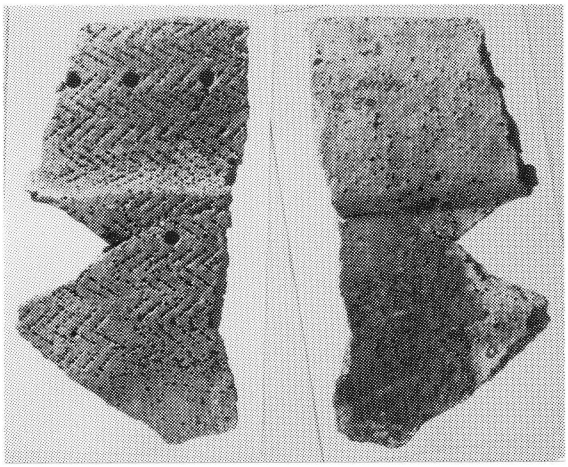

Fig 6.Sample sherd Ög 143. Ua 1726, Ua 1731. Fagervik site, Krokek parish, Östergötland, SHM 21049.Part of rim, shoulder and belly. Limestone temper. Ornamentation of comb stamps forming herring-bone pattern and pits. Bagge assigned the sherd to the Fagervik III stage, so it can be classified as Säter III pottery. 1:25.

Chemical pretreatment

If a sample is to be radiocarbon dated it is a basic prerequisite that the quantity of the radioactive isotope has only been altered by the process of radioactive decay. In other words, the sample must represent a closed system. In practice, this means that carbon which has, for instance, been added to a sample during deposition in the soil, must be removed chemically before an age determination can be made. Another example of the effect of false carbon is the admixture of organic compounds during fieldwork (mould, atmospheric carbon dioxide, etc) or by conservation when handled in the museum.

Regarding the organic remains on pottery, the subject of this study, several different sources of non-relevant carbon are possible. Humic substances in the cultural deposit of the excavated site, bacteria in the museum stores etc have to be considered. Due to this relative complexity, the chemical pretreatment should be as advanced as possible, so that the carbon which is ultimately dated really is contemporaneous with the use of the pottery.

The organic substance, in our case found on the inside of the pottery, consists almost exclusively of food traces at differing stages of carbonization. The internal age of the material should therefore be low or even negligible if terrestrial material have been used. However, some care should be observed on account of the reservoir effects of marine components. In the case of Säter/Fagervik, which is situated on the Bay of Bråviken, this effect can be estimated to not more than c 100 years during the Neolithic to be contrasted with c 400 years at sites on the west coast of Sweden. The natural mass fractionation ($\delta^{13}C$) of the sample also gives some indication of the presence of a marine component. This will be discussed below.

With experience from earlier investigations (Segerberg & Possnert 1990) of similar material from Neolithic sites at Bälinge Mossar, north of Uppsala, we thought that it would be desirable to study several different fractions of the "food crusts" in order to exclude possible effects of contaminating carbon. Despite the small amount of organic remains and the simple pretreatment procedures applied, a successful dating of all the studied sherds was obtained in that investigation, which clearly demonstrates the strength of the method.

Studies of diet composition using advanced organic

chemistry to divide food crusts into their component parts (Arrhenius 1985) have also shown that these systems are relatively well preserved. Despite deposition in the soil, relatively large amounts of, for example, intact protein were obtained. This may be why, in the case of the Bälinge Mosse material, simple chemistry produced positive results, comparable to the age of hazelnut shells and charcoal from the same cultural deposit. However, conditions of preservation vary at different sites, so every situation must be judged separately.

The way the archaeologist handles the material, right from excavation until it is submitted to the dating laboratory, is always of utmost importance. Food crusts from many earlier excavations have probably been washed away when the pottery was being cleaned. Air drying and protection during storage, in order to prevent alteration by micro-organisms and dust to penetrate into the material, is to be recommended. The time that elapses between excavation and age determination should of course be as short as possible.

The refined chemistry approach

In the present study we aimed at extracting different amino acids for the dating purpose in order to investigate the age distribution of these compared with the humics and carbonised fractions of the food crust. The main problem is, however, to obtain enough material in each subsample to be able to conduct the radiometric analysis. Even the low requirement of 0.5 mg carbon in the AMS-technique is hard to fulfil in this context of thin layers of material on small pieces of ceramics.

The organic remains were first optically classified concerning the colour, thickness, degree of cracks etc, before removed from the sherds under a magnifying glass, weighed and collected in sterile test-tubes (table 1). To get a conception of the quality of the food crust, a sample of 10 mg was taken for protein quantification according to Lowry (1951) (table 1). An amount of the sample corresponding to 500 microgram protein was then hydrolysed over night in 6M HCl, identified with HPTLC (high performance thin layer chromatography), and quantified using a spectrophotometer plotting the result against a standard curve (table 2). The system we usually in earlier investigations used for identification and quantification (Heathcoate et al 1969) was run on plates coated with cellulose layers and the amino acids were then developed with Ninhydrin. This system was useless for the present purpose since the

Table 1. Total sample amount , protein content and total protein amount for the different sherds.

Sample	total amount mg	protein amount mg/100 mg sample	total protein amount mg
Ög 258 SHM 15101	117.40	1.5	1.76
Ög 254 SHM 12931	299.49	1.2	3.59
Ög 247 SHM 15101	164.26	0.9	1.48
Ög 143 SHM 21049	137.00	0.7	0.96

dating method requires samples totally free from contamination of carbon. We therefore tested a new system where the plates were coated with silica gel containing a fluorescence indicator (Merck Silica gel 60 F254, Merck Si 50000 F254). As eluent in two directions we used, n-propanol/H_2O/25 % NH_3 (80/20/1). The sample amount applied on the plates varied between 15 to 20 microliters and were applied with microcaps and Hamilton syringe respectively. The plates were run until the eluent had reached approximately 8 cm and then dried for an hour before a second run in the other direction. The amino acids were developed under UV-light (Hauck & Halpaap 1989). Since this system did not work out perfectly we run a parallel system with Ninhydrin as developer. The amino acids were removed from the plates, after identification, and placed in sterile test-tubes. All work was performed sterilely to minimize the risk of contamination of carbon.

Results of the refined chemistry

A budget was drawn up for dating amino acids, based on a required amount of 1 mg of carbon equivalent and the average exchange of amino acids using our method (fig 7). After the protein quantification it was obvious, that the quality of the protein was too low to permit one specific amino acid to be dated. We therefore mixed all extractable amino acids from one potsherd in the test-tube. Only in the case of potsherd Ög 254 and potsherd Ög 258 was the amount of protein high enough to permit further amino acid extraction. The amino acid pattern of the two sherds differed somewhat in that sherd Ög 258 had a larger number of amino acids than the sherd Ög 254. The amino acids Glu, Ser and Asp were identified on sherd Ög 258 but not on sherd Ög 254. On the other hand, the total amount of amino acids on sherd Ög 254 was higher. It is

Table 2. Amino acid (a.a.) analysis; mg amino acid/100 mg protein.

x= detected; not weighable.
-= not detected.

a.a.	sample Ög 258	sample Ög 254
Thr	-	-
Leu	1.82	1.11
Arg	x	x
Gly	1.07	3.27
Glu	x	-
Cys	-	-
Tyr	-	-
Met	1.75	1.82
Ile	0.47	0.59
His	0.30	0.06
Ser	x	-
Ala	x	4.89
Val	1.54	1.72
Phe	-	-
Lys	-	-
Asp	x	-
Pro	-	-
Total amount aa/ 100 mg protein	6.95	13.46
total amount a.a. in sample (mg)	0.12	0.48

surprising that the amino acid that dominates all nutritive substances, Glu (cf. Livsmedelstabeller 1988), was totally absent on Ög 254 and only detectable on Ög 258. The large amount of Ala on sherd Ög 254 could be explained by the fact that Ala is the simplest of all amino acids and large quantities of it are present in strongly deteriorated products. The remains on sherd Ög 254 might therefore, before deposition in the earth, have been broken down by some natural or man-made process. However, no conclusions can be drawn regarding the origin of the food crust from the amino acid pattern. To be able to say more about the origin of the food crusts, supplementary analyses need to be done, eg trace element analyses and lipid analyses.

To be able to date amino acids with the present method c 1-3 g of food crust is required; to date one specific amino acid even more is needed. Such a great amount of food crust has not yet been found on any sherd in Sweden. The negative result in this test is in conclusion due to shortcomings in the applied amino acid extracting technique rather than an insufficient available amount of material. We will therefore try other methods in order to acquire a higher yield of amino acids and preferably large enough amounts to allow ^{14}C analysis on specific

REFINED CHEMISTRY IN ORDER TO EXTRACT AMINO ACIDS FROM THE FOOD CRUST.

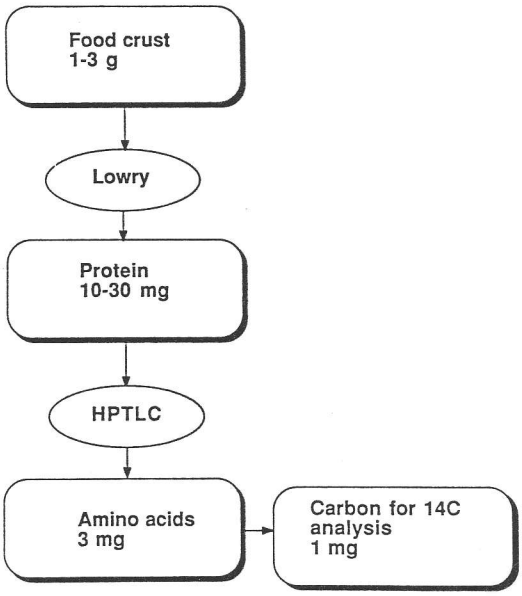

Fig 7. The refined chemistry procedure in order to extract amino acids from the organic remains.

amino acids. Separation by HPCL (high pressure liquid chromatography) gives both a better yield and also a more specified amino acid pattern. It is not hard to adjust the ordinary procedure for extracting amino acids by HPLC to fit the specific requirements of ^{14}C dating.

Conventional pretreatment chemistry

As a consequence of the low yield in the refined chemistry, a conventional pretreatment procedure was also applied to the same material. An acid-base-acid treatment according to the following details was used: 1) treatment with 1% HCl below boiling point for 6 hours-this eliminates carbonates as well as fulvic acids; 2) washing in distilled water; 3) the insoluble fraction was treated with 1% NaOH below boiling point for 6 hours, and the soluble fraction in this step was then precipitated with concentrated HCl, washed, dried and dated as fraction SOL; 4) the insoluble fraction was washed, dried and dated as fraction INS. The SOL fraction normally includes most of the humics from the soil and is thus of minor interest when macrofossils are analysed. From the earlier mentioned pilot study of the Bälinge Mossar potsherds we know, however, that this fraction is highly relevant for the age determina-

Table 3. Results of the radiocarbon analysis and yields in the sample pretreatment chemistry.

lab.code	SHM	sample total (mg)	INS (mg)	SOL (mg)	$\delta^{13}C$ (‰ vs PDB) SOL	INS	^{14}C age BP SOL	INS
Ua-1726	21049	51.6		11.8	-22.0		4560±120	
Ua-1731	21049		13.1			-23.4		4470±80
Ua-1727	15101	53.0		20.7	-23.2		4610±120	
Ua-1732	15101		17.9			-24.7		4490±70
Ua-1728	12931	52.5		32.7	-24.3		4490±120	
Ua-1733	12931		4.0					4470±80
Ua-1729	15101	14.4		2.4			4410±130	
Ua-1734	15101		3.1					4520±80

tion of organic remains on ceramics, probably because several of the organic compounds from the food are extracted in this fraction. The INS fraction, on the other hand, consists mainly of carbonised material from the crust and solids from the soil. The importance of the critical evaluation of crust quality and the removal of visible contaminants on the surface before the chemistry is applied must be stressed.

Before the mass spectrometric determination of the ^{14}C content of the different fractions with the Uppsala tandem accelerator as an ultra sensitive spectrometer was performed, all samples were first converted to CO_2 and finally to solid graphite by an iron-catalytic reaction at 800°C with the presence of hydrogen. The combustion was conducted at 800°C with CuO as an oxidiser. The carbon contents were 50% and 40-60% for the SOL and INS fractions respectively.

The natural mass fractionation was determined with a conventional mass spectrometer (Varian MAT 230S). Due to a shortage of material, only some of the fractions were available for this analysis.

Results of the ^{14}C investigation

A summary of the chemistry as well as the radiocarbon ages of the different chemical fractions is presented in table 3 and in figure 8. As can be seen, all dated fractions have almost the same radiocarbon age. This indicates that the organic content of the food crust is homogeneous as far as the age is concerned. Possibilities for dating are therefore favourable in the present case, and a refined chemistry is thus of less importance. No significant statistical difference is obtained between any fraction or sherd, and an average radiocarbon age of 4500-±40 BP can be calculated.

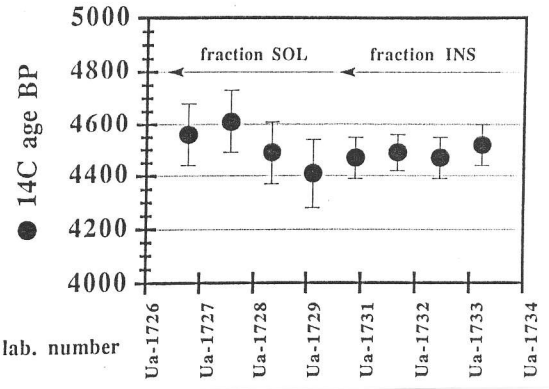

Fig 8. Results of the radiocarbon analysis of the INS and SOL fractions (see text) respectively.

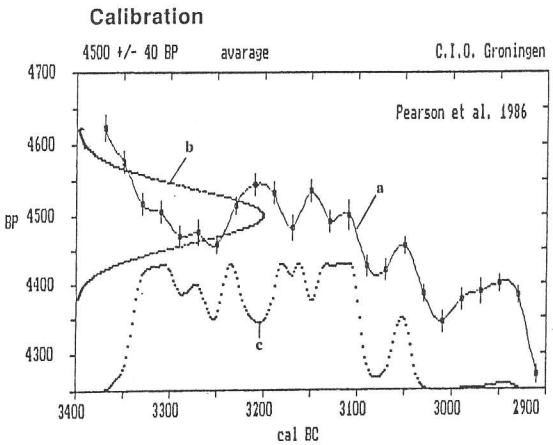

Fig 9. Calibration of the radiocarbon results.

The radiocarbon age is a technical age based upon radioactive decay and a theoretical half life for ^{14}C of 5570 years. Primary production mechanisms, eg cosmic ray variation, earth magnetic field variation etc, are therefore not included. In order to acquire a calendric age, a calibration procedure has to be applied to take care of these effects. The calibration data (Stuiver 1986) are determined by the dendrochronological archives, where the cellulose in each

tree ring uniquely reflects atmospheric ^{14}C content in the past. Figure 9 displays the time interval of interest in the present study. Curve a represents the dendrochronological data (Pearson et al 1986) expressed in cal BP (horizontal axis). It exhibits a wiggling behaviour. Curve b is a Gaussian curve, displaying the analytical radiocarbon age, where the distance from the vertical axis to the curve is proportional to the probability of that age. This is a consequence of the statistical nature of the scientific measurement which means that no point value can be given. Accuracy (statistical spread) is primarily dependent on the total number of ^{14}C atoms detected and thus of the measuring time. A calibrated age interval is then obtained by converting each point at curve b to one or more points on the calendric axis with the help of curve a. Curve b can be said to be mirrored (mathematically convoluted) in the rough surface of curve a with the probability distribution curve c as a result. One interesting direct observation from the calibration is that, because of the relatively constant value of the calibration curve, an improved accuracy in the ^{14}C determination will not affect the calibrated age in the time interval around 4500 BP. The calibrated interval corresponding to the radiocarbon age 4 500 $\pm$ 40 BP is 3100-3350 cal BC.

The five natural mass fractionation values ($\delta^{13}C$) are within the interval -22.0 to -24.7‰ versus PDB, which strongly supports that the organic remains on the sherds are of terrestrial origin. The term terrestrial has in this context the meaning that no marine animals (fish, seal, molluscs) or plants have been used in the prepared food inside the pot. An age shift as a result of reservoir effects or an internal age of the material is therefore neglectable compared with the measuring accuracy and calibration effects.

Conclusion

Radiocarbon analyses have in the present investigation been conducted on food remains of four potsherds representing two different "styles", Säter II and III, from the settlements at Säter II, Säter III and Fagervik in Östergötland. The results demonstrate the possibility of acquiring absolute dates for pottery by applying the ^{14}C technique to food remains. All the four investigated sherds fall within the time interval 4400-4600 BP (3100-3350 cal BC) and are chronologically not differentiable.

The sample sherd representing Säter II pottery, which gives an old-fashioned impression with its reminiscences of funnel-beaker pottery, produced a date in accordance with the established chronology. The three sample sherds representing Säter III pottery, which is usually thought to belong to an advanced stage of the Pitted Ware Culture, became somewhat older than expected.

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Abbreviations

SHM Museum of National Antiquities in Stockholm (Statens Historiska museum Stockholm)