

ANTHROPOLOGY

Radiocarbon chronology of Manot Cave, Israel and Upper Paleolithic dispersals

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The timing of archeological industries in the Levant is central for understanding the spread of modern humans with Upper Paleolithic traditions. We report a high-resolution radiocarbon chronology for Early Upper Paleolithic industries (Early Ahmarian and Levantine Aurignacian) from the newly excavated site of Manot Cave, Israel. The dates confirm that the Early Ahmarian industry was present by 46,000 calibrated years before the present (cal BP), and the Levantine Aurignacian occurred at least between 38,000 and 34,000 cal BP. This timing is consistent with proposed migrations or technological diffusions between the Near East and Europe. Specifically, the Ahmarian could have led to the development of the Protoaurignacian in Europe, and the Aurignacian in Europe could have spread back to the Near East as the Levantine Aurignacian.

INTRODUCTION

Although distinct populations of modern humans likely dispersed to Eurasia at different times, the group that successfully colonized Europe after ~50,000 years ago was associated with a suite of behavioral and technological innovations, broadly referred to as the Upper Paleolithic (1). Along the dispersal route, the Levant is a key region for understanding the origins and spread of Upper Paleolithic traditions and peoples (2, 3). During the temporal phase known as the Early Upper Paleolithic (EUP), there were two archeological cultures in the Levant attributed to modern humans, the Early Ahmarian and the Levantine Aurignacian (text S1) (4, 5). It has been proposed that the Early Ahmarian led to the Protoaurignacian in Europe (6) and that makers of the Aurignacian in Europe backmigrated to the Near East, producing the Levantine Aurignacian (7).

The likelihood of these hypotheses depends on the similarities between industries and their relative ages. This study focuses on the latter, adding new data to the Levantine EUP chronology to test pro-

posed relations between industries. Although chronology alone cannot prove these hypotheses, it can be consistent with them or it can refute them, if shown that a proposed descendant industry actually predated its alleged antecedent.

However, thus far, this test has been inconclusive for Levantine EUP industries because the regional chronology is not firmly established. Dates for Ahmarian assemblages differ by several millennia between sites, clustering into early appearance dates [~46 thousand calibrated years before the present (ka cal BP)] that allow for an Ahmarian-Protoaurignacian development (8) and late appearance dates (~40 ka cal BP) that refute this hypothesis (3, 9). Regarding the Levantine Aurignacian, the available dates are not precise enough to evaluate whether the industry could have derived from the Aurignacian in Europe (3). As it stands, the regional chronology is difficult to resolve because it includes chronometric samples collected from unclear contexts of old excavations and dates produced through analytical procedures that failed to demonstrate the removal of contaminants.

Here, we present a research program for radiocarbon dating, designed to ameliorate these issues through (i) the use of geochemical methods to characterize samples and their archeological contexts and (ii) experimentally determined pretreatment procedures, customized for the dated materials. The methodology is applied to newly excavated EUP assemblages from Manot Cave, Israel, resulting in a high-resolution chronology of 47 radiocarbon dates. Integrating the radiocarbon dates, geochronological analyses, and artifact analysis, the following minimum ranges are suggested for cultural phases at Manot: Early Ahmarian, from 46 to 42 ka cal BP; Levantine Aurignacian, from 38 to 34 ka cal BP; a post-Levantine Aurignacian industry, from 34 to 33 ka cal BP. These reliable dates provide a foundation for the Levantine EUP chronology, which is consistent with the hypotheses that the Ahmarian led to the Protoaurignacian in Europe and that the Aurignacian in Europe gave rise to the Levantine Aurignacian through the movement of people or ideas.

RESULTS

Site description and cultural-chronometric sequence

Excavated from 2010 to 2017, Manot is an active karstic cave in north-west Israel, about 10 km from the present-day Mediterranean (Fig. 1)

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(10). A modern human calvaria, Manot 1, was recovered from the cave surface but was not associated with artifacts. It was uranium-thorium (U-Th)-dated to a minimum age of 54.7 ± 5.5 ka (11, 12). The site's archaeological finds constitute a stratified sequence of EUP industries (Table 1, text S2, figs. S1 and S4, and tables S1 and S2). The uppermost cultural phase, located by the cave entrance (area E), is classified as post-Levantine Aurignacian and contains endscrapers, burins, Dufour bladelets, and partially retouched twisted bladelets. It is underlain by a Levantine Aurignacian assemblage, characterized by endscrapers (carinated, nosed, and flat), carinated burins, blades with Aurignacian retouch, and Dufour bladelets. The Aurignacian finds also include bone awls, antler projectile points, shell beads, and a perforated red deer canine. Near the base of the cave (area C), another Aurignacian deposit overlays an Early Ahmarian assemblage, characterized by a technology of long, narrow blade/bladelets from uni- or bidirectional cores and a toolkit of endscrapers, burins, and el-Wad points. A small number of Initial Upper Paleolithic (IUP) and Middle Paleolithic (MP) artifacts have been found in the cave, but these industries have not yet been uncovered in stratigraphic sequence.

Here, we report 47 radiocarbon dates used to establish the EUP chronology and to understand site formation processes at Manot (Fig. 2 and table S5). The dates were produced from 41 charcoal and 6 sediment samples, some of which were divided and subjected to different pretreatment procedures for a total of 86 accelerator mass spectrometry (AMS) measurements (texts S4 and S5 and tables S3 and S5). Radiocarbon dates in the text are reported as calibrated 68% probability density functions (PDFs). Dated samples came from the most intensively excavated areas, E and C (text S2 and fig. S1). By the current cave entrance at the top of the west talus, area E has preserved occupational surfaces indicated by semibrecciated sediment, concentrations of artifacts, and in situ hearths (fig. S2 and table S1). Dated charcoals were collected from hearths in area E ($n = 8$), associated with post-Levantine Aurignacian and Levantine Aurignacian artifacts (fig. S10).

Area C is near the bottom of the west talus, approximately 30 m southeast and 20 m below the current cave entrance (figs. S3 and S11). No clear-cut occupational surfaces were observed through excavation or geoarchaeological analyses. However, anthropogenic material seems to have been redeposited in sequence from primary contexts higher on the slope, resulting in a package of Aurignacian artifacts overlying a package of Ahmarian artifacts (fig. S4 and table S2). Isolated charcoals ($n = 23$), collected from a 1.5-m-long section, show increasing age with depth, dividing into a younger and older cluster. The younger cluster of 38 to 34 ka cal BP came from higher in the section (unit 4 and top of unit 5) associated with mostly Aurignacian artifacts. The older cluster of 46 to 42 ka cal BP came from lower in the section (unit 7 and bottom of unit 6), associated with mostly Ahmarian artifacts. Only three dates deviate from this pattern. The samples (RTD-7783A, RTD-7785, and RTD-7786) came from midway through the area C section and show reverse stratigraphy or decreasing age with depth. This 15-cm portion of the section ($z = 205.50$ to 205.35) is relatively rocky, suggesting stronger water activity, which could have led to mixed and missing deposits. We do not consider this area of the section representative of the cultural sequence and have excluded these dates in estimating the ages of cultural phases.

Radiocarbon pretreatment and statistical analysis

To remove contaminants from fossil charcoals before radiocarbon dating, laboratories routinely apply a number of pretreatment procedures,

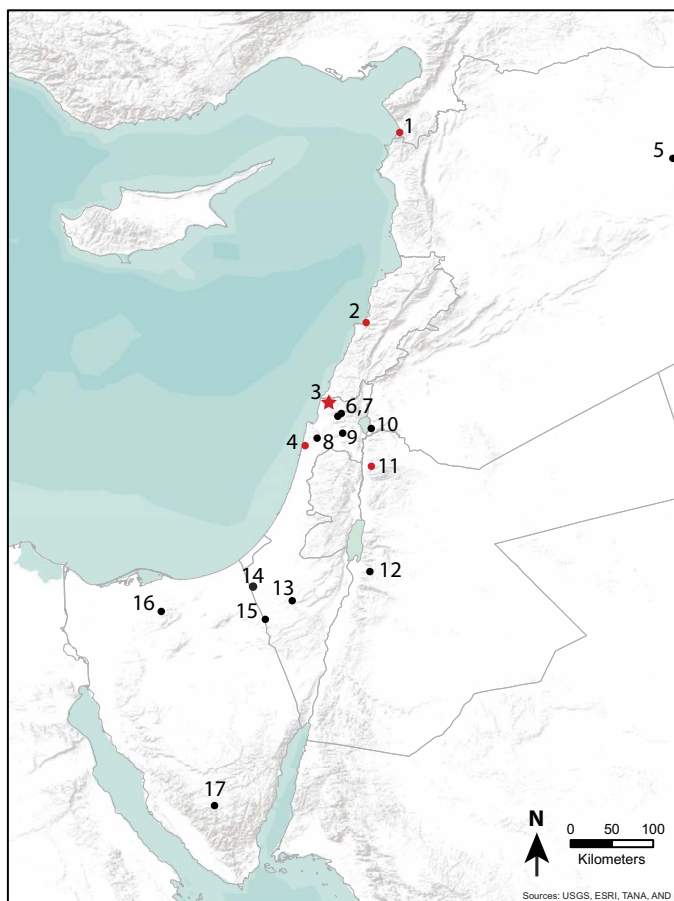


Fig. 1. Map of Levantine EUP sites mentioned in the text. Sites included in regional chronology are red. The Manot Cave is indicated by a star. 1, Üçağızlı; 2, Ksâr 'Akil; 3, Manot; 4, Kebara; 5, Wadi Kharar 16R; 6, Hayonim; 7, Meged; 8, Raqefet; 9, Qafzeh; 10, Nahal Ein-Gev I; 11, Mughr el-Hamamah; 12, Tor Sadaf; 13, Boker A/Boker Tachtit; 14, Nahal Nizzana XIII; 15, Qadesh Barnea; 16, Lagama VII; 17, Abu Noshra. USGS, U.S. Geological Survey; ESRI, Environmental Systems Research Institute; TANA, Telugu Association of North America; AND, Automotive Navigation Data.

Table 1. Excavation contexts and date ranges of cultural phases.

Cultural phase	Context dated	Date ranges cal BP 68.2% (sample number)
Post-Levantine Aurignacian	In situ occupational surfaces Area E, unit 2, layer I, loci 500 and 501	34,030–33,050 (6)
Levantine Aurignacian	In situ occupational surfaces Area E, unit 2, layer IV, locus 502	36,860–35,960 (2)
	Secondary talus deposit Area C, unit 4; area C, unit 5 above $z = 205.50$	38,260–34,050 (11)
Ahmarian	Secondary talus deposit Area C, unit 6 below $z =$ 205.35 ; area C, unit 7	45,940–41,560 (8) 49,440–41,600 (9)

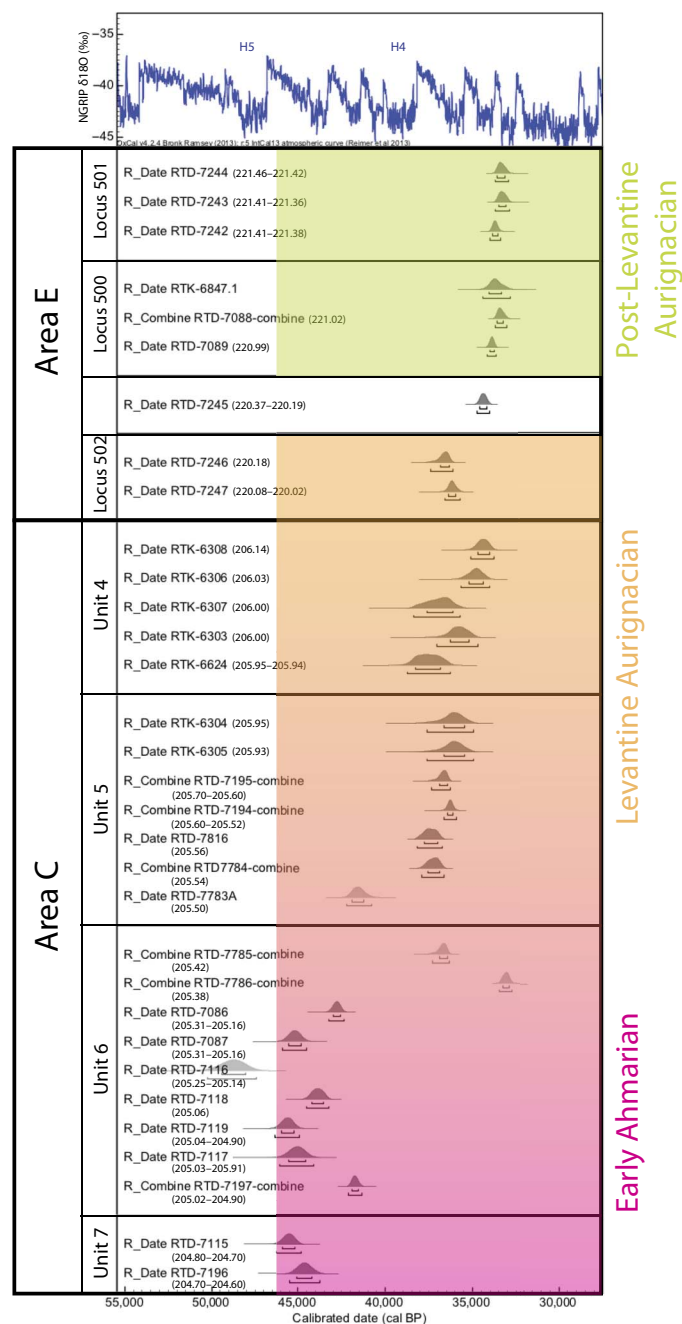


Fig. 2. Radiocarbon dates for archaeological chronology of Manot Cave. Samples from areas E and C. J squares are shown as PDFs calibrated with OxCal v4.2 (59) and IntCal13 (60). Dates are listed in stratigraphic sequence by laboratory code, followed by absolute elevation or the elevation range of the sample's excavation basket. The associated archaeological culture is indicated by color: post-Levantine Aurignacian, green; Levantine Aurignacian, orange; Early Ahmarian, pink. NGRIP, North Greenland Ice Core Project.

including the acid-base-acid (ABA) method, acid-base wet oxidation (ABOx) method, and stepped combustion. Controlled studies, comparing the effectiveness of different pretreatment procedures, have produced inconclusive results. Numerous studies have yielded older and more reliable dates with the harsher ABOx and stepped combustion methods (13, 14). However, studies of charcoals from certain Levan-

tine EUP sites found that ABA-treated fractions had better preservation parameters and older or statistically indistinguishable dates compared to their ABOx-treated pairs (text S4, fig. S8, and table S4) (8, 9, 15). These results indicate that the appropriate pretreatment method for fossil charcoal depends on the preservation state and depositional environment of the samples and thus should be experimentally determined for a given site (8, 16).

Here, experimental charcoals from Manot were separately divided into homogenized subsamples and treated with six different procedures: ABA, ABOx, and those methods, followed by stepped combustion to 630° or 900°C (text S4, fig. S6, and table S3). The fractions treated by ABA without stepped combustion produced the smallest percent modern carbon (pMC) values and Fourier transform infrared (FTIR) spectra, showing that sediment had been removed from samples (fig. S7). Radiocarbon dates of the total organic carbon in sediment were 16 to 10 ka younger than associated charcoals, so we expect any contamination from sediment to have made dates younger rather than older (text S5 and table S5). These combined results—smallest pMC values and demonstrated removal of younger-aged sediment—indicated that ABA without stepped combustion was the most effective method for charcoals from Manot. We recommend the methodology and parameters used to reach this conclusion (FTIR and pMC by different pretreatments) be used in future studies to determine the most effective method for samples from other sites, which may not be the same as Manot.

Bayesian models were produced that constrain dates based on stratigraphic information to test for outliers and estimate the span of cultural phases (text S6, fig. S12, and tables S7 to S9). However, our conclusions are based on the unmodeled ranges, which are less influenced by interpretations of stratigraphy and depositional history. The cultural span model (model 1) comprised three sequential phases of Ahmarian before Aurignacian before post-Levantine Aurignacian. It included dates from combustion features of area E and the sequence in area C (J squares with the exception of three dates described above that showed reverse stratigraphy). In this model, only 1 of 28 dates was identified as an outlier (fig. S12 and table S7). This was RTD-7116, a date of 49 to 48 ka cal BP that is significantly older than any other radiocarbon dates from Manot. Although the sample produced a finite radiocarbon age ($48,700 \pm 700$ ^{14}C years BP), it extends beyond the 50,000-year age limit of the calibration curve at 95.4% confidence and therefore may exceed the age limit of the radiocarbon method. The sample came from midway through unit 6 and could be the oldest Ahmarian date at Manot or represent an earlier phase because artifacts suggestive of the IUP and MP were recovered from the base of the sequence. The latter seems more likely, considering the tight spread of other Ahmarian-associated dates of 46 to 42 ka cal BP.

The modeled spans were the same as the unmodeled ranges, except that the Aurignacian was reduced to ~2000 years from an unmodeled range of 38 to 34 ka cal BP to a modeled span of 37 to 35 ka cal BP. The improved precision comes with the caveat that there is no continuous in situ stratigraphic boundary between the Ahmarian and Aurignacian sequences; rather, the boundary is inferred on the basis of artifact composition and clustering of dates in area C (text S6). In contrast, the end of the Aurignacian is bound stratigraphically by the overlying in situ post-Levantine Aurignacian layer in area E, dated to 34 to 33 ka cal BP. Moreover, the modeled range aligns with dates from the most secure Levantine Aurignacian context at Manot, the combustion feature in area E dated to 37 to 36 ka cal BP.

DISCUSSION

Implications for Levantine EUP chronology

In addition to Manot, there are four sites with large sequences of radiocarbon dates (>10) produced by modern analytical methods and associated with EUP assemblages (Figs. 1 and 3, text S7, and data set S1). Three of these are caves or rock shelters along the Mediterranean coast: Üçağızlı in Turkey (17), Ksâr ‘Akil in Lebanon (9, 18), and Kebara in Israel (8). The fourth is Mughr el-Hamamah in the Jordan Valley (19). Other sites in the southern arid zone, including Abu Noshra I and II, Boker A, Qadesh Barnea, and the Lagaman sites, have fewer dates, and those were mostly produced in the 1970s and early 1980s with less reliable methods (20, 21). Thus, as it stands, the Levantine EUP chronology is based on Mediterranean coastal sites, which underrepresent the assemblage variability of the region. Relations between coastal and arid sites are unclear, and the timing and character of industries may have differed between these zones.

The post-Levantine Aurignacian assemblage at Manot is tightly dated to 34 to 33 ka cal BP by charcoals from in situ combustion features. It is similar to assemblages, described as Atlitian, located in the

Mediterranean zone (text S2). The age of these assemblages has been estimated ~27 or 26 ka cal BP—younger than the Manot dates—but is considered problematic based on limited stratigraphic and chronometric data (22, 23). The secure Manot dates do align with the few dates from Atlitian layers at Ksâr ‘Akil (phase 6, level VI), approximately 35 to 32 ka cal BP (9, 18).

Dates included in the regional chronology for the Levantine Aurignacian, coming from Ksâr ‘Akil (*n* = 3) and Kebara (*n* = 7), have large uncertainties, which only constrain the timing of the phase to some time between 42 and 34 ka cal BP. The Manot data provide a relatively large sequence of high-precision dates for the industry (*n* = 13), which establishes a firm chronological peg for the Levantine Aurignacian at least between 38 and 34 ka cal BP and probably more precisely between 37 and 35 ka cal BP. The Levantine Aurignacian at Manot is also stratigraphically bound by the in situ post-Levantine Aurignacian surface dated to 34 to 33 ka cal BP. These chronostratigraphic data support views that the Levantine Aurignacian *sensu stricto* was a relatively short-lived archeological phenomenon (~2000 years), restricted to the Mediterranean vegetation belt (24).

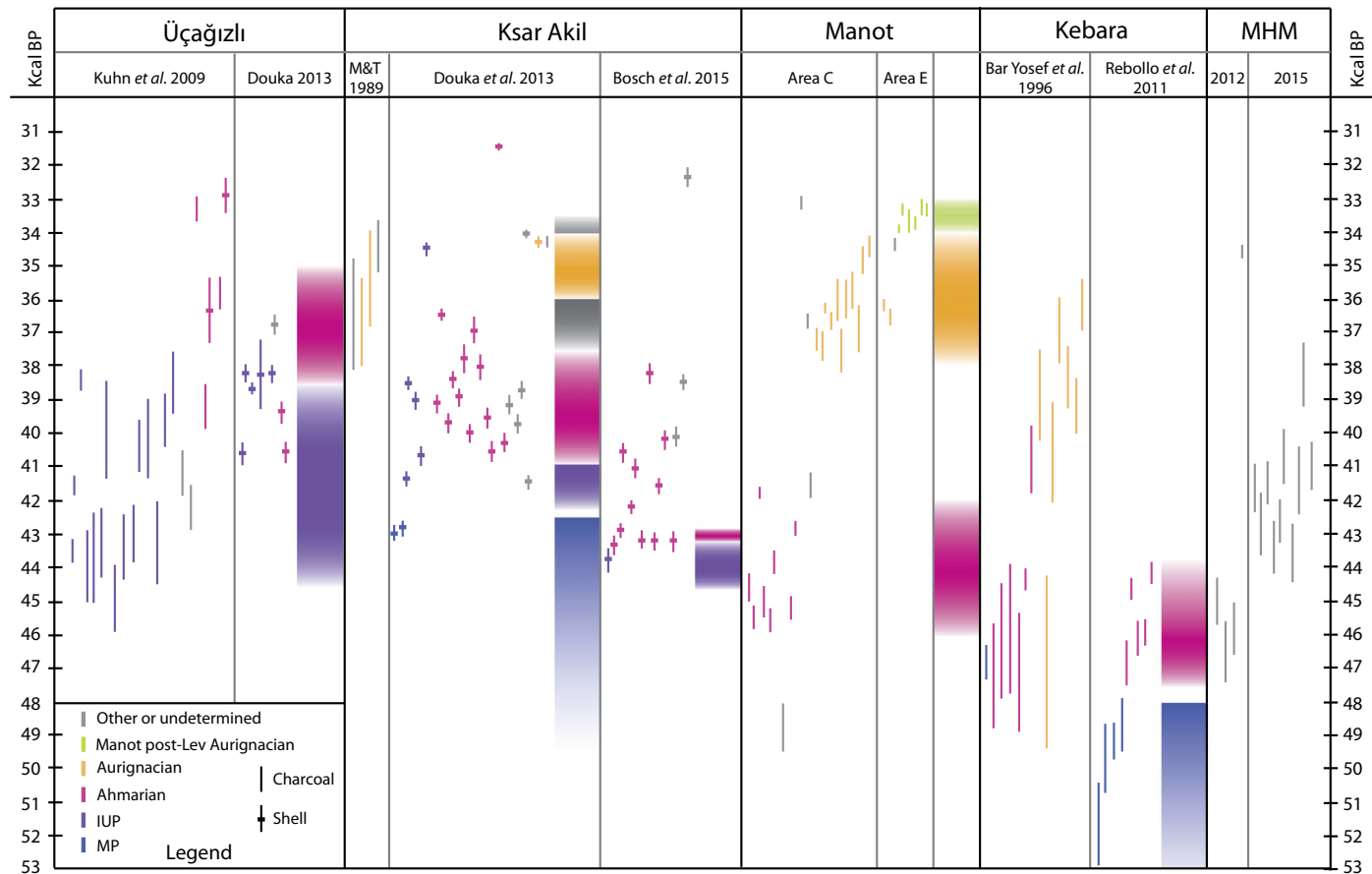


Fig. 3. Regional chronology of radiocarbon dates for stratified EUP sites between 50 and 30 ka cal BP. Vertical lines are charcoal dates, and crossed lines are shell dates. Dates are calibrated as 68.2% PDFs using the OxCal v4.2 software (59) and the IntCal-Marine13 calibration curve (60). Dates are color-coded by associated archeological industry and organized into columns by site and study. Within a given study, dates are ordered in stratigraphic sequence (from the lowest elevation or layer on the left to the highest on the right), as precisely as this information is known. The shaded blocks represent the phase ranges reported by particular studies. These ranges are the result of Bayesian models, with the exception of Manot, which shows the full unmodeled range. M&T, Mellars and Tixier (61); MHM, Mughr el-Hamamah. Dates assigned to “other or undetermined” do not necessarily represent the same industry between sites and strata. Site and date information is found in text S7 and data set S1.

The Early Ahmarian appears to have begun by 47.5 to 46 ka cal BP at Kebara (8) and Manot and then around 43 ka cal BP (18) or 40 ka cal BP (3) at Ksâr 'Akil and Üçağızlı. This 3000- to 7000-year difference may be because people at Manot and Kebara produced the Early Ahmarian several thousand years earlier than people at Ksâr 'Akil and Üçağızlı. The discrepancy may also result from mischaracterization of archeological assemblages or incorrect assumptions that named stone tool industries were made by socially related people. However, the relevant assemblages at Manot, Kebara, Ksâr 'Akil, and Üçağızlı show a high degree of technotypological similarity within a narrow geographic and stratigraphic range, corresponding to the "Northern Early Ahmarian" (25). The Early Ahmarian is thought to have developed locally from the IUP (26, 27). Although the more southern sites of Manot and Kebara do not have stratified IUP assemblages, the well-dated Early Ahmarian layers are earlier than or contemporaneous with the IUP at Ksâr 'Akil and Üçağızlı. In this case, the people of Ksâr 'Akil and Üçağızlı would have gradually developed an industry 3000 to 7000 years after it was fully developed less than 500 km to the south.

Alternatively, the disagreement could be explained by differences in the reliability of radiocarbon dates related to sample context, material, and pretreatment methods (text S7). Regarding the issue of context, radiocarbon samples from Manot were collected during recent excavations from combustion features and known stratigraphic positions, which were characterized by several geochemical methods (text S3). Radiocarbon samples from Ahmarian levels of Ksâr 'Akil were recovered in the 1930s to 1940s with outdated excavation methods, and in the decades since, some specimens likely became mislabeled in terms of provenience (9). Inaccurate proveniences may explain the large number of outliers in Bayesian models that constrain Ksâr 'Akil dates to stratigraphic position [9 of 39 dates in the study of Douka *et al.* (9) and 6 of 16 dates in the study of Bosch *et al.* (18)].

Next, late appearance dates for the Early Ahmarian from Ksâr 'Akil and Üçağızlı were produced primarily from shells, whereas the early appearance dates from Manot and Kebara were produced from charcoals. Evidence that contamination has been removed from samples should be based on independent analyses of the dated materials rather than the ages obtained. For charcoal, several such methods have been developed (28–30) and proven reliable in intercomparison studies between laboratories (8). For shell, it is difficult to detect and demonstrate the removal of diagenetic carbonate from the original biogenic carbonate (31).

Last, when disagreements arise between charcoal dates for a given event, the older dates are generally considered more reliable and the younger dates are thought to reflect contamination (32, 33). This is because a small amount of modern carbon (<1%) can make Late Pleistocene samples appear thousands of years younger (34). Our pretreatment experiments showed that for charcoals from Manot, the ABA method produced the smallest pMC values (and oldest dates) and purest FTIR spectra. The same pattern was found for charcoals from Kebara (8). We hypothesize that this is because harsher treatments (ABOX and stepped combustion) destroy more of the charcoals. Then, any surviving contaminant clay comprises a greater portion of the measured sample (28), and some of these contaminants (for example, siliceous aggregates) are reactive with atmospheric CO₂ (8). Because dated sediment at Manot is approximately 15 to 10 ka younger than associated charcoals, contamination from clay would make dates younger. Thus, we argue that the Ahmarian appeared in the Levant by at least 46 ka cal BP based on the early appearance dates from Kebara and Manot.

This age is also older than some dates for the Ahmarian from the southern arid zones of the Levant (Negev and Sinai) (20, 21). The arid zone dates should be viewed with caution because they include a small number of samples and were produced decades ago. However, they highlight a potentially interesting pattern for future research that the Ahmarian may have originated in the coastal Levant and then spread southward into the arid zones.

Implications for relations between the Near East and Europe

Here, we use the Manot chronology to evaluate proposals that the Ahmarian led to the Protoaurignacian in Europe and that the European Aurignacian led to the Levantine Aurignacian. These hypotheses are founded on the premise that archeological industries can be used to trace migrations and relations of past human groups. Although this approach is widely applied, it must be justified for each context (35). Assemblages should be systematically compared within a framework that considers artifact traits resulting from socially learned, idiosyncratic choices in material culture production to distinguish between hypotheses of independent invention and cultural transmission (36). Although this research is ongoing, it is also essential to test the conclusions against chronometric data. Accurate chronologies constrain hypotheses by refuting scenarios that do not accord with the timing of archeological industries.

It has been proposed that the Levantine Aurignacian was an intrusive industry, introduced by makers of the European Aurignacian (text S1) (7, 37–39). The view that the Levantine Aurignacian was nonlocal is supported by statistical comparison of technological and typological traits of lithics from Ksâr 'Akil, showing that Levantine Aurignacian traits (phase 5, levels VIII to VII) differ significantly from all other EUP layers (40). Regarding ties to the European Aurignacian, the broad similarities include thick scrapers made on flakes (nosed, carinated, and shouldered), Dufour bladelets, bone/antler points, and animal tooth pendants (40–43). Some of the shared features are characteristic of the Early Aurignacian [Aurignacian I (44–46)], such as Aurignacian re-touched blades, scrapers with scalar lateral retouch, and flat carinated items (7, 39). However, other features, including nosed and shouldered pieces, twisted Dufour bladelets, and simple-based antler points, resemble tools of the later Evolved Aurignacian [Aurignacian II and III (46, 47)]. Thus, linking the Levantine Aurignacian to a particular phase of the European sequence is an open question, which must be addressed through systematic comparisons of assemblages between regions. There have been few studies of this nature, although Tostevin (36) showed that blank production and toolkit morphology of lithics from Aurignacian layers of Kebara and Central Europe (Stránská skála, Czech Republic) are more similar to each other than to preceding strata at each site. In addition, Tejero *et al.* (47) demonstrated similarities between Aurignacian assemblages at Manot and Europe in the uses and production methods of osseous tools.

In terms of chronology, the secure Levantine Aurignacian dates from Manot are contemporaneous with or slightly later than the 39.5 to 35.5 ka cal BP modeled start dates of the Evolved Aurignacian in Southwest France/Northern Iberia at L'Arbreda, La Viña, and Abri Pataud and substantially later than Early Aurignacian assemblages, which begin between 43.5 and 40 ka cal BP across Europe at sites including Abri Pataud, Labeko Koba, and Willendorf II (48, 49). Thus, dates from Manot do not refute the hypothesis that the Levantine Aurignacian developed from a European Aurignacian precursor.

It is often claimed that the Ahmarian led to the European Protoaurignacian (1, 6) based on shared features of shell ornaments and

long, straight blades/bladelets produced by soft hammer reduction, possibly for projectile weapons (50). The Protoaurignacian appears to be intrusive in European sequences because it is typologically and technologically distinct from assemblages in preceding layers (51). However, systematic studies comparing Ahmarian and Protoaurignacian assemblages are lacking. The Ahmarian-Protoaurignacian hypothesis has been challenged because late appearance dates for the Ahmarian of ~40 ka cal BP from Ksâr 'Akil and Üçağzılı (3) are younger than the earliest Protoaurignacian dates of 44 to 41 ka cal BP from sites including Isturitz, Riparo Mochi, L'Arbreda, and Fumane (48). In contrast, early appearance dates from Manot and Kebara begin the Ahmarian by 46 ka cal BP, securely before the earliest Protoaurignacian, and therefore allow for the hypothesis that the Ahmarian of the Levant gave rise to the Protoaurignacian of Europe.

Testing proposed affinities between the Ahmarian and Protoaurignacian, as well as the European Aurignacian and Levantine Aurignacian, will require more systematic comparisons of the material cultural remains (that is, lithic, shell, bone, and antler artifacts) from each region. However, these hypotheses cannot be evaluated without accurate, high-precision chronologies. The results from Manot Cave provide a chronological foundation, which is critical for understanding the spread of modern humans and Upper Paleolithic traditions.

MATERIALS AND METHODS

Radiocarbon dating

The archeological chronology was based on radiocarbon dates of charcoals (Fig. 2, Table 1, and table S5). In area E, the charcoal samples were selected from combustion features, whereas in area C, charcoals were chosen to cover as much of the sequence vertically as possible. Charcoal pieces were collected by hand during excavation or from exposed sections and wrapped in aluminum foil with associated sediment. Several charcoals were collected from micromorphology blocks, as the blocks were cut and removed. Charcoals were identified using a metallographic microscope (Nikon Eclipse LV150N). The vast majority of charcoals at Manot are *Amygdalus* sp. (almond), and all dated specimens were identified as belonging to this taxon. No bones yielded collagen. Approximately 40 bones were sampled, collected from all excavation areas, and in varying taphonomic states. Although dietary and ornamental marine shells were recovered, they were not dated because it is unclear whether diagenetic carbonate can be separated from original biogenic carbonate (31).

Samples were characterized and prepared for radiocarbon dating based on tailor-made procedures at the DANGOOR Research Accelerator Mass Spectrometry (D-REAMS) Laboratory (text S5) (8, 28, 52). Before and after pretreatment, samples were analyzed by FTIR spectrometry to test the purity of the material. Approximately 50 mg of each charcoal piece was cleaned of sediment with a scalpel and homogenized by crushing with an agate mortar and pestle. Most samples were then treated with the following ABA procedure: (i) acid treatment in 1 M HCl for 30 min, followed by rinsing with Nanopure water until it reached pH 6, (ii) base treatment of 0.1 M NaOH for 15 min, followed by rinsing until it reached pH 6, and (iii) acid treatment in 1 M HCl for 1 hour in a water bath of 80°C, followed by rinsing until it reached pH 6. Because of their small size, four charcoals included in the chronology were treated with a water-base-acid regime, which followed the same procedure except that the first acid treatment was replaced by a wash with Nanopure water.

Samples were dried overnight at ~60°C, combusted to CO₂ with ~200 mg of CuO at 900°C, and then reduced to graphite in a vacuum line. Four samples were divided and underwent graphitization on the standard vacuum line and on an ultraclean line, dedicated to samples over 30,000 ¹⁴C years BP. Samples with laboratory code RTD were measured by AMS at the D-REAMS Laboratory (53), whereas those with RTK were measured at the National Science Foundation (NSF)-Arizona AMS Facility, University of Arizona. Stable isotope measurements were conducted at the Geological Survey of Israel.

Radiocarbon dates were produced for sediment to evaluate how contaminant sediment would affect charcoal dates. Four sediment samples were directly removed from dated charcoal samples, and two additional sediment samples were collected from the section. The sediment samples were crushed, homogenized, and then dissolved in 1 M HCl, followed by three rinses with Nanopure water. The remaining fraction contained the total organic carbon (TOC) and was prepared to graphite, as described above. The sediment TOC dates were between 16,000 and 10,000 years younger than their associated charcoals (table S5).

Determination of radiocarbon pretreatment

To determine the best pretreatment method for charcoals from Manot, four charcoal samples from area C were separately homogenized, divided, and prepared by different procedures (text S4). These experimental charcoals were subjected to six procedures: ABA, ABOx, and both of those treatments followed by stepped combustion at 630° or 900°C. The ABOx procedure consisted of the following: (i) 6 M HCl for 1 hour, followed by rinsing with Nanopure water, (ii) 1 M NaOH for 1 hour, followed by rinsing with Nanopure water, and (iii) 0.1 M K₂Cr₂O₇ in 2 M H₂SO₄ at 60°C overnight. After rinsing with Nanopure water, samples were oven-dried at 105°C. The step combustion procedure followed (i) precombustion at 300°C in 750 torr O₂ for 30 min (this should remove the more recent contamination), (ii) combustion at 630°C with CuO for 2 hours in vacuum and collection of the CO₂, and (iii) combustion at 900°C with CuO for 3 hours and collection of the CO₂.

In total, 31 fractions were measured from the original four charcoals. The results are reported as pMC (fig. S6 and table S3). The same background correction of 0.263 ± 0.032 pMC was applied to all fractions, which reflects graphitization and AMS steps. The pretreatment background correction was not applied so that pMC values resulting from different procedures could be directly compared. In this way, the pMC values indicate the effectiveness of the specific pretreatments; however, a background correction reflecting graphitization, AMS, and pretreatment was applied in calculating the reported radiocarbon dates in table S5. The results show that the ABA treatment procedure without step combustion produced the smallest pMC measurements and oldest ages. Explanations for this pattern are discussed in text S4.

Geoarcheological analysis

In conjunction with other geoarcheological work at Manot Cave, focused mineralogical analyses were conducted to support radiocarbon sampling and interpretation. The analyses included micromorphology, loose sediment characterization, and experimental heating of local control sediment. For micromorphological study, intact sediment blocks were taken from throughout the vertical section of area C (fig. S3) and from specific features in area E. Air-dried blocks were impregnated with polyester/styrene resin, cured, cut with a rock saw,

and sent to Spectrum Petrographics, where they were prepared into 30- μm -thin sections. The thin sections were analyzed with a petrographic microscope and described using conventional criteria and terminology (54, 55). Loose sediment samples were collected from surfaces, from sections, and with radiocarbon samples and then analyzed by FTIR. For all FTIR measurements, a few milligrams of sample were ground and homogenized with an agate mortar and pestle. Approximately 0.2 mg of the sample was mixed with ~50 mg of KBr powder and pressed into a 7-mm pellet with a hand press (Qwik Handi-Press, Spectra-Tech Industries Corporation) or a manual hydraulic press (Specac). FTIR spectra were measured at a resolution of 4 cm^{-1} for 32 scans between 4000 and 400 cm^{-1} using a Nicolet 380 (Thermo Fisher Scientific) (56, 57). Spectra and photographs of thin sections are available upon request.

Control sediment was collected from the surface of the cave base (area A) for an experimental heating study (58). The sediment was heated to set temperatures and analyzed by FTIR to determine the temperature-related transformations of the clay minerals contained in the local sediment. This calibration was then used to estimate the temperatures reached by sediment associated with putative combustion features. Fifty grams of sediment was homogenized and separated into 10 samples (5 g each). The samples were placed in ceramic crucibles and heated to different temperatures (0°C and 200° to 1000°C at 100°C increments) for 4 hours in a muffle furnace (A. Mandel, T21 type coupled with a Eurotherm 3216 temperature programmer). After heating, the sediments were analyzed by FTIR, as described above.

SUPPLEMENTARY MATERIALS

Supplementary material for this article is available at <http://advances.sciencemag.org/cgi/content/full/3/11/e1701450/DC1>

text S1. Levantine EUP
 text S2. Site description and archeological sequence
 text S3. Geoarcheological results
 text S4. Charcoal pretreatment: ABA, ABOx, and stepped combustion comparisons
 text S5. Charcoal preservation and radiocarbon results
 text S6. Bayesian modeling
 text S7. Regional chronology
 fig. S1. Top view and profile view of Manot Cave.
 fig. S2. Excavation area E with combustion features.
 fig. S3. Area C showing locations of radiocarbon samples and micromorphology blocks.
 fig. S4. Artifacts from Manot Cave.
 fig. S5. FTIR spectra of sediment exposed to different temperatures in experimental heating study.
 fig. S6. Radiocarbon measurements of Manot charcoal samples prepared by different pretreatments.
 fig. S7. FTIR spectra of charcoal sample before pretreatment, after ABA, and after ABOx.
 fig. S8. Comparison of ABA and ABOx charcoal dates from Levantine EUP sites.
 fig. S9. Characterization of *Amygdalus* sp. charcoal by scanning electron microscope and FTIR.
 fig. S10. Calibrated radiocarbon dates from area E plotted by absolute elevation.
 fig. S11. Calibrated radiocarbon dates from area C plotted by absolute elevation.
 fig. S12. Bayesian models and outlier analysis.
 table S1. Lithic assemblage in area E.
 table S2. Lithic assemblage in area C.
 table S3. Radiocarbon measurements of Manot charcoal samples prepared by different pretreatments.
 table S4. Comparison of ABA and ABOx charcoal dates from Levantine EUP sites.
 table S5. Radiocarbon samples and dates for Manot Cave.
 table S6. Excavation contexts with archeological classifications and date ranges.
 table S7. Outputs of Bayesian model 1 based on cultural phases.
 table S8. Cultural phase estimates for eight runs of model 1.
 table S9. Outputs of Bayesian model 2 based on lithostratigraphic units.
 data set S1. Published dates used to construct regional chronology.
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