^{14}C DATING OF HUMIC ACIDS FROM BRONZE AND IRON AGE PLANT REMAINS FROM THE EASTERN MEDITERRANEAN

E M Wild[1,2] • P Steier[1] • P Fischer[3] • F Höflmayer[4]

ABSTRACT. Radiocarbon dating of plant remains is often difficult due to the complete dissolution of the samples in the alkaline step of the ABA pretreatment. At the VERA laboratory, this problem was encountered frequently when numerous Bronze and Early Iron Age samples from the eastern Mediterranean were dated in the course of the special research program SCIEM2000 and in other collaborations with archaeologists focused on that area and time period. For these samples, only a ^{14}C age determination of the humic acid fraction was possible. Humic acids from archaeological samples are always assessed as a second-choice material for ^{14}C dating. It is assumed that the ^{14}C ages may be affected by the presence of humic acids originating from other (younger) organic material, e.g. from soil horizons located above a sample. Therefore, when humic acids are dated a verification of the dates is crucial. To address this basic requirement, we started some time ago to date both fractions of charred seeds, wood, and charcoal samples whenever available, i.e. the residue after the ABA treatment and the humic acids extracted from the samples in the alkaline step. The results of this comparison showed that for the investigated eastern Mediterranean archaeological sites, 50 (out of 52) humic acid dates were in agreement with the ^{14}C dates of the respective ABA-treated samples. Statistical analysis of the age differences leads to the conclusion that the extracted humic acids originated from the samples themselves or from contemporaneous material and were not appreciably contaminated by extraneous material of different age.

INTRODUCTION

In the course of the special research program “The Synchronization of Civilizations in the Eastern Mediterranean in the 2nd Millennium BC” (short title: SCIEM 2000), ~500 samples from the 2nd millennium BC were dated at the VERA laboratory. The samples were mainly plant remains that originated from ~33 Bronze Age sites in the Middle East. In addition, further samples from this region and time period were dated in other collaborations with archaeologists working in this area.

A careful selection of the samples used for ^{14}C dating is crucial for accurate dating. In order to avoid the risk of an unknown time offset between the event to be dated and the ^{14}C date of the sample, the samples should originate from an unambiguous find context. An ideal, fairly rare, find situation is exemplified by short-lived plant remains found in a (sealed) jar on a floor that can be dated in relative terms according to archaeological criteria. Also, bones excavated at archaeological sites would fulfill in most instances the above criterion for the ^{14}C sample selection. But due to an unknown preservation state of the bone collagen, it is not guaranteed *a priori* that ^{14}C dating will be successful. Further, age offsets of some years caused by variable collagen turnover rates that depend on the biological age of the dated individual could be relevant for high-precision dating. For example, Wild et al. (2000) detected a time lag of ~30 yr between the time of death and the ^{14}C age of long bones from 85-yr-old individuals from the ^{14}C bomb-peak era. This effect should be negligible for normal archaeological dating as individuals in ancient times seldom reached such a long lifespan. Apart from that, ^{14}C dates of bones from omnivores and carnivores may also exhibit a considerable time offset caused by riverine and marine components in the paleodiet, which may require complex corrections. All offset problems in dating bones can be omitted when bones from domesticated, short-lived, herbivorous animals feeding on a terrestrial diet, e.g. sheep, are used.

[1]University of Vienna, Vienna Environmental Research Accelerator (VERA), Faculty of Physics, Isotope Research and Nuclear Physics, Währinger Straße 17, A-1090 Vienna, Austria.
[2]Corresponding author. Email: Eva.Maria.Wild@univie.ac.at.
[3]Cypriote and Near Eastern Archaeology, Department of Historical Studies, University of Gothenburg, Sweden.
[4]German Archaeological Institute (DAI), Orient Department, Podbielskiallee 69-71, 14195 Berlin, Germany.

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Nevertheless, for ^{14}C dating, terrestrial short-lived plant remains like seeds or twigs seem to be the best choice, because plant remains are frequently recovered at archaeological sites. Wood from trunks (e.g. beams) or branches may also be suitable for ^{14}C dating when it is possible to estimate the time distance between the dated tree rings and the outermost tree ring, or, ideally, when the outermost tree ring or the bark is present in the sample (e.g. Friedrich et al. 2006). However, one has to be aware that a time offset may also be introduced into the chronology by the reuse of wood (e.g. beams).

The age of the finds may reveal information about the history of the site, like the time period when a place was in use. In context with other information, like the assemblage of plant and animal remains found at the site, insights into the dietary habits at the investigated time period may be gained. Particular plant species may also indicate certain climate conditions and thus by ^{14}C dating periods of climate change may be detected.

In most instances, preserved plant remains are charred due to fire exposure in ancient times. Charring can be caused by destructive events such as widespread conflagrations, but also simply by the usage of wood as firewood, which may leave charcoals at an excavation site. Usually, these sample types are chemically pretreated with the well-known acid-base-acid (ABA) method in order to remove possible contaminants like carbonates and fulvic and humic acids.

Unfortunately, from several of the sites that were investigated in the aforementioned studies a number of samples dissolved completely during the alkaline step. For example, from the archaeological site Tell Abu al-Kharaz, Jordan Valley, 26 out of 41 samples did not survive the alkaline treatment. In these cases, only the humic acids could be used for ^{14}C dating.

It is generally assumed that part of the humic acids present in a sample were borne by groundwater and may originate from other (younger) organic material, e.g. from soil horizons located above the sample. These humic acids could show divergent ^{14}C ages compared to authigenic humic acids from the sample (e.g. Olson and Broecker 1958; Alon et al. 2002) and may constitute a potential contaminant. Thus, humic acids from archaeological samples are always considered as second-choice dating material and a verification of the dating results is indispensable. In his textbook, Taylor (1987) reports the humic acid problem, to which much attention was given already in the early history of ^{14}C dating. According to Taylor and references therein, no difference between the extracted humic acids and total wood or cellulose was detected for a large percentage of wood samples. However, he mentions also studies that found in a small number of samples divergent ages for Holocene charcoal and the thereof extracted humic acids. It is assumed that a detectable difference might be found when the samples are overlaid by layers rich in organic material like e.g. peat. Due to this ambiguity, it appears to be mandatory to verify humic acids dates when they are used in archaeological studies.

Frequently, a way to control the ^{14}C method is offered by the ^{14}C method itself. For example, ^{14}C data of different sample fractions (corresponding to different steps in the pretreatment) may reveal whether any contamination is removed in a certain pretreatment step (see e.g. Taylor 1987). We used a similar approach to address the question if the humic acid ^{14}C ages of our study may be assessed as reliable. We started to date both the residues of seeds, charcoals, wood etc. after the full ABA treatment and the humic acids extracted from the samples in the alkaline step, whenever both fractions were available. This strategy was applied for those eastern Mediterranean Bronze and Iron Age sites from which samples tended to dissolve completely during pretreatment. By the end of June 2012, we had collected 50 data pairs of samples from 10 sites from this region and time period, with 4 of the investigated sites located in or close to the Jordan Valley (see Figure 1), and 1 site in Iran.

Figure 1 Map of the eastern Mediterranean from Google Earth with archaeological sites indicated where a ^{14}C age comparison of the ABA-treated sample and the extracted humic acids could be carried out. Four sites are located in or close to the Jordan Valley, which is displayed in an enlarged view in the inset.

MATERIAL AND METHODS

The plant remains ^{14}C dated in our comparative study were pretreated with the standard ABA procedure, which is used for archaeological samples at the VERA laboratory. In this procedure, the sample is first soaked in hot (60 °C) 1M HCl for 1 hr. After washing with bidistilled water (H_2O_{bidist}) to almost neutral pH, the sample is treated with 0.1M NaOH solution, again at 60 °C. During this step, the NaOH solution is repeatedly decanted and renewed until the solution remains colorless. Thus, the duration of this step is variable and depends on the amount of humic acids present in the sample. After a careful rinsing of the sample with bidistilled water, the sample is treated again for 1 hr with 1M HCl at 60 °C. The end of the procedure comprises a washing step with H_2O_{bidist}. The dried samples are further processed as described in Wild et al. (2008).

For each sample, the alkaline solutions from the ABA treatment, which contained the dissolved humic acids, were collected and combined. The humic acids were precipitated by acidifying the resulting solution with 6M HCl. After the precipitate was allowed to settle overnight, the sample was centrifuged, the supernatant discarded, and the residue washed several times with H_2O_{bidist} to about pH 3 at which the dissolution of the humic acids starts again. The pH of the sample solution was then lowered with some drops of 1M HCl and the solution was centrifuged. The supernatant was discarded and the residue was dried. The dried precipitate constitutes the material denoted by "humic acid fraction" in this comparison. The NaOH solution was simply decanted or sucked off from the sample residue, which is the usual practice at the VERA laboratory when applying the ABA method. Therefore, it could not be excluded that the humic acid fractions contained also some particles from the solid sample residue of the alkaline step. To check if a considerable amount of particulate matter is present in the solution, 5 samples (VERA-4024, VERA-5301, VERA-5436, VERA-5081, and VERA-5550) were processed for a second time, and the collected and combined alkaline solutions of these samples were filtered through a 25-µm pore size Teflon® filter. The inspection of the filters revealed that negligible amounts of solid particles retained by this pore size were present in the tested solutions. The ^{14}C data of the second determination were concordant with the first dating results of both sample fractions (see below).

RESULTS AND DISCUSSION

The ^{14}C data of the ABA-humic acids comparison are listed in Table 1. This comprehensive data set allows for a detailed statistical analysis. Together with the ^{14}C results, the information about sample material and provenience (archaeological site) is given. The suffix HS in the sample's laboratory number is used for the humic acid fraction of the sample. Further, the age difference between the ABA-treated sample and the related humic acids is given in the table as well as the 1σ uncertainty of the difference. Table 1 also shows the results of the reproducibility test for the samples VERA-4024, VERA-5301, and VERA-5436. In these tests, the humic acid fractions were filtered as described above. These data pairs are indicated as repeated in the respective row below the data obtained for the same samples when applying our standard ABA procedure. As already expected due to the negligible amounts of particles found on the filters, the humic acid ages yielded by both preparation methods were in excellent agreement. The samples VERA-5081 and VERA-5550 were also included in this reproducibility test, but both samples dissolved completely. Therefore, only the humic acid fraction could be dated. The determined ^{14}C ages also agreed with the results of the first dating run (VERA-5550HS: 2913 ± 34 BP compared to VERA-5550HS_2: 2887 ± 34 BP and VERA-5081HS: 2723 ± 40 BP compared to VERA-5081HS_2: 2767 ± 40 BP).

Large age differences between the ABA-treated sample and the extracted humic acids were determined for 2 specimens. These data (listed in the last 2 rows of Table 1) were treated as outliers and were not considered in the statistical analysis. One of the outlier samples consisted of fragmented olive stones (VERA-5577) from the archaeological site Tell el-Burak. The ABA-treated sample yielded a ^{14}C age of 2967 ± 28 BP whereas the associated humic acids were dated to 3168 ± 30 BP. The difference between both dates was 201 ± 41 ^{14}C yr. In a second independent ^{14}C dating of this sample and the corresponding humic acids, a perfect agreement of the data pair was determined with a difference of 0 ± 47 ^{14}C yr. These data (VERA-5577_2 and VERA-5577HS_2) were incorporated into the analyzed data set.

The other sample, which was excluded from the analysis, was a charcoal (VERA-3996) from the Lebanese site Tell Arqa. The sample itself was dated to ~11,000 BP whereas the humic acid date was 3616 ± 34 BP (VERA-3996HS). The sample was assigned to the Bronze Age period; thus, the 11,000 BP date must be assessed as not reliable. The corresponding humic acid age falls into the Bronze Age period. Unfortunately, in a second dating attempt the sample dissolved completely and only the humic acid age of 3573 ± 34 BP (VERA-3996HS_2) could be determined, which is concordant with the humic acid date obtained earlier.

From the data set comprising 50 ^{14}C age pairs (including the dates from the reproducibility test, but without the 2 outliers), a mean value of the age differences of –1.5 ^{14}C yr with a 1σ internal uncertainty of 7.0 ^{14}C yr is calculated. This value is compatible with zero within limits of error and thus leads to the assumption that the humic acids may originate from the sample itself or from contemporaneous material. For further verification, a χ^2 test was performed. This implies the assumption that the age differences are normally distributed as one would expect when calculating differences of independent ages of 2 coeval samples. It should be noted here that the ^{14}C ages themselves are not normally distributed, but for ^{14}C ages younger than 30 ka (Ward and Wilson 1978) a normal distribution can be assumed. A χ^2 value of 59 was determined for the 50 ^{14}C age differences, which is smaller than the critical value of 66.3 for 49 degrees of freedom and a 5% significance level. An additional corroboration for the pure statistical origin of the age differences may be deduced from the differences expressed in units of their 1σ uncertainty (last column in Table 1).

Table 1 ^{14}C ages of the fully ABA-treated samples and the extracted humic acids and the thereof calculated differences are given together with the sample location and the material type. In the last column the values of the differences divided by their 1σ uncertainty are given which corresponds to the expression of the differences in units of their 1σ values.

Location	Sample material	VERA # (ABA)	^{14}C age[a] (BP)	VERA # (humic acids)	^{14}C age[a] (BP)	(ABA–HS)[a,b] [^{14}C yr]	$\frac{(ABA-HS)}{\lvert 1\sigma \rvert}$
Tell Zira^ca	Small charcoal pieces	5438	3570 ± 36	5438HS	3437 ± 37	133 ± 52	2.59
Tell Zira^ca	Small charcoal pieces	5434	3640 ± 38	5434HS	3557 ± 38	83 ± 53	1.55
Tell Zira^ca	Charcoal	5435	3548 ± 30	5435HS	3537 ± 31	11 ± 43	0.26
Tell Zira^ca	Charcoal	5436	3491 ± 37	5436HS	3528 ± 36	–37 ± 51	–0.72
Tell Zira^ca (repeated)	Charcoal	5436_2	3438 ± 36	5436HS_2	3503 ± 34	–65 ± 50	–1.31
Tell Zira^ca	Charcoal	5437	3532 ± 35	5437HS	3549 ± 36	–18 ± 50	–0.35
Pella	Cereals	5304	3018 ± 37	5304HS	3027 ± 38	–9 ± 53	–0.17
Pella	Cereals	5313	2747 ± 38	5313HS	2798 ± 37	–51 ± 53	–0.96
Pella	Cereals	5311	2940 ± 37	5311HS	2926 ± 37	14 ± 52	0.26
Pella	Olive stone	5301	2928 ± 34	5301HS	2908 ± 35	20 ± 49	0.40
Pella (repeated)	Olive stone	5301_2	2967 ± 33	5301HS_2	2913 ± 35	54 ± 48	1.12
Atchana	Seeds	5331	3585 ± 40	5331HS	3544 ± 35	40 ± 53	0.75
Atchana	Olive pit	5340	3500 ± 36	5340HS	3477 ± 38	23 ± 52	0.44
Batrawy	Charcoal	5425	4179 ± 38	5425HS	4182 ± 51	–4 ± 64	–0.05
Tell Abu al-Kharaz	Seeds from floor	5273	3184 ± 27	5273HS	3173 ± 26	11 ± 37	0.31
Tell Abu al-Kharaz	Ash from oven	5277	2563 ± 21	5277HS	2576 ± 26	–13 ± 34	–0.40
Tell Abu al-Kharaz	Charcoal, twig	5279	2504 ± 23	5279HS	2552 ± 26	–47 ± 35	–1.36
Tell Abu al-Kharaz	Charcoal	5467	2511 ± 35	5467HS	2532 ± 34	–21 ± 48	–0.43
Tell Abu al-Kharaz	Charcoal/twigs	5545	2853 ± 36	5545HS	2953 ± 36	–100 ± 51	–1.96
Tell Abu al-Kharaz	Seeds, millet	5550	2987 ± 38	5550HS	2913 ± 34	74 ± 51	1.45
Tell Abu al-Kharaz	Chickpeas	5546	2921 ± 36	5546HS	2921 ± 34	–1 ± 49	–0.01
Tell Abu al-Kharaz	Grain (barley?)	5548	2938 ± 35	5548HS	2895 ± 36	43 ± 50	0.85
Tell Abu al-Kharaz	Charcoal of twigs	5081	2709 ± 36	5081HS	2723 ± 40	–14 ± 54	–0.26
Tell Abu al-Kharaz	Charcoal of twigs	5070	2450 ± 36	5070HS	2470 ± 38	–19 ± 52	–0.37
Tell Abu al-Kharaz	Charcoal of twigs	5073	2567 ± 38	5073HS	2593 ± 39	–26 ± 55	–0.48
Tell Abu al-Kharaz	Charcoal of twigs	5069	2466 ± 39	5069HS	2476 ± 38	–11 ± 54	–0.20
Rehov	Charred seeds	3221	2727 ± 37	3221HS	2725 ± 36	2 ± 51	0.05
Rehov	Charred seeds	3222	2737 ± 33	3222HS	2713 ± 26	24 ± 42	0.57
Santorini	Wood, olive branch	5614	3282 ± 21	5614HS	3359 ± 33	–78 ± 39	–2.00
Santorini	Wood, olive branch	5615	3280 ± 24	5615HS	3321 ± 24	–40 ± 34	–1.20

Table 1 ^{14}C ages of the fully ABA-treated samples and the extracted humic acids and the thereof calculated differences are given together with the sample location and the material type. In the last column the values of the differences divided by their 1σ uncertainty are given which corresponds to the expression of the differences in units of their 1σ values. *(Continued)*

Location	Sample material	VERA # (ABA)	^{14}C age[a] (BP)	VERA # (humic acids)	^{14}C age[a] (BP)	(ABA–HS)[a,b] [^{14}C yr]	$\frac{(ABA-HS)}{\lvert 1\sigma \rvert}$
Santorini	Wood, olive branch	5620	3277 ± 25	5620HS	3345 ± 24	–68 ± 34	–1.99
Santorini	Wood, olive branch	5610	3399 ± 25	5610HS	3342 ± 26	57 ± 37	1.56
Santorini	Wood, olive branch	5083	3270 ± 36	5083HS	3326 ± 77	–56 ± 85	–0.66
Santorini	Wood, olive branch	5082	3332 ± 38	5082HS	3369 ± 36	–37 ± 52	–0.71
Santorini	Root	5084	3354 ± 32	5084HS	3368 ± 34	–14 ± 47	–0.29
Tepe Sialk	Charcoal	3462	4825 ± 31	3462HS	4770 ± 32	55 ± 44	1.24
Tell el-Burak	Fragment of olive stone	5579	3567 ± 32	5579HS	3512 ± 32	55 ± 45	1.22
Tell el-Burak	Fragments of *Vicia ervilia*	5575	3161 ± 39	5575HS	3140 ± 39	21 ± 55	0.38
Tell el-Burak	Fragment of olive stone	5577_2	2926 ± 34	5577HS_2	2926 ± 33	0 ± 47	0.00
Tell Arqa	Seeds	4010	3830 ± 34	4010HS	3874 ± 33	–45 ± 47	–0.96
Tell Arqa	Charcoal	4016	4245 ± 39	4016HS	4110 ± 31	135 ± 50	2.70
Tell Arqa	Seeds	4019	4095 ± 32	4019HS	4083 ± 32	13 ± 45	0.28
Tell Arqa	Seeds	4020	4043 ± 38	4020HS	4056 ± 32	–14 ± 50	–0.27
Tell Arqa	Seeds	4024	4140 ± 37	4024HS	4209 ± 32	–69 ± 49	–1.41
Tell Arqa repeated	Seeds	4024_2	4170 ± 37	4024HS_2	4151 ± 33	19 ± 50	0.38
Tell Arqa	Olive stone	5117	4049 ± 38	5117HS	4049 ± 34	–1 ± 51	–0.01
Tell Arqa	Broken olive stones	5122	4089 ± 35	5122HS	4237 ± 39	–148 ± 52	–2.83
Porsuk	Wood	5750A_2	3336 ± 18	5750HS	3345 ± 39	–9 ± 43	–0.21
Porsuk	Wood	5751A_2	3363 ± 20	5751HS	3354 ± 48	9 ± 52	0.17
Porsuk	Wood	5752A_2	3390 ± 23	5752HS	3348 ± 53	42 ± 58	0.73
				Average age difference ±1σ		–1.5 ± 7.0	
Tell Arqa	Charcoal	3996	11,052 ± 46	3996HS	3616 ± 34	7436 ± 57	130.87
Tell el-Burak	Fragment of olive stone	5577	2967 ± 28	5577HS	3168 ± 30	–201 ± 41	–4.87

[a] 1σ uncertainty.
[b] Calculated from unrounded data.

In Figure 2, the determined values are displayed versus the sample numbers, which are arbitrarily assigned. It is evident that the differences (given in 1σ units) scatter between +3σ and –3σ. Inspecting the scatter of the values in more detail shows that 34 data (corresponding to 68% of the analyzed dates) fall in the ±1σ range, 13 data (26%) in the positive and negative 1σ to 2σ ranges and 3 (6%) in the positive and negative 2σ to 3σ ranges. The insert in Figure 2 displays this distribution graphically, which is obviously similar to a normal distribution, where we expect that 68.3% of the data fall in the ±1σ range, 95.4% in the ±2σ range, and 99.7% in the ±3σ range.

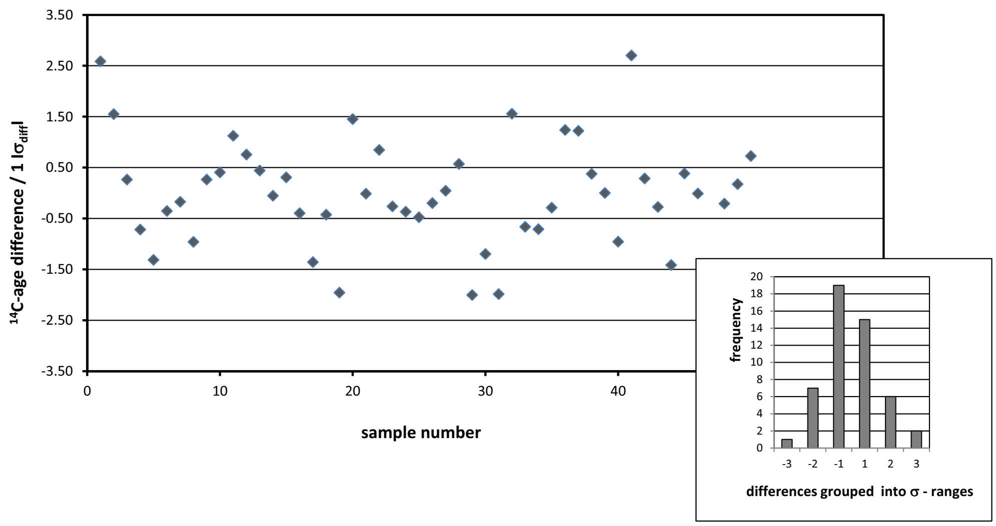

Figure 2 ^{14}C age differences of the ABA-treated sample and the humic acids expressed in 1σ units versus the arbitrary sample number. The insert shows the frequency distribution of these differences grouped into the indicated σ ranges.

In Figure 3, the differences (again in units of their 1σ values) are ordered by increasing values versus the fraction of samples yielding equal or smaller values. For comparison, the cumulative normal distribution is also displayed in this figure. The theoretical curve fits very well to the empirically determined data. This supports again the assumption that the age differences are normally distributed and is together with the zero compatible mean value a further indicator that the data pairs are independent dates of coeval samples. Bronk Ramsey et al. (2010) used a similar approach to show that the ^{14}C ages determined for known-age tree-ring samples from the USA and Ireland are in agreement with the IntCal04 data (Reimer et al. 2004) whereas a regional offset of 19 ± 5 yr was found for samples from Egypt.

Our analysis of the ^{14}C age differences of the ABA-treated samples and the respective humic acids supports the assumption that the humic acids extracted from the samples originate from the plant remains themselves. Another explanation would be that the humic acids come from contemporaneous organic material other than the sample. This seems to be more unlikely as it would imply that the samples have been embedded in coeval soil horizons rich in humic acids, which would have been detected in the excavated archaeological layers. The good agreement of the data pairs is also found in the calibrated dates. As an example, in Figure 4, the OxCal plot for part of the samples originating from the mid-2nd millennium BC is displayed. The probability distributions of corresponding dates are shown in the same colors (gray or black) in order to demonstrate their excellent concordance.

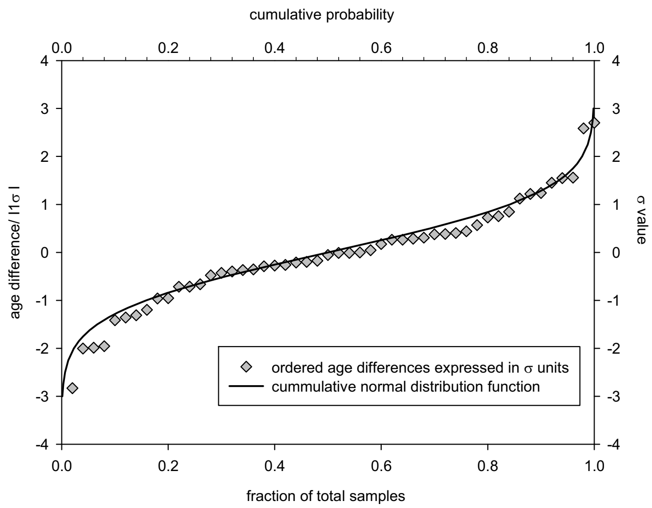

Figure 3 ^{14}C age differences (in 1σ units) ordered by increasing values versus the fraction of samples yielding smaller or equal values. The solid line displays the theoretical cumulative normal distribution function.

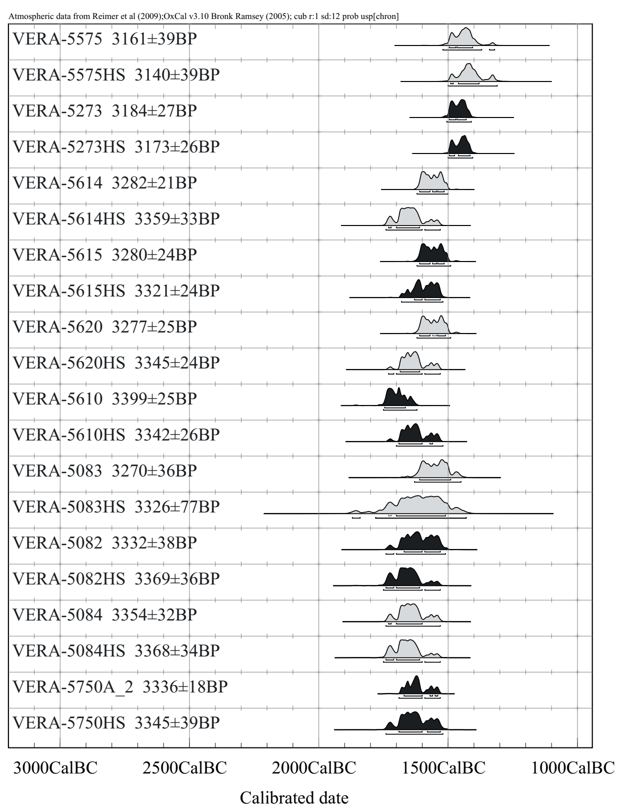

Figure 4 OxCal plot (Bronk Ramsey 2001) of calibrated ages of the data pairs for the mid-2nd millennium BC. Probability distributions of data pairs are equally colored.

A concordance of ^{14}C results from fully ABA-treated samples and the respective humic acids was also found by the Oxford ^{14}C laboratory for 4 short-lived samples from the Egyptian Bronze Age site Tell el Daba (Kutschera et al. 2012). In accord with our conclusion, this finding is interpreted as an indication for the absence of contamination of the samples by humic acids from the environment.

CONCLUSION

The comparison of ^{14}C dates determined for ~50 ABA-treated samples and the thereof extracted humic acids showed that no statistical significant differences could be detected for the investigated eastern Mediterranean Bronze and Iron Age sites. This finding supports the conclusion that the humic acids originate from the plant material itself. Further, for the investigated sites and time period, the results of our study corroborate the reliability of ^{14}C ages of humic acids for cases when the samples completely dissolved during the ABA treatment. According to the results of our comparison, it seems worthwhile to date humic acids when samples do not survive the full ABA method, but it is inevitable to verify the reliability of the humic acid dates.

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