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4 - RADIOCARBON DATES FOR THE SIUREN I SEQUENCE

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Twenty-seven charcoal and bone samples from Units F, G and H in Siuren I were sent by members of the 1990s excavation team to different C14 laboratories (Louvain-la-Neuve, Oxford, Groningen and Beta Analytic Inc.) in the 1990s and 2000s. Table 1 gives the results obtained (in stratigraphic order from top to bottom), while table 2 shows the (long) series of samples that failed, with laboratory comments.

Charcoal samples

The dating process of the Siuren I lower (Units H-G) and middle (Unit F) parts of the archaeological sequence began immediately with the site's new excavations undertaken from 1994-1997. During wall cleaning of Bonch-Osmolowski's 1926-1927 trench (sq. 12 B-H) in 1994, three charcoal samples and three ungulate bones were selected for dating of different levels in Units F and G (see Demidenko *et al.* 1998:377). The charcoal samples were sent by M. Otte to the Louvain-la-Neuve laboratory (Belgium) where two dates were obtained: 10,520 ± 150 BP (Lv-2131) for sub-level Fb2 of level Fb1-Fb2 (tabl. 1, #1) and 250 ± 60 BP (Lv-2132) for level Ga (tabl. 1, #5), while the third sample was not dated and sent back to Liège, with the following comment (translated): "*very nice charcoal, but less than 0.5 gr; see AMS*" (letter from Ét. Gilot, Université catholique de Louvain, Unité de Chimie inorganique, analytique et nucléaire, 17 December 1994) (see tabl. 2, #14a). This sample was then sent to Oxford, but with no better result, due to unusual $\delta^{13}C$ (-27.1‰) (Clare Owen, fax from the Oxford University Radiocarbon Accelerator Unit, 25 February 1998) (see tabl. 2, #14b).

The dates on charcoal have been considered as being certainly too young for any Upper Paleolithic. They are likely due to the presence of modern plant roots along the 1920s trench walls.

Bone samples

The three ungulate bones were sent by M. Otte to the Oxford laboratory (United Kingdom) and two of the bones contained enough collagen for AMS dating. As a result, the bone sample from sub-level Fb2 of level Fb1-Fb2 yielded the result of 29,950 ± 700 BP (OxA-5155) (tabl. 1, #3) and the bone sample from level Ga was dated to 28,450 ± 600 BP (OxA-5154) (tabl. 1, #6). The bone sample from level Fb2, west section, "*was not dated because the bone gave an unusual $\delta^{13}C$ when we combusted it, which implies some sort of contamination or degradation of the collagen. Rather than have an unreliable radiocarbon, we decide to abandon the analysis*" (letter from R.E.M. Hedges, Oxford University Radiocarbon Accelerator Unit, 20 June 1995) (see tabl. 2, #15).

Due to the very unsuccessful attempts in charcoal sample dating, it was been then decided to use ungulate bone samples only for any new AMS dates at Siuren I. The lowermost archaeological subdivision of the site (Unit H) was dated in the late 1990s on an ungulate bone, again by Oxford; the result obtained very similar to the two previous ones: 28,200 ± 440 BP (OxA-8249) (tabl. 1, #12). But the four other samples sent to Oxford at the same time for Units G and H "*all failed to produce dates. A report from the Chemistry laboratory indicates that all of these samples failed to yield sufficient collagen to date*" (letter from D. Jenkins, Oxford University Radiocarbon Accelerator Unit, 12 January 1999) (see tabl. 2, #17, 20, 21 and 25).

State-of-the-art in the late 1990s

On the basis of the three statistically identical Oxford AMS dates, additionally taking into consideration the very rapid sedimentation processes that took place in the rock-shelter during the deposition of these cultural bearing sediments (Bonch-Osmolowski 1934; Gromov 1948; Ivanova 1969, 1983; Chabai 2000, 2004; Demidenko 2000), and the fauna, microfauna and malacofauna data (López Bayón 1998; Markova, this volume; Mikhailesku, this volume), the following geochronological positions have been proposed for the two Paleolithic find complexes from Units H-G and from Unit F (Demidenko 2000, 2002b).

The 1990s Units H-G (corresponding to the 1920s Lower layer) with several successive visits to the rock-shelter by Neandertals of Crimean Micoquian Tradition (with a few finds) and by Early/Archaic Aurignacian of Krems-Dufour *Homo sapiens* (identified through very numerous artifacts), were considered as belonging to Arcy Interstadial (*ca.* 31,000-30,000 BP). The 1990s Unit F (corresponding to the 1920s Middle layer) con-

tains occupations only by *Homo sapiens* of the Late/Evolved Aurignacian of Krems-Dufour type tradition, either at the end of the Arcy Interstadial (ca. 30,000 BP), or, more likely, during the Maisières Interstadial (ca. 29,000-28,000 BP).

After such geochronological interpretations, members of the Siuren I 1990s excavation team continued to accept them (e.g. Demidenko & Otte 2007; Demidenko 2008a, 2008b; Chabai 2004a) being aware, at the same time, of doubts by some colleagues regarding both industrial and geochronological interpretations. On one hand, some Russian and Ukrainian colleagues (e.g. Anikovich 2003; Sapozhnikov 2002, 2005) continued to consider the Siuren I Early/Archaic and Late/Evolved Aurignacian of Krems-Dufour type finds complexes as either uncertain Aurignacoid or Gravettoid-Epi-Aurignacian and Aurignacoid-Epigravettian ones, absolutely rejecting their Aurignacian *sensu stricto* attribution and placing them geochronologically in different sub-periods of the Last Glacial Maximum (22,000-18,000 BP). None of our arguments based on the Siuren I 1990s excavation data (e.g. Chabai 2004a: 27-30; Demidenko 2000, 2002b, 2008a, 2008b; Demidenko & Nuzhnyi 2003-2004) were taken into consideration by the ex-USSR colleagues and it was quite impossible to imagine what else we could do to convince them.

Only the opinions of Western colleagues might help in future to change this "Eastern problem". However, at the moment, most of our Western colleagues either remain silent on the Siuren I Upper Paleolithic complexes after the 1990s excavations in their European Aurignacian-related publications, or actually support some strange interpretations proposed by Anikovich and Sapozhnikov, like the following: "*Siuren 1 (Crimea) (Vekilova 1957; Otte et al., 1996). Level Fb1 = late Aurignacian = 29 550 BP (?) or mixed Mousterian – Epigravettian layer (?)*" (Djindjian *et al.* 2003:42). The "Western problem" is connected to the proposed Siuren I Archaic Aurignacian geochronology. Often verbally accepting the proposed Aurignacian archaeological definitions for Siuren I during different conference paper presentations, including those for the 1990s Units H-G, Early/Archaic Aurignacian of Krems-Dufour type being an actual equivalent for the more common terms such as Aurignacian 0/Archaic Aurignacian/Proto-Aurignacian with Dufour microliths of Dufour sub-type, the vast majority of our Western colleagues were usually not able to agree with our geochronology for these Siuren I Aurignacian finds (Arcy Interstadial and the 1990s two AMS OxA dates around 28,000 years BP). Such a negative opinion is, to some extent, understandable as most of the Proto-Aurignacian assemblages in Western Europe are now usually radiocarbon dated to a period of 37,000-36,000 to 34,000-33,000 BP. Therefore, the Arcy Interstadial time period for the Siuren I Proto-Aurignacian is still "too late" for most of our Western colleagues.

What was (and is) still possible to say regarding the geochronological issue?

The simple answer is that the 30,000-28,000 years BP period is still within the Aurignacian time span and not part of the much younger LGM, as was previously suggested by some Eastern European colleagues. Therefore, it cannot be excluded that *Homo sapiens* with the so-called European Proto-Aurignacian did indeed penetrate into the south of Eastern Europe after their occupation of the southern and middle territories of Western and Central Europe, which could explain why their best-represented site in Eastern Europe, Siuren I, would contain such "late" archaeological levels. Moreover, the vast territories of Eastern Europe certainly have a poor representation of Aurignacian *sensu stricto*, whether *in situ* sites or find spots with abundant and industrially very clear flint assemblages (Demidenko 2004b, 2009), that makes it difficult to evaluate the directions of Aurignacian *Homo sapiens* movements into Eastern Europe.

Unfortunately, the Siuren I Proto-Aurignacian geochronological problem has also automatically led to an almost complete silence on the Siuren I Late/Evolved Aurignacian and its great importance for the entire European Late/Evolved/Recent Aurignacian. Although the most complete understanding at present of this Aurignacian industrial and geochronological stage (ca. 32,000-28,000) has been made on the basis of Western European materials (e.g. Demars 1992; Demars & Laurent 1989; Djindjian 1993a, 1993b, Rigaud 1983, 1993, 2000; Bordes 2005), the Siuren I related Unit F flint finds not only fit perfectly into the French Late/Evolved/Recent Aurignacian data with its "whole carinated piece package" (bladelet "carinated" cores and both carinated end-scrapers and burins), but also with the single 1990s OxA AMS date around 29,000 BP. The Siuren I Unit F Late/Evolved Aurignacian is also characterized by the largest sample of Dufour and pseudo-Dufour microblades of Roc-de-Combe sub-type (68 specimens) for all of Europe. Additional technological studies of the Siuren I materials should contribute significantly to understanding of the European Late/Evolved Aurignacian.

All in all, the Aurignacian data obtained from the Siuren I 1990s excavations have not been much accepted as such by most of our colleagues, either in the East or in the West. Therefore, a new dating program was undertaken. At the end of the 2000s, new possibilities opened to obtain absolute dates, specifically AMS, for Units H-G and F.

Bone artifacts

Bone artifacts are present in both Units F and G (the Unit H assemblage lacks bone artifacts), with five items in F and eight items in G, including two retouchers about which it has been argued (after the site's 1990s excavations; e.g. Demidenko 2000) that they actually belong to Middle Paleolithic (Micoquian) Neandertal occupations, while the other items (five points and an awl) are associated with Upper Paleolithic (Archaic Aurignacian) *Homo sapiens* occupations during the rapid depositional processes of Unit G. As a result, with the dating of these bone artifacts, there was a chance to obtain, not only dates for Unit G, but possibly *separate* dates for the Micoquian and Archaic Aurignacian occupations for Siuren I, Unit G. It was also hoped that if new AMS dates for Unit F were older, even slightly older (ca. 32,000 BP), than the ones from Unit G, this would directly point out to a series of problems with collagen preservation for the Unit G ungulate bones, which we suspected.

The possibility of direct AMS dating of the Siuren I bone artifacts resulted from an agreement with Ph. Nigst (then at the

Max Planck Institute for Evolutionary Anthropology, Leipzig). In December 2009, S. Talamo, a specialist in AMS dating, took samples from five bone artifacts from Unit G and three from Unit F, after use-wear analysis was concluded (see Demidenko & Akhmetgaleeva 2008). Bone sample pretreatment was conducted by her at the Max Planck Institute, with treated samples to be sent later to Oxford for AMS dating. The pretreatment analysis was absolutely disappointing for Unit G: none of the five samples had enough collagen for radiocarbon dating (mail from Ph. Nigst, 12 October 2010). On the other hand, all three samples from Unit F bone tools had both good quantities of collagen and very good C/N ratios, so their "final samples" were sent to Oxford for dating. Results will be published by Demidenko, Nigst and Talamo, and cannot be reproduced here, but fall within the interval of ca. 28,500-26,500 BP (Ph. Nigst, pers. comm.).

More bone samples

In addition to the dating program on bone artifacts, new attempts were done on ungulate bones untreated by Paleolithic humans from Units H, G and F. Most of the fauna from Units H, G and F from the rock-shelter's 1990s excavations was in Paris for zooarchaeological analysis by M. Patou-Mathis, while a few bones from Units H, G and F were also stored at the University of Liège, specifically selected during and immediately after the 1995-1997 excavations for future AMS dating.

So, initially for new dating, eight bones from Paris were sent by M. Otte to Beta Analytic Inc. (Florida, USA) in the first half of 2009 and two bones were determined to having sufficient collagen. The two uncalibrated AMS dates obtained are associated with the rock-shelter's lower cultural bearing sediments: 28,070 ± 190 BP (Beta-260919) for sub-level Gb1 and 30,490 ± 220 BP (Beta-260924) for Unit H. These Beta dates are again in accordance with the previously obtained AMS dates for Siuren I lower and middle cultural bearing sediments.

The six samples that were not dated "*did not yield any separable collagen and cannot at that time be dated*" (mail from Chris Patrick, Beta Analytic Inc, 10 July 2009) (tabl. 2, #16, 18, 22-24 and 26). They came from Units F to H, with four belonging to Unit G.

Bone cross-samples

Next, new dating attempts were made on bones from Liège with the following idea: to obtain two sets of dates in Groningen (the Netherlands) and Oxford, on six samples from three ungulate bones, for Units F, G and H. The three bones were each cut into two parts by P. Haesaerts in December 2009.

Half of the bone from sub-level Fb2 was sent by P. Noiret to Groningen, while the other half was brought by Ph. Nigst to Leipzig (Max Planck Institute) for pretreatment, before being sent to Oxford. For this specific sample, the two dates obtained are almost identical: 30,910 ± 240 BP in Groningen (GrA-46552; C/N: 3.6) (tabl. 1, #4) and close to 30,300 in Oxford (Ph. Nigst, pers. comm.; to be published by Demidenklo, Nigst and Talamo). And Haesaerts's comment about the cutting process was that only this bone from Unit F "smelled good", indicating (1) that its organic component was well preserved and (2) that the two other bones (not "smelling") were less well preserved, probably mineralized. And, indeed, no better results than previously were obtained for Units G and H…

Pretreatment in Leipzig for the bones from Units G and H indicates "*too few collagen preserved*" (mail from Ph. Nigst, Max Planck Institute for Evolutionary Anthropology in Leipzig, 9 August 2010), whereas Noiret sent the other parts of these two bones to Groningen. The bone from sub-level Gb2 "*did not contain enough carbon and could not be measured*" (letter from J. van der Plicht, Rijksuniversiteit Groningen, Centrum voor IsotopenOnderzoek, 1 June 2010). The bone from Unit H gave a result, but it certainly indicates too young an age: 22,040 ± 120 BP (GrA-46553) (tabl. 1, #11). Having not had a "good smell" during the cutting process could indicate a low collagen content for these samples from Units G and H. We also have to say that J. van der Plicht "*can not find anything wrong with the 22k date. It is simply measured like this. So either it is truly that old, or it is contaminated somehow, or the association is wrong*." And when asked about the C/N ratio, van der Plicht added: "*We do not have Nitrogen numbers for this bone; we had used all collagen for the C isotopes (incl. dating). The C content (C%) is not great but acceptable, according to experience. But not enough for nitrogen, hence we do not have C/N. Perhaps that is a bad sign and the bone indeed is not well preserved […]. We only have the complete analysis for the [Fb2] sample, which appears to be the only acceptable sample for this Siuren series*" (mail from J. van der Plicht, 27 January 2011). And the words "not well preserved" means that "*the organic yield was lower than for a 'normal' bone*" (mail from J. van der Plicht, 22 March 2011).

Considering the two results of 29,000-30,000 BP obtained at that time for Unit F, it is not possible to consider that the Unit G could be "truly that old" (*i.e.* 22,000 BP). Considering the consistency of the lithics and the amount of failed samples for Unit G and Unit H due to low collagen content (see tabl. 2), it is quite unlikely that the association is wrong. So, van der Plicht's third explanation is our favorite: the samples are themselves problematic, contaminated one way or another, probably poorly preserved in both Units G and H, and with contamination not successfully removed during pretreatment, as we suspect when considering the last series of bone samples described below.

"Last" bone samples

Finally, a last (almost desperate) attempt to obtain results took place in 2011. P. Noiret sent another set of five bone samples (stored in Liège since the 1990s excavations) to Beta Analytic, which yielded four results, but no clear solution to the question of the age of Siuren I's industries. The sample from sub-level Fb2 provided the following result: 29,440 ± 200 BP (Beta-293364) (tabl. 1, # 3), in remarkable accordance with the other results obtained earlier for this sub-level.

But the three samples from sub-unit Gb2 all gave younger ages: 13,020 ± 70 BP (Beta-293363), 19,680 ± 100 BP (Beta-293661) and 22,220 ± 120 BP (Beta-293362) (tabl. 1, # 7-9). The comment from Beta for these four samples says, surprisingly, that "*they each provided plenty of carbon for accurate measurements and all the analyses proceeded normally*" (letter from D. Hood, Beta Analytic

Inc., 10 March 2011). And the fifth sample (sub-unit H; tabl. 2, #27) again "*did not yield any separable collagen and cannot at this time be dated*" (mail from Chr. Patrick, Beta Analytic Inc., 25 February 2011). This last attempt thus provided no reliable data for Units G and H.

Discussion

It is still possible to continue dating other ungulate bones from Units G and H at Siuren I, but is it worth it? We can, at any rate, discuss the series of existing dates for Unit F, on one hand, and Units G and H, on the other hand.

The Unit F AMS dates can be presented in stratigraphic order from top to bottom of the archaeological level sequence (tabl. 1), remembering that some artifacts were subject to vertical movement within the unit, as shown by refitting of the Unit F flints by Demidenko. So, all of the AMS dates for the Unit F sequence are between ca. 31,000 and 26,500 uncal BP, and it is probably possible to narrow this range to ca. 31,000-28,000 BP if we consider only sub-level Fb2. These dates are fully in accordance with the known Western European Late/Evolved/Recent Aurignacian, when, of course, the dates of such are reliable.

The AMS dates for the site's lower cultural bearing sediments (Units H and G) are less consistent, but still merit consideration. The dates, when considered from the bottom to the top of the sequence, show the following two-fold results. On one hand, the dates are virtually the same as those in Unit F, being between 30,000 and 28,000 uncal BP; thus, the already postulated rapid sedimentation processes at Siuren I rock-shelter for Units H through F might have further support.

On the other hand, it is worth recalling that the five bone artifacts from Unit G had insufficient collagen for AMS dating, whereas all of the Unit F bone artifacts had sufficient collagen for dating. Similarly, only two bones from the faunal remains gave no results for Unit F, while this was the case for eight bones from Unit G and three from Unit H (see tabl. 2). This may indicate overall poor bone preservation in the Units H and G deposits in terms of collagen content, which is why the AMS dates obtained may be too young. If this is true, then indeed the Siuren I Proto-Aurignacian find complexes should be older, perhaps as in Western Europe, somewhere between 37,000-36,000 to 34,000-33,000 BP.

The stratigraphy of the Siuren I/Units H-F sediments allows us to put forward a hypothesis on such AMS dating results for Unit F, on one hand, and Units H-G, on the other hand. It is possible that some difference in the presence of limestone *éboulis* influenced bone preservation throughout the Siuren I archaeological sequence (Yevtushenko, this volume). The Unit F deposits, excavated in the 1990s in a 12 sq.m. area, are characterized by medium to low occurrences of angular limestone *éboulis* within varying silty clayey and loamy sandy loose sediments (lithological strata 10 through 12). In contrast, Unit G deposits (lithological strata 14 through 16) for the same 12 sq.m excavated in the 1990s, are mainly characterized by the presence of very numerous angular limestone *éboulis* within different sandy sediments. Accordingly, limestone *éboulis* are much more common within Unit G deposits than in Unit F and may have had some influence on ungulate bones. At the same time, the single archaeological level (lithological stratum 17) in Unit H is separated from the overlying Unit G sediments by a thick and solid limestone block, and seems much more similar to the Unit F sediments than to Unit G, identified within a dark yellowish-brown clay with rare limestone *éboulis*. Thus, the "bad" limestone *éboulis* might play some role for Unit G dating, but not for Unit H.

Final considerations

Of course, there is a question – what can be done to make the absolute chronology for Siuren I, Units H and G clearer? It is quite probable that we should change the datable material, which is not at all a simple solution, as we will see below.

First, there was some discussion between Yu. Demidenko and D. Richter (Max Planck Institute for Evolutionary Anthropology, Leipzig) about the possibility of using TL dating at Siuren I. The TL solution, however, cannot be applied as thick burnt flints are virtually absent in assemblages from Units H and G. At best, only a couple of flints have a maximum thickness of about 5 mm. Possible future excavations at Siuren I, which would be in a very limited area (*ca.* 2-3 sq.m.), would probably not recover thick burnt flints there, given their absence in the 1990s 12 sq.m excavations.

Second, these possible future and limited excavations might lead us to find samples of material that was in the 1990s the "good datable material" – charcoal. Most of the 1990s archaeological levels in both Unit F and Units H-G contain fireplaces and/or hearths, in addition to some ashy clusters, although, most important for this subject, only hearths in level Fb1-Fb2 (actually, in sub-level Fb2) contained definite charcoal pieces, while, aside from only one fireplace in Unit H (object #1) with some small charcoal fragments, the hearths and fireplaces in the levels of Units H and G lacked charcoal, having only ashy fill. Accordingly, both dating of ash and chances of finding good charcoal pieces in hearths/fireplaces during any new limited excavations at the site do not seem to be very realistic. The possibility to have in the future two sets of AMS dates – on charcoal and bone samples – for the site's lower cultural bearing sediments for comparison appears to be unlikely.

Third, the 1990s excavation find complexes of Units H through F also contain, aside from beads of fossil marine shells *Apporhais pes pelicani* in Unit G, some shell beads of freshwater river mollusks, terrestrial snails and/or marine mollusks that were contemporaneous with Palaeolithic human occupations during sedimentation processes of these archaeological units. These shell beads can be directly AMS dated. Such an attempt is worth trying for Siuren I as it is not reliant on new excavations at the rock-shelter; some very new shell AMS dates show very promising results (see Douka, in press, for level IX of Ksar Akil, Lebanon).

#	Unit	Level	Year excav.	Square	Material	Date BP	Sigma	Laboratory	Year process.	δ 13C (0/00)
1	F	Fb2	1994	profile II of trench 1927	charcoal	10520	150	Lv-2131	1995	unknown
2	F	Fb2	1995	8E	bone	29540	200	Beta-293364	2011	-19,1
3	F	Fb2	1994	profile I of trench 1927	bone	29950	700	OxA-5155	1995	-19,2
4	F	Fb2	1995	8E	bone	30910	240	GrA-46552	2010	-19,64
5	G	Ga	1994	profile I of trench 1927	charcoal	250	60	Lv-2132	1995	unknown
6	G	Ga	1994	profile II of trench 1927	bone	28450	600	OxA-5154	1995	-19,2
7	G	Gb1	1995	8C	bone	28070	190	Beta-260919	2009	-20,0
8	G	Gb2	1995	8C	bone	13020	70	Beta-293363	2011	-20,0
9	G	Gb2	1995	8C	bone	19680	100	Beta-293361	2011	-19,6
10	G	Gb2	1995	8C	bone	22220	120	Beta 293362	2011	-20,6
11	H	H	1997	6E	bone	22040	120	GrA-46553	2010	-20,0
12	H	H	1997	6D	bone	28200	440	OxA-8249	1998	-17,8
13	H	H	1997	6D	bone	30490	220	Beta-260924	2009	-17,7

Table 1 - Siuren I. Radiocarbon datings.

#	Unit	Level	Year excav.	Square	Material	Laboratory	Year process.	Comment
14a	F	Fb1	1994	profile I of trench 1927	charcoal	Louvain	1995	too small
14b	F	Fb1	1994	profile I of trench 1927	charcoal	Oxford	1995	OxA-6987 ; δ 13C = -27,1
15	F	Fb2	1994	profile II of trench 1927	bone	Oxford	1995	unusual δ 13C
16	F	Fb2	1995	8C	bone	Beta	2009	not any separable collagen
17	G	Gb2	1996	6C	bone	Oxford	1998	low collagen
18	G	Gb2	1996	7C	bone	Beta	2009	not any separable collagen
19	G	Gb2	1995	8C	bone	Groningen	2010	not enough carbon
20	G	Gc1	1996	8C	bone	Oxford	1998	low collagen
21	G	Gc1	1996	8D	bone	Oxford	1998	low collagen
22	G	Gc1	1996	8D	bone	Beta	2009	not any separable collagen
23	G	Gc2a	1996	7D	bone	Beta	2009	not any separable collagen
24	G	Gd	1996	6D	bone	Beta	2009	not any separable collagen
25	H	H	1996	9D	bone	Oxford	1998	low collagen
26	H	H	1997	6D	bone	Beta	2009	not any separable collagen
27	H	H	1997	6E	bone	Beta	2011	not any separable collagen

Table 2 - Siuren I. Unsuccessfull samples.