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WHEN ^{14}C DATES FALL BEYOND THE LIMITS OF UNCERTAINTY: AN ASSESSMENT OF ANOMALIES IN WESTERN MEDITERRANEAN BRONZE AGE ^{14}C SERIES

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ABSTRACT. The aim of this article is to bring to light a serious problem affecting radiocarbon dates produced at least from 2009 onwards by the AMS Leibniz laboratory at the Christian-Albrechts-Universität (Kiel, Germany). Archaeological observations and cross-checkings between several laboratories confirm that in a significant number of dates, clear deviations of the results from chronological schemes based on stratigraphical sequences and hundreds of measurements have occurred, which usually implies an aging of the ^{14}C values.

INTRODUCTION: EL ARGAR BRONZE AGE CHRONOLOGY AND LA BASTIDA PROJECT

The absolute chronology of the western Mediterranean during later prehistory is a field of inquiry that has experienced significant development since the early 1990s. Greater awareness of the potential of radiocarbon dating from well-contextualized samples allowed to overcome former traditions relaying on cross-cultural dating (Renfrew 1973; González Marcén et al. 1992). As a result, fine-grained periodizations are available for several regions and periods, reaching a level of precision and detail comparable or even higher than that currently used in the study of ancient eastern Mediterranean urban societies.

El Argar is one of these Iberian archaeological groups having an excellent ^{14}C database. From the late 19th century onwards, and thanks to the discoveries of Siret and Siret (1887), El Argar has been recognized as one of the "classic" European Bronze Age societies. The Argaric archaeological record of southeast Iberia includes a complex settlement pattern covering different ecological niches, large permanent settlements formed by a dense network of stone-built domestic and public precincts, single or double intramural inhumations, and highly standardized ceramic, metallic (copper, bronze, and silver) and lithic assemblages. Most scholars agree that the Argaric political organization was a highly stratified or state society, and that gender and class differences and asymmetries articulated social relations.

At the end of the 1980s, we started a ^{14}C dating program aimed at establishing a chronological framework for the later prehistory of southeast Iberia (Castro et al. 1993–1994, 1995, 1999; Lull 2000). Given the advantages of accelerator mass spectrometry (AMS) dating, it became possible to date not only charcoal, but also seeds and bone samples coming from closed settlement and funerary contexts. Today, excluding the new dates from La Bastida and Tira del Lienzo, which will be discussed below, nearly 300 ^{14}C dates are available, most of them corresponding to short-lived samples coming from well-preserved contexts. As a result, the absolute chronology of El Argar as a whole can be placed between about 2200 and 1550 cal BCE (Lull et al. 2005, 2010, 2011, 2013). A tripartite internal phasing has been proposed, and more detail is reached in multistratified sites with a succession of intramural burials. This has allowed tracing of the development of specific pottery and metal types along the nearly 7 centuries of El Argar by combining ^{14}C and stratigraphic data (Castro et al. 1993–1994, 1999; Lull 2000; Lull et al. 2011, 2013). In sum, the Argaric archaeological record is one of the most firmly dated Early Bronze Age groups in Europe.

The interdisciplinary La Bastida Project (http://www.la-bastida.com) was set up in 2008 to study one of the largest known Argaric settlements. La Bastida is a 4.5-ha hilltop settlement located in the province of Murcia (Spain), between the mountain ranges of Sierra de la Tercia and Sierra Espuña, on the left bank of the narrow riverbed named Rambla de Lebor. The excavations carried out so far

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in La Bastida over a surface of 6200 m^2 have revealed a deep stratigraphic record probably covering the complete Argaric timespan. Architectural remains, including an impressive fortification system and a large water reservoir, point to an urban status, suggesting that we are dealing with a political and economic center of regional scale, perhaps with a capacity to influence the whole Argaric territory (Lull et al. 2014).

About 7 km east of La Bastida, another stratified Argaric settlement named La Tira del Lienzo has been excavated in the context of the project. In this case, we are dealing with an exceptional architectural complex located on a small hilltop in the margins of the fertile Guadalentín Valley. A rectangular central building is surrounded radially by small rooms and the whole complex is enclosed by a wall. According to cross-dating criteria based on pottery types, this site was occupied during the middle and late phases of El Argar (about 2000–1550 cal BCE).

The dating program carried out in La Bastida and Tira del Lienzo aims to define the general chronology of the occupation in both sites, its architectural development through time, and to refine the chronology of specific Argaric artifact types and funerary contexts. The starting conditions for such a program are excellent, since we benefit from the previous knowledge on the general Argaric chronology. Moreover, both sites have provided a large number of well-preserved domestic and funerary contexts and abundant short-lived samples, such as charred seeds, faunal remains, and human bones.

DATING PROBLEMS IN ARGARIC CONTEXTS

Excavations at La Bastida and Tira del Lienzo between 2009 and 2011 provided organic samples from particularly relevant and well-defined occupation layers or sealed contexts, such as tombs. As the excavations developed, we submitted 48 samples to the Leibniz Labor für Altersbestimmung und Isotopenforschung, Christian-Albrechts-Universität (Kiel, Germany): 40 were bone (human and animal) and the remaining 8 charred botanical material. Soon, it became clear that the preservation of collagen in certain human and faunal bones was average to poor, contrary to the situation observed in other Argaric settlements. The quality of samples was established in the usual way based on collagen yield, carbon content, and C:N ratio of the extracts. In total, 13 bone samples out of 40 from La Bastida proved to be problematic in terms of collagen preservation and had to be discarded.

The results from the first set of samples were reported by the Leibniz Labor in July 2009 and several inconsistencies were observed, either between the dates and their archaeological context or between bone and botanical samples of the same archaeological context. In general, the impression emerged that *some* dates were older than expected, when considering stratigraphic data and typological criteria from other well-dated Argaric contexts. Later, ^{14}C dates of *some* carbonized samples also turned out to be significantly older than their archaeological contexts. We will describe three specific contexts, which showed that a problem was affecting dating procedures at the Leibniz laboratory.

Burial 23 from La Bastida Building 3

Building 3 belongs to the last architectural phase of La Bastida, which corresponds to the Late Argar period (about 1750–1550 cal BCE). Inside a bench, a typical burial in a ceramic vessel (*pithos*) was discovered, containing the remains of two children. The disarticulated bones of child BA23/1 were carefully placed on top of the articulated skeleton of BA23/2, implying that a certain timespan elapsed between both funerary events. The ages at death of BA23/1 and BA23/2 were respectively 9–11 months and 7–10 months old. Both individuals were sampled for ^{14}C dating (Table 1).

Table 1 Radiocarbon results for the double burials BA23 and BA18 (*calibration using CALIB 7.0.1. Stuiver and Reimer 1986; IntCal13. Reimer et al. 2013; NR: not reported).

Context	Sample	Report date	Lab nr	BP	*cal BC 2σ	Collagen yield (%)	C content of collagen (%)	C:N
BA 23/1	First sample (two left 2/10 ribs)	Nov. 2009	KIA-40014-1.1	3975 ± 25	2570–2513 (51.9%) 2503–2461 (48.1%)	3.33	46.10	NR
BA 23/1	First sample remeasurement	July 2010	KIA-40014-1.2	3785 ± 25	2288–2141 (100.0%)	NR	NR	NR
BA 23/1	Second collagen extraction from the first sample	July 2010	KIA-40014-2	3405 ± 30	1858–1854 (0.5%) 1771–1624 (99.5%)	3.89	49.34	NR
BA 23/1	Third collagen extraction from the first sample	June 2013	KIA-40014	3450 ± 24	1877–1839 (20.6%) 1827–1794 (11.0%) 1784–1690 (68.4%)	NR	41.37	NR
BA 23/1	Second sample (first left rib)	July 2010	MAMS-10991	3427 ± 26	1872–1844 (7.2%) 1813–1801 (1.8%) 1777–1658 (90.5%) 1650–1646 (0.6%)	NR	NR	NR
BA 23/2	First sample (left femur fragments)	Nov. 2009	KIA-40015-1	3360 ± 20	1732–1719 (3.5%) 1692–1614 (96.5%)	5.29	45.90	NR
BA 23/2	First sample remeasurement	July 2010	KIA-40015-2	3420 ± 25	1866–1849 (3.3%) 1773–1642 (96.7%)	5.05	50.25	NR
BA 23/2	Second collagen extraction from the first sample	June 2013	KIA-40015	3480 ± 24	1883–1742 (98.5%) 1708–1702 (1.5%)	NR	41.42	NR
BA 18/2 latest inhumation	Femur fragment	Jan. 2010	KIA-40012	3609 ± 24	2029–1901 (100.0%)	1.41	44.4	3.6
BA 18/2 latest inhumation	Remeasurement of femur sample	June 2013	KIA-45101	3568 ± 29	2020–1993 (5.9%) 1982–1875 (86.0%) 1843–1817 (5.0%) 1798–1780 (3.1%)	NR	36.22	NR
BA 18 barley from bowl	Analysis of humic acids	Nov. 2009	KIA-40100	3400 ± 20	1746–1637 (100.0%)			
BA 18 barley from bowl	Analysis of alkali residue	Jan. 2010	KIA-40100-2	3410 ± 62	1885–1603 (95.6%) 1584–1544 (4.1%) 1538–1535 (0.3%)			
BA 18 barley from sediment	Analysis of humic acids	Nov. 2009	KIA-40099	3405 ± 20	1747–1643 (100.0%)			

Figure 1 Double child inhumation in pithos burial (BA 23). All bones from infant 23/1 lie on top of 23/2, whose skeleton rests fully articulated on the bottom of the pot.

Surprisingly, although the quality of bone preservation was good, the date of the first interment (BA23/1: KIA-40014-1.1) was about 800–900 calendar years older than the second (BA23/2: KIA-40015-1). In fact, KIA-40014-1.1 points to a Copper Age chronology, 3 centuries before the beginning of the Bronze Age and long before La Bastida was settled. The Leibniz lab repeated the measurement of the first collagen extraction (KIA-40014-1.2), resulting in a new date 200 ^{14}C yr younger than the first one but still too old, pointing to the transition between Copper and Bronze Ages, before the Argaric practice of child burial in ceramic vessels was established. Because of this discrepancy and after expressing our doubts, the Kiel laboratory repeated the collagen extraction for both BA 23/1 (KIA-40014-2) and BA 23/2 (KIA-40015-2). Simultaneously, a different sample of BA 23/1 was sent to the ^{14}C laboratory in Mannheim, Germany (Curt-Engelhorn-Zentrum für Archäometrie). This time, these three new dates were consistent with each other and in agreement with the archaeological context. In June 2013, the Kiel laboratory provided new dates for both skeletons using newly prepared material from the original samples. These results are also consistent. Neither Kiel nor Mannheim reported any difficulty with regard to the quality of the material.

Burial 18 from La Bastida Building 3

Burial 18 is a large ceramic jar with the remains of two adult men. It was found under the floor of the same building where tomb 23 was discovered (Figure 2). The age-at-death estimates are 22–27 yr old for the first inhumation (BA-18/1) and 20–25 yr for the second (BA-18/2). Among their grave goods was a bowl that contained carbonized barley seeds (identification provided by Hans-Peter Stika, U. Hohenheim). Barley was also recovered from the sediment inside the tomb.

Bone and seed samples were chosen in order to establish the absolute chronology of this burial belonging to the final occupation phase of La Bastida. Foot bones from both skeletons provided insufficient collagen and were discarded. A new sample from the femur of the second internment had a poor collagen yield but was considered acceptable and therefore dated (KIA-40012). Nevertheless, this date is nearly 3 calendar centuries older than the three dates from the barley seeds, which provide a very consistent result both as a group, as well as in relation to the expected chronology of the occupation of Building 3. In 2013, a new measurement from the BA-18/2 femur sample (KIA-45101) gave a slightly younger result, but still far from being in agreement with the dates from the seeds.

Figure 2 Double burial of two adult men in tomb BA 18. Barley seed samples where found inside the bowl on top of BA-18/2 legs, which is seen in the right side of the image.

Building 7 of Tira del Lienzo

A concentration of charred *Pistacia lentiscus* seeds was recovered from the floor level of Building 7. These seeds were well preserved because Building 7 burned down, sealing a set of artifactual and organic materials. These botanical remains were identified by H-P Stika (University of Hohenheim), and a sample was sent to the Kiel laboratory in August 2010. The ^{14}C result (KIA-43101) was about 1000 calendar years earlier than the expected date, pointing to pre-Beaker Chalcolithic times and 500 yr earlier than the beginning of the Bronze Age. A second set of seven seeds dated by Kiel gave a comparable result (KIA-43101-A) (Table 2). As mentioned in the Introduction, no previous Tira del Lienzo evidence can be dated earlier than ~2000 BCE.

Given these unexpected results, a third set of seeds from the same *Pistacia* concentration was sent to the Mannheim laboratory and a fourth one to the ^{14}C laboratory of the Museum of Anthropology in Montevideo (Uruguay). Both new dates (MAMS-11818 and URU-0558=URU-0559) (Table 2) fell into the expected timespan of the first quarter of the 2nd millennium cal BCE, thereby indicating that the two initial results from Kiel were extreme outliers.

In order to clarify the anomalous results, Kiel sent to Mannheim three pretreated samples (KIA-43101 original, KIA-43101, and KIA-43101-A) from the two sets already measured. The new dates (MAMS-13125, -13124 and -13123) differed greatly from former Kiel results and matched both Mannheim's and Uruguay's. This cross-checking showed that Kiel measurements were incorrect and needed further clarification. A new preparation measured in Kiel in 2013 produced a closer date, although still slightly older than the results from the other two laboratories (Table 3).

Table 2 Radiocarbon dates of *Pistacia* seeds from Tira del Lienzo Building 7.

Context	Sample	Report date	Lab nr	BP	cal BC 2σ
TL-H7-88	1st set of seeds	Sept. 2010	KIA-43101	4125 ± 30	2867–2803 (27.2%) 2777–2581 (72.8%)
TL-H7-88	2nd set of seeds	Sept. 2010 & Jan. 2011	KIA-43101-A	4075 ± 30	2855–2812 (14.4%) 2746–2725 (2.7%) 2697–2562 (71.0%) 2534–2493 (11.9%)
TL-H7-88	1st or 2nd set of seeds	June 2013	KIA-43101	3545 ± 25	1952–1866 (69.0%) 1849–1774 (31.0%)
TL-H7-88	3rd set of seeds	Dec. 2010	MAMS-11818	3495 ± 25	1889–1747 (100.0%)
TL-H7-88	KIA-43101 original	July 2011	MAMS-13125	3513 ± 18	1898–1766 (100.0%)
TL-H7-88	KIA-43101	July 2011	MAMS-13124	3508 ± 19	1892–1760 (100.0%)
TL-H7-88	KIA-43101-A	July 2011	MAMS-13123	3496 ± 18	1883–1756 (100.0%)
TL-H7-88	4th set of seeds	April 2011	URU-0558= URU-0559	3473 ± 35	1888–1730 (90.6%) 1721–1692 (9.4%)

Granary H2 of Tira del Lienzo

A large grain storage area was discovered next to the eastern enclosure wall of Tira del Lienzo. A first set of barley and wheat seeds was sent to Mannheim (MAMS-11820) in 2011 (Table 3). Later, a second set from the same context went to Kiel (KIA-44973) in order to check if former dating problems had been ruled out. Once again, Kiel's result was too old whereas Mannheim's matched the expected chronology based on archaeological grounds.

Table 3 ^{14}C dates of *Hordeum* sp. and *Triticum* sp. seeds from Tira del Lienzo Building 2.

Context	Sample	Report date	Lab nr	BP	cal BC 2σ
TL-H2-2	Second set of barley & wheat seeds	10/06/2013	KIA-44973	3760 ± 25	2282–2248 (11.4%) 2232–2217 (2.9%) 2215–2129 (76.4%) 2087–2048 (9.4%)
TL-H2-40	First set of barley & wheat seeds	05/01/2011	MAMS-11820	3587 ± 32	2032–1879 (99.3%) 1837–1830 (0.7%)

DATING PROBLEMS ELSEWHERE

The Bronze Age sites we have discussed up to now are very close to each other and shared similar ecological conditions in the past. In order to discard a potential environmental bias in ^{14}C results, we extended our sampling to other chronological periods and regions.

Las Paleras, close to the Castle of Alhama (Murcia), is a Medieval site located 15 km northeast of La Bastida and excavated by a team of archaeologists led by José Baños (City Council of Alhama de Murcia). A destruction layer found under the castle offered the opportunity to both analyze and date a remarkably well-preserved context of botanical remains. The chronology of that context had been established by the excavators between the 7th and the 10th centuries AD based on ceramic typology. In southeast Iberia, the accepted view is that the occupation of prominent elevations, such as the Castle of Alhama, started during the 5th century AD and became a key element of the Andalusian landscape from the 8th until the 13th century (e.g. Menasanch 2004).

A sample of charred barley seeds identified by H-P Stika was submitted for ^{14}C dating. The result obtained by Kiel was highly surprising (KIA-42808) (Table 4), since it would imply that hilltop settlements were already established during Late Roman times, and not as a consequence of the social and political instabilities following its collapse. Given the fact that such a conclusion would imply a paradigmatic shift in Late Roman and Medieval archaeology, it seemed much safer to have a second ^{14}C reference, especially considering the fact that no 3rd–4th century pottery was found in the sampled layer. For this reason, a new barley sample from the same context was sent to the laboratory of Mannheim. The result is 500–600 calendar years younger than Kiel's (MAMS-11860, Table 5), thus confirming the current view about the development of early Medieval fortified hilltop settlements. A new date provided by the Kiel laboratory in July 2013 is still 100 ^{14}C yr older than MAMS's and significantly different at 95% level (*T* test: 11.52; $\chi^2 = 3.84$; $df = 1$).

Table 4 Radiocarbon dates from the first settlement of Las Paleras (Alhama de Murcia).

Context	Sample	Report date	Lab nr	BP	cal AD 2σ
CCPAL-E10-6026	First set of barley seeds	17/08/2010	KIA-42808	1696 ± 20	258–283 (11.7%) 322–402 (88.3%)
CCPAL-E10-6026	Third set of barley seeds	04/10/2013	KIA-42808	1227 ± 20	694–703 (1.8%) 707–745 (25.9%) 764–880 (72.3%)
CCPAL-E10-6026	Second set of barley seeds	05/01/2011	MAMS-11860	1131 ± 20	782–786 (0.4%) 877–982 (99.6%)

The second series comes from a very different and distant geographical region: Bronze Age Menorca (Balearic Islands). Here, two funerary sites were excavated and sampled by our research team. The first, Es Forat de Ses Aritges, is a small rockshelter closed by a cyclopean wall and used as a collective burial precinct. A set of six human bone samples was analyzed in the 1990s by the KIK-IRPA laboratory in Brussels and dated at Utrecht, offering a period of use ranging from the beginning of the second half of the 1st millennium cal BCE (KIK-962/UtC-7856: 3170 ± 35 BP) to the transition between the 2nd and 1st millennium cal BCE (KIK-974/UtC-7852: 2875 ± 35 BP) (Lull et al. 1999). The second, Ses Arenes de Baix, is a megalithic monument formed by an elongated chamber covered by a complex cairn, which was also used as a collective funerary precinct. A series of seven dates from human bone provided by Brussels and Kiel laboratories in 2004 suggests a period of use covering the second and third quarters of the 2nd millennium cal BCE (KIK-3065/KIA-23150: 3390 ± 35 BP and KIK-3082/KIA-23403:3025 ± 25 BP as limit dates) (Gili et al. 2006; Micó 2005).

Three samples from Es Forat de ses Aritges and two from Ses Arenes de Baix were run at Kiel and results reported in August 2011 (Table 5). All of them tend to fall close to the upper limit of the series already dated and one is clearly an outlier (KIA-44844). Given that these dates seemed too old, a redating of the whole series of Minorcan samples was advised. The collagen extraction was undertaken at the KIK-IRPA laboratory in Brussels and the Kiel laboratory provided a new series of results in January 2012. As Table 5 shows, all the 2012 results on samples prepared in Brussels are significantly younger than those dated in 2011 by up to almost 500 calendar years (see KIA-46771). Moreover, the new results fit well with the series dated in the 1990s and with the general chronology of the Balearic Bronze Age, which is based on more than 400 ^{14}C dates (Micó 2005).

Table 5 Radiocarbon dates from Menorca (ARB: Ses Arenes de Baix; FA: Es Forat de ses Aritges; NR: not reported).

Context	Sample	Report date	Lab nr	Date BP	cal BC 2σ	Collagen yield (%)	C content of collagen (%)	C:N
ARB-A8	Human talus	Aug. 2011	KIA-44840	3408 ± 25	1762–1633 (100.0%)	4.4	47.8	NR
ARB-A8	Collagen graphitized of KIA-44840	Jan. 2012	KIA-46767	3030 ± 30	1395–1332 (27.7%) 1328–1206 (71.2%) 1203–1196 (0.9%) 1138–1135 (0.2%)	NR	NR	NR
ARB-A7	Human talus	Aug. 2011	KIA-44841	3410 ± 25	1767–1636 (100.0%)	9.4	46.6	NR
ARB-A7	Collagen graphitized of KIA-44841	Jan. 2012	KIA-46768	3385 ± 30	1747–1619 (100.0%)	NR	NR	NR
FA-19-10500	Human talus	Aug. 2011	KIA-44842	3100 ± 25	1427–1294 (100.0%)	9.5	46.9	NR
FA-19-10500	Collagen graphitized of KIA-44842	Jan. 2012	KIA-46769	2730 ± 30	928–813 (100.0%)	NR	NR	NR
FA-23-7944	Human talus	Aug. 2011	KIA-44843	3235 ± 30	1609–1579 (11.2%) 1563–1435 (88.8%)	16.2	46.7	NR
FA-23-7944	Collagen graphitized of KIA-44843	Jan. 2012	KIA-46770	3075 ± 25	1411–1270 (100.0%)	NR	NR	NR
FA-41-5275	Human talus	Aug. 2011	KIA-44844	3485 ± 25	1886–1743 (99.4%) 1707–1704 (0.6%)	10.2	48.7	NR
FA-41-5275	Collagen graphitized of KIA-44844	Jan. 2012	KIA-46771	3035 ± 35	1406–1206 (98.6%) 1204–1196 (0.9%) 1139–1135 (0.5%)	NR	NR	NR

DISCUSSION

The cases described so far suggest that a serious problem affected measurements obtained from the Leibniz laboratory. Other results from different Argaric Bronze Age series have also raised questions, although the differences between the measured results and expected dates are not as large as in the examples presented. On the other hand, there are also ^{14}C dates from Kiel that seem to be in agreement with the archaeological expectations. In November 2012, six previously dated samples were prepared and/or measured again by the Leibniz lab and a final series of dates was provided in July 2013. Six cases prove to be consistent, and at least in three of them (KIA-40019, -40020, -40021) the dating of a fragment of the same animal bone in Mannheim suggests that these results are correct (Table 6). KIA-42493 stands as an ambiguous example and the remaining six offer significant differences that require further discussion (Table 6): in five of them redating values point to a younger age, but the sixth is older.

Table 6 Original dates and repeats of Argaric Bronze Age samples from Kiel and Mannheim laboratories.

Lab nr and sample	Report date	BP	Test sample significance (95%) (χ^2 = 3.84 for all calculations; *df* = 1)[a]
KIA-39737 Basketry fragment	Oct. 2009	3727 ± 25	Same dates (*T* = 0.0072).
KIA-39737	April 2013	3730 ± 25	
KIA-40016 Human bone, collagen	Oct. 2009	3551 ± 23	Different dates (*T* = 7.0190). Redating: younger age.
KIA-45102	June 2013	3461 ± 25	
KIA-40017 Faunal bone, collagen	Oct. 2009	3647 ± 29	Different dates (*T* = 19.2627). Redating: older age.
KIA-40017	June 2013	3827 ± 29	
KIA-40018 Faunal bone, collagen	Oct. 2009	3755 ± 32	Same dates (*T* = 0.0420).
KIA-45103 Repeat of KIA40018	June 2013	3764 ± 30	
KIA-40019 Faunal bone, collagen	Oct. 2009	3561 ± 29	Same dates (comparison between extreme results KIA-45105 and MAMS-13492: *T* = 1.3950).
KIA-45105 Repeat of KIA40019	June 2013	3588 ± 26	
MAMS-13492 Repeat of KIA40019	Sept. 2011	3547 ± 23	
KIA-40020 Faunal bone, collagen	Oct. 2009	3640 ± 30	Same dates (comparison between extreme results KIA-40020 and KIA-45106: *T* = 1.5605).
KIA-45106 Repeat of KIA40020	June 2013	3693 ± 30	
MAMS-13493 Repeat of KIA40020	Sept. 2011	3678 ± 25	
KIA-40021 Faunal bone, collagen	Oct. 2009	3620 ± 30	Same dates (comparison between extreme values KIA-40021-repeat 2 and MAMS-13494: *T* = 3.5475), except measurement KIA-40021-repeat 1.
KIA-40021-repeat 1	July 2011	3365 ± (not provided)	
KIA-40021-repeat 2	Nov. 2012	3585 ± 30	
KIA-45107-repeat 3 of KIA-45107	Nov. 2012	3600 ± 25	
MAMS-13494-repeat 4 of KIA-45107	Sept. 2011	3669 ± 33	
KIA-40123 Human bone, collagen	Oct. 2009	3630 ± 22	Different dates (comparison between extreme results KIA-40123 and KIA-40123-repeat 2: *T* = 10.9107; also for KIA-40123 and KIA-40123-repeat 3: *T* = 5.0721). Redating: younger age. Same dates for KIA-40123 and KIA-40123-repeat 1 (*T* = 1.8063) and, also, for internal comparisons in the group KIA-40123-repeats 1, 2 and 3.
KIA-40123-repeat 1	Sept. 2011	3580 ± 30	
KIA-40123-repeat 2	Sept. 2011	3520 ± 25	
KIA-40123-repeat 3	Nov. 2012	3555 ± 25	
KIA-42492 Human bone, collagen	July 2010	3600 ± 25	Same dates (comparison between extreme results KIA-42492 and KIA-42492-repeat 3: *T* = 3.38).
KIA-42492-repeat 1	Sept. 2011	3645 ± 30	
KIA-42492-repeat 2	Nov. 2012	3625 ± 30	
KIA-42492-repeat 3	Nov. 2012	3665 ± 25	

Table 6 Original dates and repeats of Argaric Bronze Age samples from Kiel and Mannheim laboratories.

Lab nr and sample	Report date	BP	Test sample significance (95%) (χ^2 = 3.84 for all calculations; *df* = 1)[a]
KIA-42493 Human bone, collagen	July 2010	3765 ± 30	Different dates (comparison between extreme values KIA-42493-repeat 1 and repeat 2: *T* = 3.9054). Redating: younger age. Same dates (comparison between KIA-42493 and younger redating result: *T* = 1.4235).
KIA-42493-repeat 1	Sept. 2011	3795 ± 25	
KIA-42493-repeat 2	Nov. 2012	3710 ± 35	
KIA-42493-repeat 3	Nov. 2012	3750 ± 25	
KIA-42495 Human bone	July 2010	4154 ± 36	Different dates (*T* = 65.7076). Redating: younger age.
KIA-42495 (2013)	June 2013	3747 ± 35	
KIA-42496 Human bone, collagen	July 2010	3825 ± 30	Different dates (comparison between KIA-42496 and KIA-42496-repeat 4: *T* = 28.125, and with KIA-42496-repeat 3: *T* = 24.5). Redating: younger age.
KIA-42496-repeat 1	Sept. 2011	"much too old"	
KIA-42496-repeat 3	Nov. 2012	3615 ± 30	
KIA-42496-repeat 4	Nov. 2012	3600 ± 30	
KIA-43166 Human bone, collagen	Sept. 2010	4000 ± 25	Different dates (comparison between KIA-43166 and KIA-43166-repeat 4: *T* = 196.02, and with KIA-43166-repeat 3: *T* = 176.72). Redating: younger age.
KIA-43166-repeat 1	Sept 2011	3800 ± (not provided)	
KIA-43166.repeat 2	Sept 2011	3595 ± (not provided)	
KIA-43166-repeat 3	Nov. 2012	3530 ± 25	
KIA-43166-repeat 4	Nov. 2012	3505 ± 25	

[a]Calculations performed with CALIB 7.0.1 (Stuiver et al. 2015) using IntCal13 data (Reimer et al. 2013).

After having remeasured a large number of samples, the following become clear:

- Outliers have occurred irrespective to the kind of material dated, whether bone or seeds; i.e., the quality of one type of sampled material (e.g. collagen preservation) cannot be the only explanation for the deviations.
- Differences in Kiel results were independent of the site or the age of the samples.
- There seems to be no clear pattern in the magnitude of the deviations, nor in identifying which individual samples are affected by errors, apart from the fact that almost all wrong results are older than the remeasured ones.
- Samples repeated in 2013 are free of major problems, although two results (KIA-40017, KIA-42808) continue to show some type of "aging" effect and lay beyond the ranges provided by stratigraphic and typological criteria supported by ^{14}C series.

Older dates than expected may have different causes. One possibility is that anomalous results on human bone samples are the product of a marine reservoir effect, as a consequence of diets based on aquatic resources. However, the discrepant ^{14}C results include dates on seeds of terrestrial C_3 plants, and reservoir effects cannot explain the discrepancies in redated samples nor the differences between results from Kiel and those from other laboratories. Furthermore, stable isotope data from La Bastida itself (Table 7) show no marine signature and are similar to results on Bronze Age human populations from inner southern Iberia (Nájera et al. 2010)

Table 7 Stable isotope data from La Bastida (data provided by Matthias Hüls, Leibniz Labor). Samples were analyzed by IRMS in at least two independent centers on behalf of the AMS Leibniz Labor.

Results (‰)		Human La Bastida	Fauna La Bastida
$\delta^{15}N$	Range	10.00 / 12.71	8.15 / 9.55
	Mean	11.56 ± 0.29	8.94 ± 0.34
$\delta^{13}C$	Range	–18.96 / –17.21	–20.02 / –19.08
	Mean	–18.38 ± 0.18	–19.50 ± 0.22

CONCLUSIONS

The cases presented here from La Bastida, Tira del Lienzo, Las Paleras, Ses Arenes de Baix, and Es Forat de ses Aritges show that ^{14}C analysis performed by Kiel gave unexpected and non-reproducible results from 2009. The redating of several samples has confirmed that a number of those ^{14}C results are several hundred years too old. There is no obvious pattern in the magnitude of the observed deviations, nor in which samples are affected. The deviations were not caused by a marine reservoir effect among the human samples, and the presence of an old carbon contaminant during sample preparation or an AMS dating problem seems likely.

The dating problems discussed herein were found due to the very detailed stratigraphic and typological sequences available. In this study, unexpected results have been detected in samples from KIA-39260 (July 2009) to KIA-44844 (August 2011). Hence, it is our view that all organic samples prepared and measured in Kiel during this period may be affected, and some of the dates repeated in 2012 and 2013 may also be inconsistent.

Given the apparently random character of the anomalous ^{14}C dates, other past and current research projects may have been affected as well (Peška 2013; Friedrich et al. 2014). We have been collaborating intensively with the Leibniz Labor at Kiel ever since the first anomalies were detected in 2009. In order to check the first results and to search for patterns that might lead to identify the dating problem or problems, repeat dates were run at different laboratories on our behalf. New samples were provided whenever possible and repeat measurements were also performed on pretreated material and on new extractions of leftover aliquots from the same bone fragment or seed. We do appreciate Leibniz Labor's efforts to address all inconsistencies detected, particularly after a change in its direction, but an in-depth explanation of the possible causes has not yet been established. Nevertheless, the amount of information gathered over these years needs to be reported, especially since the problems detected might affect an unknown number of dates extending at least over 2009–2011. We do hope that a full diagnosis of the problems will be available soon.

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REFERENCES

Castro P, Chapman R, Gili S, Lull V, Micó R, Rihuete C, Risch R, Sanahuja ME. 1993–1994. Tiempos sociales de los contextos funerarios argáricos. *Anales de Prehistoria y Arqueología de la Universidad de Murcia* 9–10:77–105.

Castro P, Chapman RW, Colomer E, Gili S, González Marcén P, Lull V, Micó R, Montón S, Rihuete C, Risch R, Ruiz M, Sanahuja ME, Tenas M, Van Strydonck M. 1995. La serie radiocarbónica de Gatas (Turre, Almería). Diacronía y fasificación del depósito arqueológico. *Anuario Arqueológico de Andalucía* 1992:27–36.

Castro P, Chapman R, Gili S, Lull V, Micó R, Rihuete C, Risch R, Sanahuja ME. 1999. *Proyecto Gatas 2. La dinámica arqueoecológica de la ocupación prehistórica*. Seville: Consejería de Cultura de la Junta de Andalucía.

Friedrich S, Schlenker B, Schunke T. 2014. Radiometrische Mehrfachbeprobungen an archäologischen Befunden aus Salzmünde, Saalekreis: Ein Diskussionsbeitrag zur Interpretation von ^{14}C-Daten. *Archäologie in Sachsen-Anhalt* 7/14:24–31.

Gili S, Lull V, Micó R, Rihuete C, Risch R. 2006. An island decides: megalithic burial rites on Menorca. *Antiquity* 80(310):829–42.

González Marcén P, Lull V, Risch R. 1992. *Arqueología de Europa 2250–1200 A.C*. Madrid: Síntesis.

Lull V. 2000. Argaric society: death at home. *Antiquity* 74(285):581–90.

Lull V, Micó R, Rihuete C, Risch R. 1999. *La Cova des Càrritx y la Cova des Mussol: Ideología y Sociedad en la Prehistoria de Menorca*. Barcelona: Consell Insular de Menorca, Ajuntament de Ciutadella y Fundació Rubió Tudurí Andrómaco.

Lull V, Micó R, Rihuete C, Risch R. 2005. Property relations in the Bronze Age of southwestern Europe: an archaeological analysis of infant burials from El Argar (Almeria, Spain). *Proceedings of the Prehistoric Society* 71:247–68.

Lull V, Micó R, Rihuete C, Risch R. 2010. Límites históricos y limitaciones del conocimiento arqueológico: la transición entre los grupos arqueológicos de Los Millares y El Argar. In: Bueno P, Gilman A, Martín Morales C, Sánchez-Palencia FJ, editors. *Arqueología, sociedad, territorio y paisaje. Estudios sobre Prehistoria reciente, Protohistoria y transición al mundo romano. Homenaje a Mª Dolores Fernández-Posse*. Madrid: Bibliotheca Praehistorica Hispana XXVIII, Consejo Superior de Investigaciones Científicas. p 75–94.

Lull V, Micó R, Rihuete C, Risch R. 2011. El Argar and the beginning of class society in the western Mediterranean. In: Müller J, Hansen S, editors. *Sozialarchäologische Perspektiven prähistorischer Gesellschaften: die Entstehung von sozialer Ungleichhheit*. Berlin: Deutsches Archäologisches Institut, Col. Archäologie in Eurasien 24. p 381–414.

Lull V, Micó R, Rihuete C, Risch R. 2013. Political collapse and social change at the end of El Argar. In: Meller H, Bertemes F, Bork H-R, Risch R, editors. *1600 – Cultural Change in the Shadow of the Thera Eruption?* Halle: Landesmuseum für Vorgeschichte. p 283–302. In German.

Lull V, Micó R, Rihuete C, Risch R. 2014. The La Bastida fortification system: new light and new questions on the Early Bronze Age societies in the Western Mediterranean. *Antiquity* 88(340):395-410.

Menasanch M. 2004. *Secuencias de cambio social en una región mediterránea: análisis arqueológico de la depresión de Vera (Almería) entre los siglos V y XI*. British Archaeological Reports, International Series 1132. Oxford: Archaeopress.

Micó R. 2005. *Cronología absoluta y periodización de la Prehistoria de las Islas Baleares*. British Archaeological Reports, International Series 1373. Oxford: Archaeopress.

Nájera T, Molina F, Jiménez-Brobeil S, Sánchez Romero M, Al-Oumaoui I, Aranda G, Delgado-Huertas A, Laffranchi Z. 2010. La población infantil de la Motilla del Azuer: un estudio bioarqueológico. *Complutum* 21(2):69–102.

Peška J. 2013. Das Besiedlungsbild in der Blütezeit der Frühbronzezeit im Gebiet des mittleren Donauraumes. In: Meller H, Bertemes F, Bork H-R, Risch R, editors. *1600 – Cultural Change in the Shadow of the Thera Eruption?* Halle: Landesmuseum für Vorgeschichte. p 387–409. In German.

Reimer PJ, Bard E, Bayliss A, Beck JW, Blackwell PG, Bronk Ramsey C, Buck CE, Cheng H, Edwards RL, Friedrich M, Grootes PM, Guilderson TP, Haflidason H, Hajdas I, Hatté C, Heaton TJ, Hoffmann DL, Hogg AG, Hughen KA, Kaiser KF, Kromer B, Manning SW, Niu M, Reimer RW, Richards DA, Scott EM, Southon JR, Staff RA, Turney CSM, van der Plicht J. 2013. IntCal13 and Marine13 radiocarbon age calibration curves 0–50,000 years cal BP. *Radiocarbon* 55(4):1869–87.

Renfrew C. 1973. *Before Civilization*. London: Jonathan Cape.

Siret L, Siret H. 1887. Les Premières Âges du Métal dans le Sud-est de l'Espagne. Antwerpen (1890 edition in Spanish at Barcelona. Available at http://www.arqueomurcia.com/index.php?a=pu_libro_siret&PHPSESSID=1f79957ee1a9b0501984a34ee3ccb604.

Stuiver M, Reimer P, Reimer R. 2015. CALIB Radiocarbon Calibration. URL: http://calib.qub.ac.uk/calib/.