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DISCOVERING A NEW EARLY STARČEVO-CRIȘ SITE IN SOUTHWESTERN TRANSYLVANIA AT FOLT-*SUB VII* (HUNEDOARA COUNTY, ROMANIA) AND A PROCEDURE FOR DATING EARLY NEOLITHIC SHARD SAMPLES

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Abstract: During several field surveys carried out in the southwestern part of Transylvania (Hunedoara County, Romania) in the last decade, by different members of the Archaeological Department of the Museum of Dacian and Roman Civilisation in Deva, we found, near the Mureș river banks, new archaeological sites, one of which belonging to the Early Neolithic. An interesting discovery was made near County Road 107A, where, on the second terrace of the Mureș bank, on the right side of the river valley (the northern side), at a distance of 1 km, in a straight line, from the centre of Folt village, in a place called by the villagers *Sub Vii*, we found an early Starčevo-Criș settlement. On the southern edge of the Folt-*Sub Vii* site we noted, on the surface of ploughland, three Early Neolithic features, rich in fragments of pottery, bones and stone tools. On the feature called by us Cx. 3, with Early Starčevo-Criș pottery, possibly from the beginning of the sixth millennium BC, we experimented to obtain C14 data, comparing the radiometric measurements of two bones and a fragment of pottery. In the Laboratory of Horia Hulubei National Institute for R&D in Physics and Nuclear Engineering (IFIN-HH), Bucharest (Măgurele), the Extracted Degraded Archaeological Material that has undergone diagenetic alteration (EDAM) or so-called Collagen Type I, and different fractions of carbon-bearing materials separated from shards were dated by AMS method, using 1 MV HVEE Cockroft-Walton Tandetron™. Containing endogenous C14, the Oxidant Resistant Elemental Carbon (OREC) fraction complied in terms of calibrated intervals with EDAM results.

Keywords: *Southwestern Transylvania, field surveys, Starčevo-Criș ceramic, interdisciplinary methods of research, absolute chronology*

INTRODUCTION

Archaeological field surveys conducted on both sides of the Mureș Valley in what is now Hunedoara County, in southwestern Transylvania, have contributed to the identification of new sites, along with the rediscovery of other archaeological sites less known in historiography. Such is the case of the site Folt-*Sub Vii*, where repeated field surveys have identified an early Neolithic settlement, which can be attributed to the early stages of the Starčevo-Criș cultural complex.

Due to the site being exploited for agriculture, the marks of three archaeological features, possibly of a dwelling type, were apparent on the surface of the soil, as observed on the basis of concentrations of archaeological materials, represented mainly by ceramic fragments, lithic tools and osteological materials. Most representative in this sense was the case of the feature Cx. 3, in fact a concentration of ceramic, lithic and osteological pieces.

To establish the chronology of Cx. 3, beyond the typological and stylistic descriptions of the pottery, which provide few clues to the relative age of the feature, radiocarbon dating was performed on the osteological material, and then an attempt was made to date a ceramic fragment – sample 22 (617.6 RoAMS code). The ceramic sample, dated at the Horia Hulubei National Institute for R&D in Physics and Nuclear Engineering (IFIN-HH), Bucharest (Măgurele), Romania, represented a challenge for the co-authors of this study, the working method described in the lines below being still little used in Romanian archaeology.

THE LOCATION OF THE SITE FOLT-*SUB VII*

The *Sub Vii* site is located to the northeast of Folt village (Fig. 1/A–B), on the second terrace on the righthand side of the Mureș Valley; on the southern side, the archaeological point is bordered by the DJ107A (Uroi–Geoagiu) county road[1].

The terrace that contains the Folt site, the *Sub Vii* point, is uneven; more precisely, it is more elevated in the eastern, northern and northeastern areas (Fig. 2–3). Other landmarks that outline the outskirts of the site are the connecting road towards Boiu village, located to the west of the site (approx. 740 m) and the access road to Cigmău village to the east (approx. 150–300 m). The Boiu Valley (in the north–south direction) is closer to the archaeological point from the west (approx. 250 m).

Regarding the natural environment within which the site is located, at the southern margin of the final sub-mountainous and hilly formations of the Metaliferi Mountains, the extensions of which reach the Mureș Valley, we must emphasise the presence and immediate proximity of sedimentary,

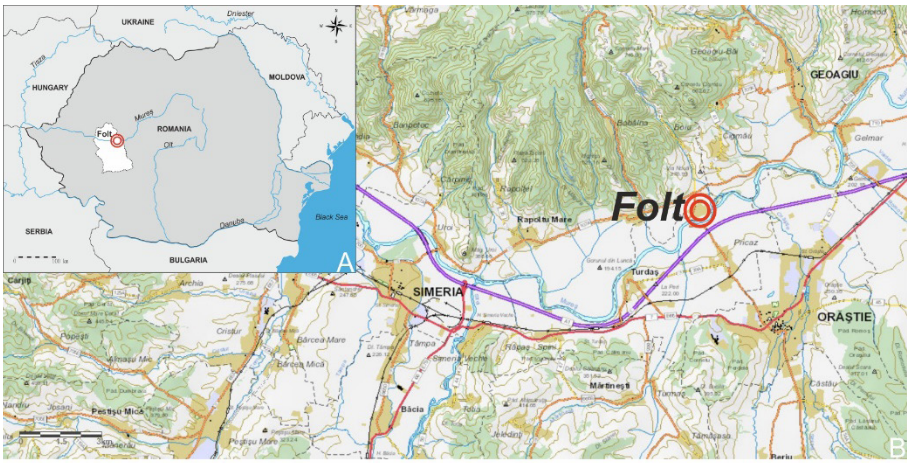

Fig. 1. A. Map of Romania showing the localisation of Folt village (map by I.-C. Codrea and I. A. Bărbat); B. The location of Folt village on the map of the southwestern area of Transylvania (adaptation from http://geoportal.gov.ro/geoportal/home/webmap/viewer.html) (Accessed: 10.08.2020).

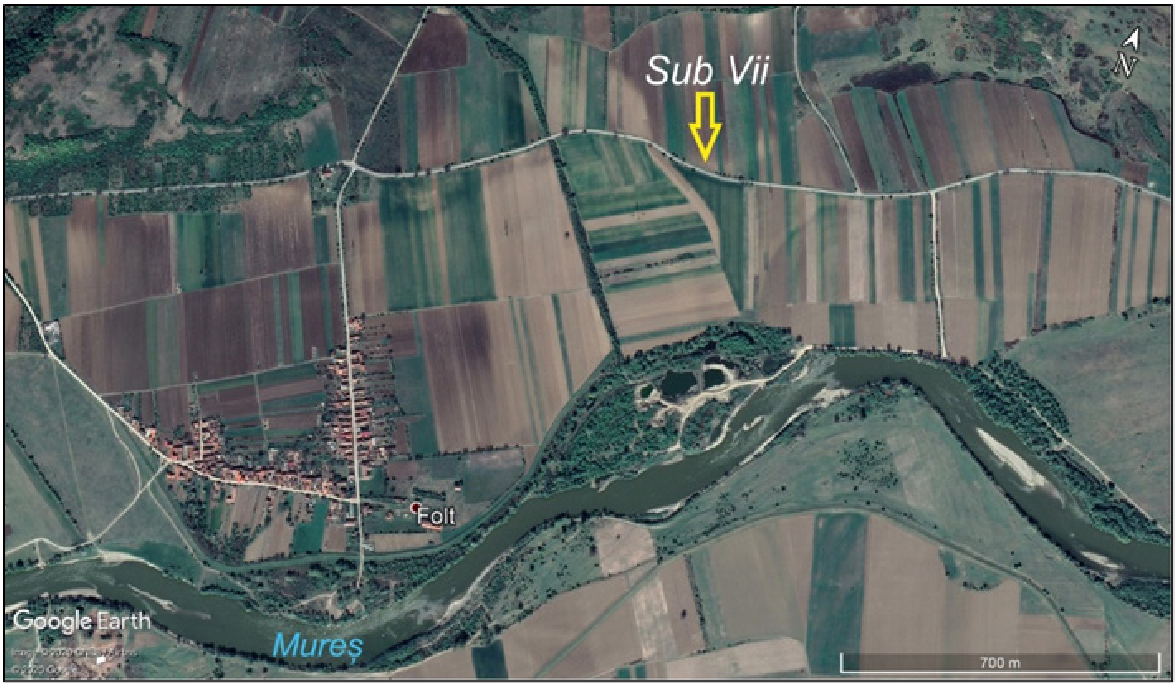

Fig. 2. The position of the *Sub Vii* site (marked by the yellow arrow) on the orthophotomap of the area in which Folt village is located (adaptation from Google Earth) (Accessed: 10.08.2020).

calcareous and metamorphic rocks. Moreover, we believe that the presence of several springs and fertile lands in the

[1] ANDRIȚOIU 1979, 23; ANDRIȚOIU 1986–1987, 51; ANDRIȚOIU 1992, 122; BOROFFKA 1994, 43; CĂSTĂIAN 1995, 39; URSUȚIU 2002, 89; LUCA 2008, 75.

Fig. 3. A. Aerophotography containing the Folt-*Sub Vii* archaeological point, as seen from the west (the yellow dotted line marking the approximate outline of the site); B. The view of the terrace, as seen from the southeastern Folt-*Sub Vii* point, bordered to the south by the DJ107A County Road, and the Cx. 3 feature location (Photo: I. A. Bărbat).

Mureș Valley, as well as that of wood resources, provided certain fundamental conditions for the occupation of the terrace by human communities.

THE RESEARCH HISTORY

After the work undertaken to modernise the Folt–Geoagiu road at the end of the 1970s, and as a result of a field survey carried out during the same period by the employees of the Hunedoara-Deva County Museum[2], a new archaeological site was identified at the location known as Folt-*Sub Vii*[3]. From a chronological viewpoint, all of the materials uncovered at that time, published four decades ago, can be attributed to the Middle and Late Bronze Age as well as to the Early Iron Age (the Basarabi culture) or to the Migration Period (5th–6th centuries AD)[4]. We must also add that, in the aforementioned context, the schoolteacher I. Ilieș recovered ceramic materials that he then stored in the Folt village school[5].

During the 1990s, less than two decades after the

[2] Presently the Museum of Dacian and Roman Civilisation Deva.

[3] ANDRIȚOIU 1979, 23; ANDRIȚOIU 1992, 122; CĂSTĂIAN 1995, 39; URSUȚIU 2002, 89.

[4] ANDRIȚOIU 1979, 23–24; ANDRIȚOIU 1986–1987, 51; ANDRIȚOIU 1992, 122; BOROFFKA 1994, 43; CĂSTĂIAN 1995, 41; URSUȚIU 2002, 89; LUCA 2008, 75.

[5] CĂSTĂIAN 1995, 41.

discoveries made at the end of the 1970s, a new field survey aimed to illuminate new information in addition to the data already published regarding the Folt-*Sub Vii* site. The results of the periegeses of that time merely confirmed once more the presence of the same categories of artefacts specific to the Bronze Age[6].

The field surveys on the Mureș Valley in the Hunedoara County that began in 2008 were resumed and continue to the present day, contributing to the "rediscovery" of certain sites that had once been considered to be "characteristic" only of the Bronze Age or other historical periods, assumptions which also included Folt-*Sub Vii*[7] (Fig. 3/A–B). The periegeses carried out at this site confirmed the results obtained in the past, but they also offered certain surprises, as did the discovery of several Early Neolithic dwellings characteristic of the Starčevo-Criș cultural complex. Although such archaeological materials were identified on the entire southern border of the terrace at different points, the cited bibliography contains no mention of the identification of certain artefacts characteristic of the Neolithic Period at the *Sub Vii* point[8].

An inspection of some of the archaeological materials discovered at the end of the 1970s at the Folt-*Sub Vii* point and stored at the Museum of Dacian and Roman Civilisation, Deva after the field surveys also led to the identification of ceramic shards characteristic of the Starčevo-Criș cultural complex[9]. This led us to assume that the archaeological materials had been published selectively more than four decades ago, and that the Early Neolithic ceramic items had most likely been overlooked or "set aside"[10].

During the archaeological supervision for the excavation of the county road 107A, in the spring of 2022, ceramic artefacts and traces of prehistoric archaeological features were detected on the left side of the road, between the localities of Folt and Cigmău, at the contact between the first and second terraces of the Mureș valley, called *Sub Vii*, not far from the right bank of the aforementioned river. More precisely, the aforementioned works allowed the observation of the footprint of archaeological contexts on an area of 173.69 sqm, at km 40+920 of DJ107A.

Subsequent rescue excavation, in November and December of 2022, between the villages of Folt and Cigmău, north of road 107A, led to the identification of archaeological features from the Bronze Age, the first Iron Age, and also from the post-Roman period.

THE ARCHAEOLOGICAL FIELD SURVEY RESULTS[11]

The field surveys of 2015 conducted throughout the surface of the Folt-*Sub Vii* site showed that all the archaeological materials that undoubtedly belonged to the Early Neolithic period chronologically had been uncovered particularly on the southern border of the terrace. Presently, three areas containing ceramic shards and osteological and lithic items have been identified, all of which most likely represent the markings of several archaeological contexts disturbed by the agricultural works on the terrace. The Early Neolithic features uncovered through periegeses appeared in the southwestern (Cx. 1[12] and Cx. 2[13]) and southeastern (Cx. 3[14]) sectors of the terrace. Other observations were made in the area of the erosion marks and the ditches created by the agricultural works. It was established that at a depth of approximately –0.20 to –0.30 m from the current surface of the arable soil, there are archaeological features that are, in the case under scrutiny, characteristic of the Early Neolithic. Regarding the role played by features Cx. 1, Cx. 2 and Cx. 3, we can only assume, in the present state of the research, their possible belonging to the dwelling category.

As mentioned previously, as a result of the field surveys, different categories of artefacts from the Early Neolithic were gathered from the surface of the soil. Among these materials, with the exception of a small selection of osteological and lithic items, the ceramic materials, exclusively fragmentary, can be considered to be the most representative.

Out of the representative lithic items, some of which were identified around feature Cx. 3, we must mention several carved and polished tools. In the case of the items obtained by knapping stone, we must mention the mesial portion of a high-quality chert blade (Fig. 4/1a–1b) with a trapezoidal transverse section (acquired, maybe, through pressure technique), bearing a dark smoky translucid shade[15]. The blade segment bears markings of wear on both sides as well as signs of a macro-polish; at the time of its use, the item had been broken in front of an *encoche*, which implies that the artefact had been gloved.

Among the items realised through rock polishing, we identified the presence of a fragmentary rock chisel (Fig. 4/2a–2b), namely a shoe-last chisel bearing approximately plane-convex longitudinal and transverse sections; possibly made from mudstone (?)[16].

Osteological material was gathered from the surface of Cx. 1, Cx. 2 and Cx. 3. Archaeozoological assessments

[6] CĂSTĂIAN 1995, 41.

[7] This research was conducted under the field assessment authorisations issued by the Ministry of Culture, Cultural Heritage Division, scientific coordinator Dr I. A. Bărbat, at that time employed as an archaeologist at the Museum of Dacian and Roman Civilisation Deva.

[8] ANDRIȚOIU 1979, 23–24; ANDRIȚOIU 1986–1987, 51; ANDRIȚOIU 1992, 122; BOROFFKA 1994, 43; CĂSTĂIAN 1995, 41; URSUȚIU 2002, 89; LUCA 2008, 75.

[9] We wish to express our gratitude to our colleague Costin-Daniel Țuțuianu (archaeologist at the Museum of Dacian and Roman Civilisation, Deva), for pointing out the cache of archaeological materials discovered in Folt-*Sub Vii* in 1978.

[10] The two ceramic shards are part of a cache of 54 archaeological items, the inventory numbers of which are between 29.898 and 29.900. According to the General Inventory Registry, vol. 3, p. 131 (mss), all of the objects were discovered during the periegesis of 1978; it also mentions that the items belong to the Wietenberg culture, Hallstatt period and 5th–6th centuries AD. Interestingly, on the box containing the archaeological items from Folt-*Sub Vii*, the year of discovery is marked as 1977.

[11] The present approach is a short description of the main results obtained during the field surveys; the artefacts are presented briefly and selectively.

[12] GPS coordinates (Garmin Montana 650t): Pt. 1 (45° 53' 6.6'' N, 23° 9' 47'' E); Pt. 2 (45° 53' 6.5'' N, 23° 9' 46.8'' E); Pt. 3 (45° 53' 6.7'' N, 23° 9' 46.7'' E); Pt. 4 (45° 53' 6.8'' N, 23° 9' 46.8'' E); Elevation 212–213 m. The surface area covered by the archaeological materials is approximately 25 sqm.

[13] GPS coordinates (Garmin Montana 650t): Pt. 1 (45° 53' 7'' N, 23° 9' 47.1'' E); Pt. 2 (45° 53' 7.1'' N, 23° 9' 47.3'' E); Pt. 3 (45° 53' 7'' N, 23° 9' 47.3'' E); Pt. 4 (45° 53' 6.9'' N, 23° 9' 47.2'' E); Elevation 213–214 m. The surface area covered by the archaeological materials is approximately 25 sqm.

[14] GPS coordinates (Garmin Montana 650t): Pt. 1 (45° 53' 7.5'' N, 23° 9' 50.1'' E); Pt. 2 (45° 53' 7.4'' N, 23° 9' 49.8'' E); Pt. 3 (45° 53' 7.1'' N, 23° 9' 49.9'' E); Pt. 4 (45° 53' 7.2'' N, 23° 9' 50.3'' E); Elevation 215–216 m. The surface area covered by the archaeological materials is approximately 65 sqm.

[15] Max. length = 2.06 cm; Max. width = 1.43 cm.

[16] Max. length = 4.58 cm; Max. width (edge) = 3.08 cm; Max. width (head) = 2.49 cm.

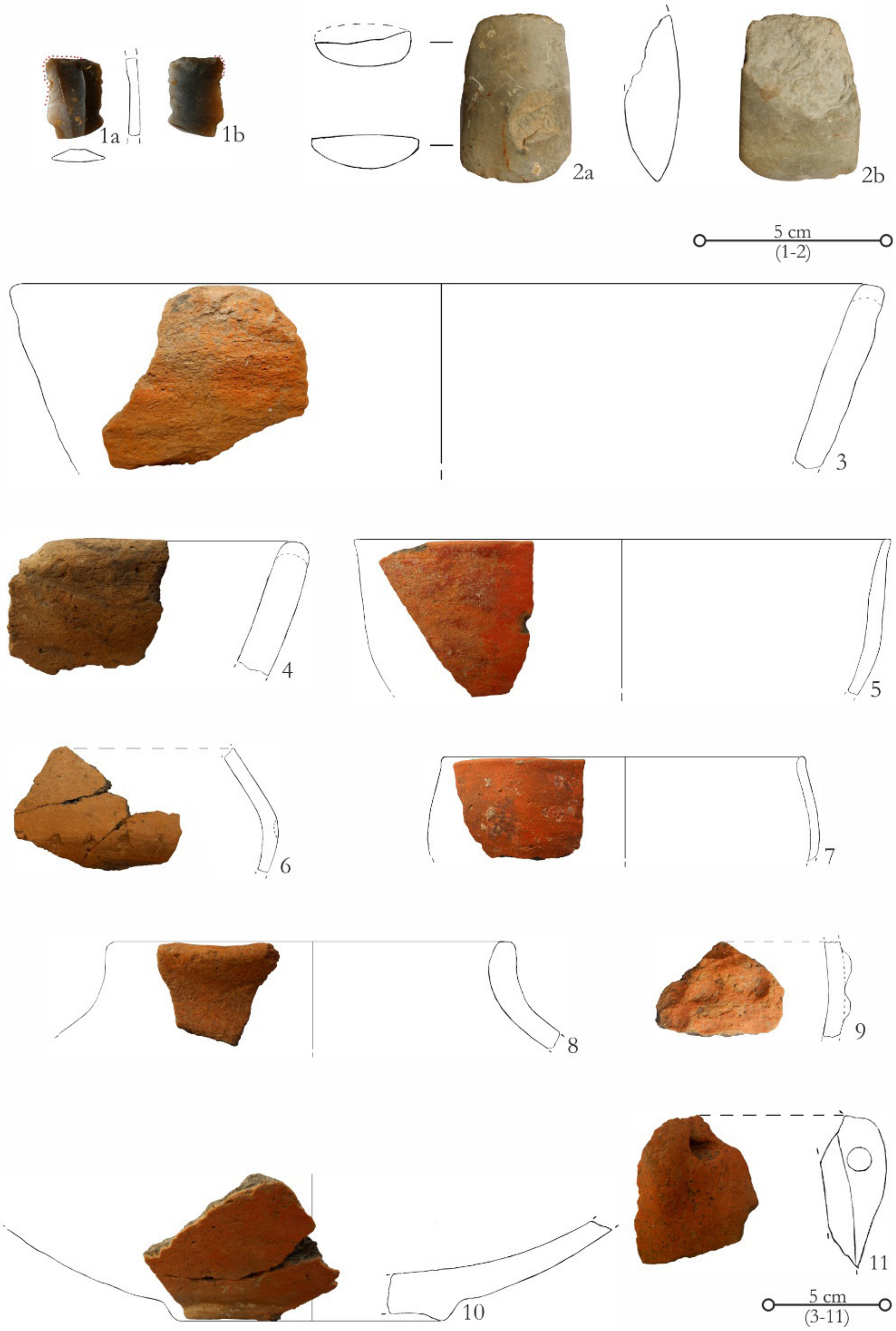

Fig. 4. Archaeological materials from feature Cx. 3; 1a–1b. The mesial portion of the macro-polished chert blade (see the red dots); 2a–2b. Mudstone (?) fragmentary chisel; 3–11. Ceramic shards (Photo: I. A. Bărbat; drawing S. Ivan).

indicated the presence of a fragment of a long bone (Fig. 5/1) and of a phalanx (Fig. 5/2) from a *Bos primigenius* in the aforementioned feature[17]. We must also add that the presence of a *Bos primigenius* was recently noted in an Early Neolithic site nearby (approx. 8.34 km in a straight line), namely in Rapoltu Mare-*Șeghi*[18].

The Early Neolithic ceramics gathered from the surface of the site are typical of the Starčevo-Criș cultural complex. Most of the ceramic shards had been manufactured from clay mixed with chopped chaff, sand and, more rarely, pebbles. The vessels generally display reduction burning, and the colours vary between red, scarlet, yellow, brown and black. The red-scarlet ceramics that sometimes bear polishing markings were more often encountered in the area of feature Cx. 3 (Fig. 4/3–11). Other ceramic items with "dirtier" slips

[17] We are grateful to late archaeozoologist Valentin Dumitrașcu (Institute of Archaeology "Vasile Pârvan", Bucharest) for the fauna assessments.
[18] STANC *et alii* 2020, 212.

that are sometimes slightly exfoliated and often bear yellow/brown-scarlet shades were discovered particularly in the sector of the Cx. 1 and Cx. 2 features.

The Early Neolithic ceramic items identified in all three possible archaeological features as well as outside of them are extremely scarce from a typological viewpoint, maybe with the exception of Cx. 3. Here the ceramics are typically represented by globular/spherical vessels (Fig. 4/7–8) and bitronconical vessels (Fig. 4/6) or resemble basins of tronconic bowls (Fig. 4/3–5). Usually, such ceramic containers bear no ornamentations – the decorations are very rare and appear in the form of: grooves on the lips of the tronconic vessels (Fig. 4/3–4), pin pricks made with the tip of a bone, wooden or stone object on the bitronconic vessels (Fig. 4/6), while the globular/spherical items bear "fingertip" pricks (Fig. 4/9).

THE RELATIVE CHRONOLOGY

In order to establish relative chronology, we are compelled to call upon the central "guiding fossil" – ceramics. Based on the clues provided by ceramic types, forms and ornaments, we can assess the existence of at least two distinctive points in time at which the terrace had been occupied during the Early Neolithic.

The first chronological horizon points to the use of the higher area of the terrain in the form of a promontory located in its southeastern part, where the majority of ceramic materials bear a monochromatic red-scarlet shade, with well-preserved and sometimes polished slips. The burning and high quality of the ceramics, the absence of incised ornaments of the sprayed or displayed barbotine, the rarity of grooved motifs, the presence of globular shapes and the open-type forms compel us to include the ceramic items from Cx. 3 in the Starčevo-Criș IC–IIA stages as they have been defined by G. Lazarovici[19].

The second period of occupation, represented by features Cx. 1 and Cx. 2 from the southwestern part of the site, can be synchronised with the end of the Starčevo-Criș IIA phase and the beginning of phase IIB[20]. In this chronological segment, across a series of Early Neolithic sites, some authors consider that a cultural "retardation" or "isolation" phenomenon occurred[21]. Such a chronological frame is supported, firstly, by ceramic manufacturing technology – usually containing more sand in the clay, it is sometimes more poorly burned and the slip and the engobe display poorer adherence, leading to their exfoliation. The chromatics of the ceramics have also visibly changed – the exteriors of the vessels sometimes bear shades of yellow, scarlet and brown. Characteristic of features Cx. 1 and Cx. 2, we must note the presence of open ceramics, including basins and tronconic bowls, as well as closed types, such as globular and spherical vessels. Ornaments are rare; the more-often encountered types are either grooved, as indicated by the alveoli on the lips and walls of the vessels, or applied (alveolar bands). No decorative motifs created through incision, barbotine or painting have been identified.

The aforementioned open-shaped pots, although not presented in entirety, is relevant to the Starčevo-Criș IC phase, IIA phase and perhaps even the beginning of the subsequent phase, IIB, if one takes the criteria established by G. Lazarovici for the periodisation of Early Neolithic ceramics into account[22]. This chronological horizon includes a series of settlements from Transylvania, Banat, Oltenia and Muntenia, and we can refer to several sites located in the neighbouring areas of the Folt-*Sub Vii* archaeological point, including the Mureș Corridor and its tributaries: Șoimuș-*Teleghi*[23], Hațeg-*Câmpul Mare*[24], Rapoltu Mare-*Șeghi*[25], *La Vie*[26], Folt-*Valea Boiului*[27], Turdaș-*La Luncă*[28], Cugir-*La Arini*[29], *Podereauă*[30], Sălіștea-*Str. Drejman nr. 431*[31], Șeușa-*La Cărarea Morii*[32] and others.

The fact must be emphasised that the relative chronology achieved through "traditional" analysis, based on the typological correspondences of the ceramics as well as the assay of the entire lot of artefacts compel us to exercise caution when discussing the origins of the Old Neolithic discoveries from Folt-*Sub Vii* within a certain chronological frame. By all appearances, it is very likely that we are facing an archaeological site that contains a dwelling contemporary with the Early Starčevo-Criș horizons south and north of the Lower Danube, which can be dated to the first two centuries of the 6th millennium BC.

THE ABSOLUTE CHRONOLOGY. AN INTERDISCIPLINARY APPROACH

To obtain absolute chronological data, several analyses were performed within the RoAMS Laboratory at the Horia Hulubei National Institute for R&D in Physics and Nuclear Engineering (IFIN-HH), Bucharest (Măgurele).

The rarity of well-preserved osteological material suitable for radiocarbon dating, with the exception of a long bone fragment (Fig. 5/1) and a phalanx (Fig. 5/2) of *Bos primigenius*, influenced us to choose an alternative sequence of analyses. Thus, the absolute age of a ceramic fragment with chaff sampled from the same concentration of archaeological materials in Cx. 3 (Fig. 5/3a–3b) was determined.

1. Radiocarbon data obtained for extracted "collagen" (Extracted Damaged Archaeological Material, EDAM, formally attributed as being Type I collagen) from bones:

In the context presented above, the dating strategy using

[19] LAZAROVICI 1977, 34–37; LAZAROVICI 1979, 41–44, 62–64; LAZAROVICI 1984, 58–62; LAZAROVICI 1998, 13–31; MAXIM 1999, 34–43; LAZAROVICI 2006, 138, 140–141, 144; ROMAN 2008, 82–84, 133–142; TUDORIE 2013, 27, 56–60.
[20] LAZAROVICI 1977, 36–37; LAZAROVICI 1979, 43–46, 63–64; LAZAROVICI 1984, 60–64; LAZAROVICI 1998, 13–31; MAXIM 1999, 40–45; ROMAN 2008, 84, 142; TUDORIE 2013, 27–28, 59–60.
[21] LAZAROVICI *et alii* 1989–1993, 321–322; LAZAROVICI/MAXIM 1995, 200; LAZAROVICI 2001, 38.
[22] See the literature referenced in footnotes 20 and 21.
[23] BĂRBAT 2018, 60; MALAXA *et alii* 2022, 4–10.
[24] ROMAN/DIACONESCU 2002, 9.
[25] STANC *et alii* 2020, 211–212.
[26] BĂRBAT 2013, 19.
[27] Unpublished materials.
[28] LUCA 2012, 47–51; LUCA/TUDORIE/CIUTĂ 2012, 7–12; LUCA/TUDORIE 2013, 7–11; LUCA 2019, 28–36.
[29] POPA 2000, 20–21; POPA 2011, 47–48.
[30] POPA 2000, 21; POPA 2011, 48.
[31] LUCA/TUDORIE 2012, 22–27.
[32] CIUTĂ 2000, 63–70, 73–76; CIUTĂ 2005, 119, 153; CIUTĂ 2009, 63–76.

Fig. 5. Archaeological materials in the context of Cx. 3; 1. Long bone fragment of the species *Bos primigenius*; 2. Phalanx of *Bos primigenius*; 3a–3b. Ceramic fragment (bottom) (Photo: I. A. Bărbat).

absolute chronology began with the osteological material, namely a long bone fragment (Fig. 5/1), RoAMS code 615.6, and a phalanx (Fig. 5/2), RoAMS code 616.6.

To select the parts that would be sacrificed for radiocarbon dating, representative areas of the archaeological pieces were avoided. In order to guarantee obtaining a reliable fraction of collagen for radiocarbon dating (hereinafter simply referred to as collagen), information from an already published document was used [33].

Both samples provided relatively well-preserved collagen, which was extracted using the Acid-Acid-Ultrafiltration method, graphitised on the Elementar – AGE 3 system[34] and measured with the 1 MV Walton-Cockroft Tandetron accelerator[35] in IFIN-HH.

Absolute chronology established these CRA (Conventional Radiocarbon Age) values[36]:

CRA ± SD = 6815 ± 42 BP (615.6)

CRA ± SD = 6915 ± 46 BP (616.6).

Calibration using the OxCal Online Program provided, on the date the calibrations were made (January 2018), the following intervals:

5771–5632 cal BC (for σ = 2; probability of 95.4%), for 615.6

5901–5714 cal BC (for σ = 2; probability of 95.4%), for 616.6.

These radiocarbon data were taken as a benchmark for the next dating sequence, namely the dating of the organic remains from the ceramic fragment associated with bones in the context of the discoveries (Fig. 5/3a–3b).

2. Radiocarbon data obtained for extracted "organic deposits" (residues that come from organic materials) in ceramic fragment with chaff (Fig. 6):

As mentioned previously, from the archaeological material discovered in Cx. 3, a ceramic fragment was chosen – the lower part of a possible globular vessel (black-grey, coarse, reduced burner), code RoAMS 617.6, displaying a paste rich in organic matter that allows for the performance of measurements *via* AMS (accelerated mass spectrometry).

Archaeological literature has long held the idea that the use of organic materials in the preparation of paste of Early Neolithic vessels, such as crushed chaff (straw), added plasticity to the clay, thus preventing the breakage of ceramic forms during drying or burning[37].

Literature that specialises in publishing the results obtained from archaeometric research provides, in turn, an entire series of information on dating ceramics by absolute chronology – in this case, by radiocarbon dating of organic material separated not from the outside or inside of the fragment but from the ceramic material itself, which is more rarely used.

Based on this information, the fragment was chemically pre-treated in several steps, providing datable fractions. Obtaining the radiocarbon ages for each and associating the intervals with those obtained for the osteological material ultimately led to the selection of the most reliable date and the refinement of the absolute chronology.

Considering the pros and cons of dating the pottery, as well as the other situations presented elsewhere in the literature, using a strategy customised to the type of pottery analysed, it has become possible to separate "microcharcoal" fraction (fine coal, separated from the ceramic mass, after burning the straw added to the paste in a reduced amount of oxygen).

Radiocarbon data may provide a much older age than the estimated period. In this case, the additional carbon-bearing source is most likely important enough to affect the final result and comes from clay itself or from coal (fossil, semi-fossil) added to the paste. No other intentional additions of carbon or graphite by surface application or as pigment were observed.

Microcharcoal or soot from the burning of very old wood (or semi-fossilised trees) may appear in the vessel from its manufacturing and possibly its use in cooking. This was not the case this time either. The soot appears on the surface

33 SOFICARU *et alii* 2018, 10–47.
34 WACKER/CHRIST/SYNAL 2010, 931–934.
35 SAVA *et alii* 2019, 649–648.
36 WACKER/NĚMEC/BOURQUIN 2010, 976–979.
37 CIUTĂ/ANGHEL/SABĂU 2000, 107; CIUTĂ 2005, 74, 77–81; TUDORIE 2013, 45.

anyway and can be removed mechanically before or during ceramic fragment pre-treatments.

On the other hand, the calibrated age obtained may be within the estimated period if the aforementioned sources of contamination do not exist or are in extremely small quantities so as not to affect the result.

It is also possible to obtain a date notably younger than expected due to overlapping and mixing of other organic materials than microcharcoal which are present underneath surface or inside the ceramic fragment, from more recent historical horizons, especially belonging to the Bronze Age, which were already signalled during the excavations.

Ceramic fragments, after mechanical cleaning, ultrasonic washing in ultrapure water Milli-Q® 8 and drying to constant mass, were broken into small pieces. They were analysed both visually and under microscope.

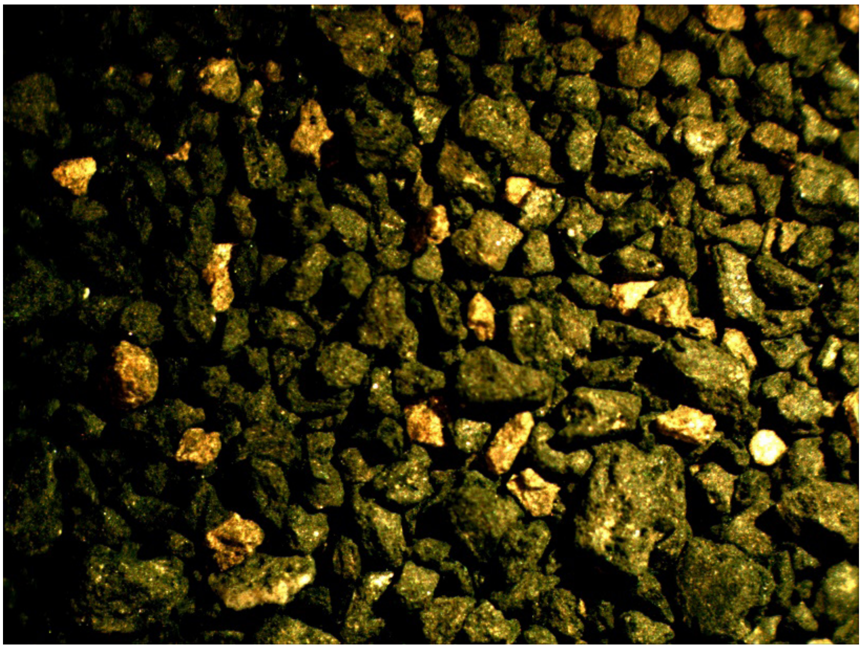

Fig. 6. Details captured under microscope of the inner texture of the ceramic fragment (Photo: C. A. Simion)

Both free channels left in the clay mass after burning (pores loaded with exogenous materials during manufacture or use) and channels in which the original chaff was preserved in the form of coal within tubular formations of burnt clay (forming microcharcoal filaments in the paste by burning the organic material initially added during combustion in the kiln) were discovered. In many other alveoli in which the chaff was not preserved, the space it occupied was filled over time with other materials of inorganic and organic nature from the soil in the post-depositional period. These materials may or may not contain carbon, which, in either case, is no longer the same age as the original chaff.

It is important to date the original chaff/microcharcoal containing endogenous carbon as well as the other categories of organic matter containing exogenous carbon (especially humic matter, fulvic acids and other organic degradation products that are in constant motion in the soil, both horizontally and vertically during post-depositional period). The latter, from the environment, especially exogenous carbon-bearing compounds, are much less trapped in the matrix of burnt clay than those from the original carbon and are practically represented by other chemical forms that are much easier to separate or destroy during pre-treatments. The most mechanically, physically and chemically resistant carbon is known as OREC (Oxidant Resistant Elemental Carbon) and represents a unitary or chemically inert chemical species of microcharcoal. It can come from the paste and be added to the formation of the pot, or it can come from the chaff in the process of burning in the kiln, having or not different ages.

Trapped in the paste, the organic matter is protected from the exchange that can take place with carbon dioxide as a result of the burning of wood or other fuel that maintained the fire. Therefore, it is unlikely for the "old wood effect" (which sometimes manifests itself when cremating corpses) to take place during the burning of the chaff at kiln temperatures.

These categories, which represent endogenous and exogenous carbon, are impossible to separate efficiently by mechanical and physical methods, the insistence of the processes leading to their mixing or to complications in the subsequent stages of thermal and chemical pre-treatments. Therefore, a strategy must be imagined by which the large groups of organic matter remaining in the pores of the burnt clay can be separated, purified and dated individually, rendering the calibrated ages obtained valuable indicators of the scenarios of vessel formation, (re) use, and the post-depositional period.

Carbonate deposits and other inorganic chemical forms are easily removed in solutions of acidic mediums. Therefore, it will be involved in all variants. Classes of natural organic compounds soluble at $pH < 7$, such as fulvic acids, will also pass into acidic solutions. Thus, in other reaction media remain either the fractions soluble in the basic medium (another part of fulvic acids, humic materials, lipids, etc.) or those that remain unassailable in both acidic and basic media, including natural carbon and humin. This final group of natural carbon and/or humin remains the most inert segment, even in the concentrated solutions in which both the solid matrix of the paste and the components containing inorganic or organic carbon are solubilised or can be destroyed in turn. Other resistant organic chemical species, such as intermediate humin, can be gradually destroyed in oxidising acidic solutions and at room temperature.

Absolute chronology; from the choice of methods and representative fractions to pre-treatment, graphitisation, AMS measurement, Conventional Radiocarbon Age values and calibrated data, and their interpretation in relation to relative chronology

3. The exercise followed four important steps:

Phase I, represented by the final analyte 617.6, followed the chemical digestion in admixtures of concentrated inorganic acids of all forms mentioned above. In the final stage, in the presence of boric acid, the mixtures of organic matter deposited in time, immature, were released. None of the acids used, including boric acid, contain carbon in their formulas. At the end, a black-brown deposit is obtained that represents everything deposited in the pores of the ceramic fragment from the moment of its formation until the moment when all its pores were refilled with soil materials, thousands of years before unearthing. The old carbon deposits remain in the clay. The young deposits from the surface were mostly removed through washing in ultrapure water and ultrasound treatment, and the remaining ones were destroyed in concentrated nitric acid and boric acid.

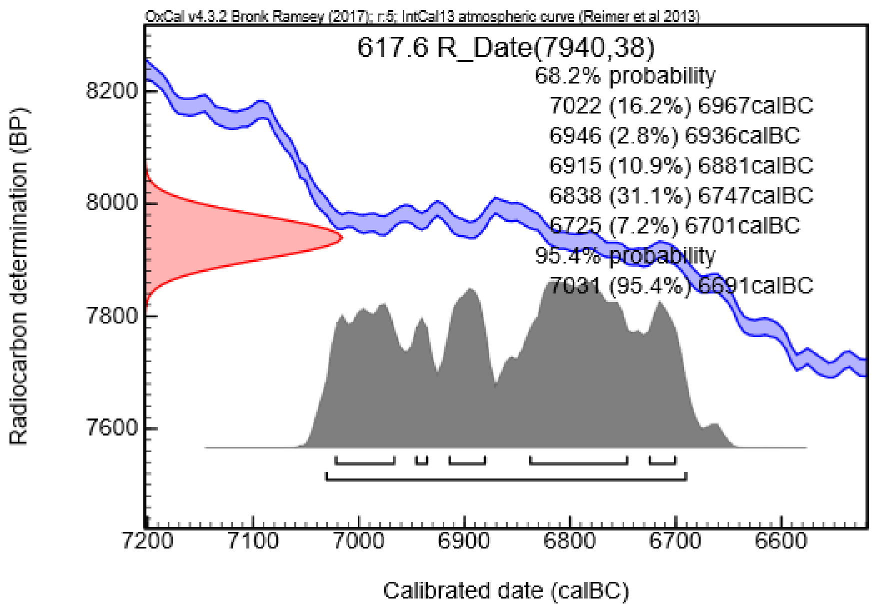

Fig. 7. Histogram for Phase I; 617.6 fraction

This final fraction was brought to a neutral pH using ultrapure water and then dried using freeze-drying. Graphitisation was performed in the Elementar – AGE 3™ system, and natural carbon intimately deposited on the iron catalyst was introduced into the target and measured with the 1 MV accelerator. The calibrated result is presented in Fig. 7.

Compared to estimates, the result is unexpectedly old. The difference from the estimated age could be due to the older immature carbon in the clays, the burning of fossil or semi-fossil fuel in the kiln or to the "old wood effect" (although this third scenario is very unlikely). However, it may also come from soil materials other than microcharcoal variety that are much older than the object that entered the layer in the post-depositional period.

In order to scientifically explain the result, we moved into Phase II.

Phase II led to the final analyte 617.6HA1_HA2, which comes essentially (after the removal of all carbonates in a strongly acidic medium) from the separate fraction in a basic medium, such as humic acid. A standard Acid-Base-Acid treatment adapted to a new portion of ceramic sample was applied. First, carbonates, fulvic acids and acid-soluble substances were removed, then emphasis was placed on recovering the entire humic acid fractions, which are soluble in basic medium. In the solid phase, the mineral matrix and other insoluble substances remain, namely humin and different species of carbon, some of them representing OREC. Finally, the combined humic acid fractions are obtained and purified by re-precipitation in a strongly acidic medium, washed at pH ≤ 5 and freeze-dried.

Forwards, repeating Phase I steps for AMS measurements, the following calibrated result was obtained:

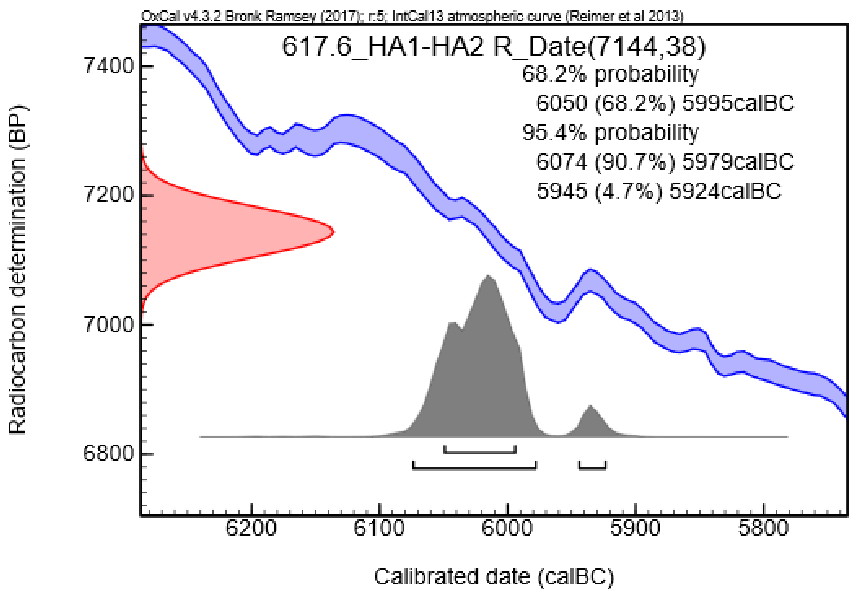

Fig. 8. Histogram for Phase II; 617.6HA1_HA2 fraction

This result is much younger than the previous one, and it is closer to the estimates (Fig. 8). They are fractions in transformation, younger than the carbon trapped in the pottery at the moment of the post-depositional period and the subsequent old forms of degradation of the soil matter deposited, reaching the humin stage. To see the extent to which the two results are correlated, another set of natural compounds was isolated, corresponding to Phase III.

Phase III has as a final analyte 617.6A. The severe acid treatment applied to a new portion of ceramic sample destroyed all previously involved materials, the intermediate basic solution separated the humic fractions and the acid treatment neutralised the carbon dioxide introduced by the sodium hydroxide solution. The final step (an adaptation of the classic Acid-Base-Acid treatment) will provide the oldest organic fraction, so-called pure humin, deposited together with OREC carbon on the inert mineral matrix. In theory, it is older and more chemically inert than

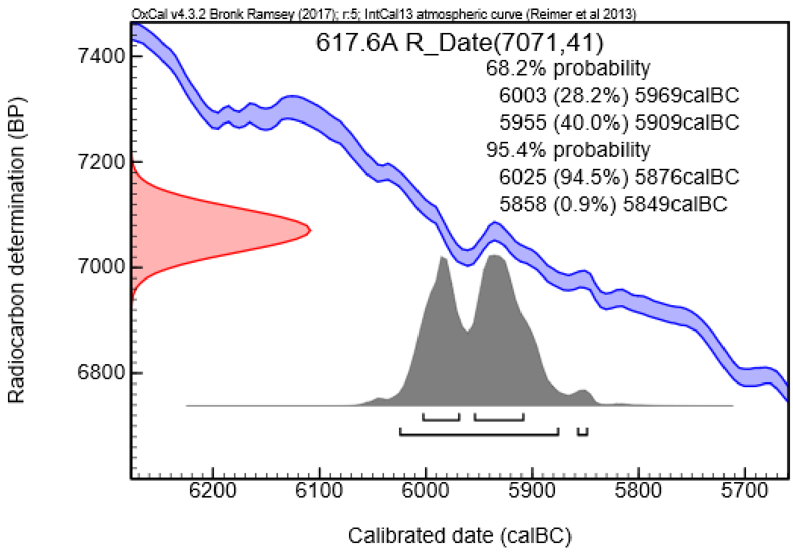

Fig. 9. Histogram for Phase III; 617.6A fraction

previous humic fractions. The obtained histogram is presented in Fig. 9.

Practically, the humic fractions are slightly older than the humin mixed with OREC. As humin is usually the oldest fraction of humic materials, at most equal in age to the rest, this means that the OREC fraction is likely the youngest component of the mixture. Due to the immobilisation of these mature fractions in the channels/pores of the ceramic fragment, other mechanisms leading to inversion between the ages of humic acids and humins are unlikely in this particular case. To verify this, we isolated the fraction 617.6B from the fourth portion of the sample, during Phase IV.

Phase IV is represented by the fraction 617.6B and contains the remaining OREC carbon, the most resistant to all previous attacks, obtained through attack by strongly acidic media, then by exposure to strongly oxidising conditions for 72 hrs. All organic forms were destroyed in the process, except for the remaining elemental carbon. The dating of this fraction provided the calibrated age (Fig. 10):

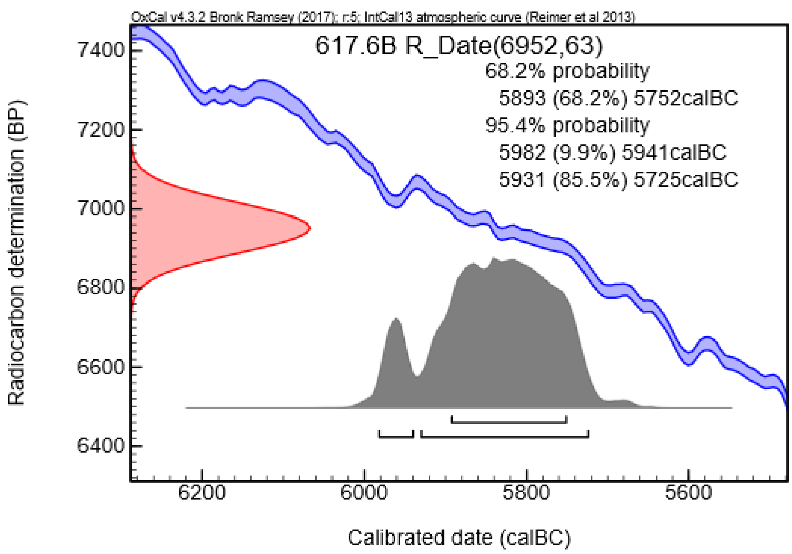

Fig. 10. Histogram for Phase IV; 617.6B fraction

We note that this new data best represents the expected time horizon and the presence of carbon in the soil, the added charcoal or "old wood effect" being unlikely or not influencing the result if they are present. The OREC fraction is relatively younger than the humin, the two components of previous Phase III being separated this time.

Through subsequent adaptation of some pre-treatment methods specific to radiocarbon dating (as those for Phases III and II), we obtained the safest fraction from the chaff in Phase IV. The age is younger compared to humic fractions by approximately 100–200 years and explains this inversion of ages. Practically, the environment in which the ceramic fragment was buried is rich in humus visibly older than when it entered the post-depositional period, becoming a source of persistent contamination, as has been noted since the 1960s in the case of bone dating[38]. The soil did not, either in the manufacturing process nor in the post-depositional stage, provide microcharcoal integrable into the fraction from the chaff. Rather, the Bronze Age–specific housing horizon made its own contribution, hence the differences between the four separated and dated fractions.

INTEGRATING THE RESULTS; CORRELATION WITH RELATIVE CHRONOLOGY

We resume the six calibrated data for σ = 2, with a probability of 95.4% that the age falls within the ranges below:

5771 (95.4%) 5632 calBC for 615.6: long bone fragment, RoAMS code 615.6

5901 (95.4%) 5714 calBC for 616.6: phalanx, RoAMS code 616.6

7031 (95.4%) 6691 calBC for 617.6 fraction

6074 (90.7%) 5979 calBC; 5945 (4.7%) 5924 calBC for 617.6HA1_HA2 fraction

6025 (94.5%) 5876 calBC; 5858 (0.9%) 5849 calBC for 617.6A fraction

5982 (9.9%) 5941 calBC; 5931 (85.5%) 5725 calBC for 617.6B fraction.

For the time horizon established by the relative chronology 5900–5800 BC/BCE, the result offered by the long bone fragment is not the expected one, providing a more recent date by approx. 200 years. Most likely, cross-contamination of collagen with newer humic materials, similar to those separated from the ceramic fragment material or different in origin, occurred. The resumption of the analyses on the phalanx leads to the convergence of the relative chronology with the absolute one; the result for sigma = 1 (probability of 68.3% that the age is within sub-interval), the most restrictive criterion, offers a refinement to **5839 (68.3%) 5740**

38 HEDGES/VAN KLINKEN 1992, 279–291.

calBC. It places the most probable moment of the animal's death somewhere before 5800 BC/BCE.

As for the four fractions, indeed Phases III and IV best characterize the moment of co-existence in the same time horizon of the vessel and the animal. Phase IV, considered to be the safest, shows an almost perfect overlap within the limits of the measurement errors for the **5931 (85.5%) 5725 calBC** interval, but at the same time, the restrictive criterion leads to the interval **5893 (68.3%) 5752 calBC**, very close to the similar one, obtained for the phalanx.

Obtaining convergent results by absolute chronology for the phalanx and the safe fractions isolated from in the ceramic fragment, the final combination of uncalibrated data through the same OxCal Program (IntCal 13) will lead to the histogram below (Fig. 11):

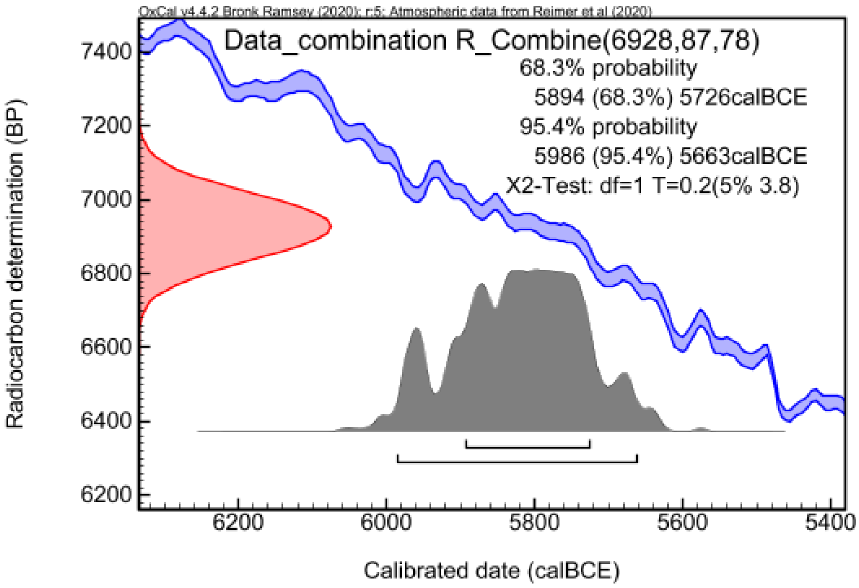

Fig. 11. The combination of uncalibrated radiocarbon data for phalanx and Phase IV separated from the ceramic fragment with chaff meets the convergence criteria

The centre of gravity of the histogram is placed in the range 5894–5726 cal BC/BCE and expresses, with maximum probability from the point of view of absolute chronology, the placement of the time horizon in the range 5900–5800 BC/BCE. However, the calibration curve in this region enters a plateau at the interval 5860–5700 calBCE, so that any value has practically the same probability of occurrence, limiting the performance of the result.

Comparison of the results of the four Phases for the ceramic fragment with those for bones reveals interesting information. For example, the contaminants in the post-depositional stage of the pottery are visibly older. Thus, one would expect the result for the long bone to have been altered in the same direction. As mentioned above, the results offered a visibly younger date than all the samples analysed. However, the value obtained for the phalanx places the death of the animal, with certain probability, a few decades after the formation of the vessel. Somehow, the piece was manufactured, fell out of use and entered the post-depositional period before and in a different way than the osteological material, the latter being affected, at the time of the animal's death, by newer exogenous carbon from higher, younger layers.

CONCLUSIONS

The ceramic material, just like the osteological one, is also subject to a series of contaminations, having the same exogenous sources in the post-depositional period. As only purified collagen is reliable, in the case of the carbon dating of ceramic fragments, certain fractions are only reliable if they undergo the same purification mechanisms. In the case of ceramics, it is easier to understand why too old or too new fractions appeared and, with an optimal separation strategy, how one can clearly detect them. Collagen is more complicated because: 1. such results can also occur due to humus from soils, peat, decomposed wood and so on, which can create cross-contamination reactions with collagen and non-collagenous bone proteins in the post-depositional period; and 2. such reactions can be accelerated in the first steps of pre-treatment in the laboratory, leading to the impossibility of their separation by traditional methods.

The context can provide much older exogenous carbon than that at the time of vessel formation, becoming a source of contamination that, if not carefully removed, can lead to a visibly older age than estimated. This is another borderline case compared to the most common encountered in dating pottery without chaff, in which younger ages can appear, although, at first sight, the mechanism and sources of contamination could be interpreted as being the same. Because ceramics store in the paste latent information on the age of carbon in the same time horizon in which other artifacts can be discovered (wood, osteological material, charcoal), they can provide information on the age and nature of contaminants, illustrating a possible scenario of their deposition over time. As well as why, if they are not effectively removed, these contaminants can lead to discrepancies between the ages of the samples in context or even incorrect estimates of the age of the analysed layer. These results may become visibly aged or rejuvenated, although interactions between the same contaminants and other types of material (wood, bones, etc.) may be slightly different than those with ceramics.

A carbon source older than the moment at which the vessel was formed might be the peat used for cooking or heating. This may be the oldest existing source of carbon for this study, and its interaction with the environment in the post-depositional stage may provide humus that is also old but, when combined with other humus in the soil layers, can lead to another apparent age, younger than the source fuel, but older than the chaff from which the ceramic vessel was made. This older exogenous carbon may be in excess in the first moments after the vessel entered another period of its existence, occupying the pores so that the humic materials formed naturally during the deposition of the upper layers of soil/sediment no longer had access to the empty spaces within the ceramic material.

The isolation, purification and dating of the OREC fraction becomes the most reliable source of information for the pottery (with chaff) if the pre-treatment strategy is carefully adapted to each case.

From a technical and methodological point of view, dating of the whole organic fraction in such cases is not reliable even if the mechanical and physical cleaning processes are considered to be efficient.

Separate dating of humin and humic acid fractions (as is usually done in the literature on other types of substrates, though not on ceramics) is not sufficient to obtain confidence in the result for the entire fraction, even if the results are relatively close.

The most reliable is the OREC fraction. Even if the other contaminants are removed with the usual Acid-Base-Acid treatment process, the final analyte may include natural chemical compounds containing exogenous carbon. These can lead to false positive or negative age values; the present case is eloquent in proving that exogenous fractions are not always newer than the original fraction in the chaff.

Finally, we can conclude that the methods used in the classical physicochemical pre-treatment procedure of the Acid-Base-Acid type as well as the processing of separate fractions, presented in the literature for samples of soil/sediment or for charcoal/microcharcoal, must be adapted to the type of composite ceramic material. The different acid concentrations as well as the succession of the use of admixtures (which ensure the digestion of samples), the precipitation of useful fractions and the oxidation of groups of compounds containing exogenous carbon must be chosen carefully to separate the characteristic fractions into groups of natural compounds. Their dating involves the detection of the scenario from the post-depositional period, the nature of the interactions with the environment and the types of contaminants.

After examining the arguments above, we can state with great confidence that the estimate obtained by relative chronology was correct and that the vessel from which the fragment came as well as the associated osteological material falls within the corresponding time horizon, ranging from 5900 to 5800 BC/BCE (Neolithic).

The C14 data series – both the one obtained on the phalanx of *Bos primigenius* and the one resulting from the analysis of the ceramic fragment – could represent a moment of abandonment of Cx. 3, especially as the two samples come from the upper part of the aforementioned feature. For a clearer assessment of the chronology of the oldest Neolithic horizon at the Folt-*Sub Vii* site, we propose that a systematic archaeological excavation of the feature area Cx. 3 is absolutely necessary in order to unearth other samples and compare the new ages with the data obtained by the authors of this study in the present scientific experiment.

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