Supplementary Information for:

Integrative Geochronology Calibrates the Middle and Late Stone Ages of Ethiopia’s Afar Rift

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   S1B: Laser ablation data for ostrich eggshell samples
   S1C: Uranium series data for ostrich eggshell samples
   S1D: Ar data for Didale Glass Shard Tuff sample MA 15-07
   S1E: Electron probe micro-analysis (EPMA) secondary standard data
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1. Methods

1.1 Field methods

Middle Awash field operations are guided by logistical and scientific project priorities and protocols (1, 2). The study area affords actively and extensively eroding deposits with freshly exposed fossils and artifacts and little vegetation cover. Aerial and satellite imagery guide reconnaissance transects; rock samples are taken in coordination with archaeologists and paleontologists; and sub-meter spatial positioning of stratigraphically controlled surface collection of individual specimens is accomplished by differential GPS (DGPS). Field work at Halibee began in 1999-2000 and MSA excavations were initiated in 2015. Field survey at Oulen Dorwa began in 1981, and the first LSA excavations were conducted in 2018. Field imagery of Halibee and Oulen Dorwa are shown in Figs. S1-S11; Figs. S12-S20 present examples of the paleoanthropological materials recovered in the Halibee member beds.

Chronostratigraphic control is foundational to paleoanthropological investigations in the Middle Awash. Stratigraphic placement of the Late and Middle Stone Age (LSA and MSA, respectively) occurrences in the Halibee member was initially accomplished by combining ground-truth field observations with high resolution multispectral and panchromatic satellite and aerial imagery to establish the relative sequence of deposition and erosion of the various stratigraphic packages involved. Measured sections (Fig. 2 of main text) were linked by tracing volcanic ash and other marker horizons on foot and integrating this ground-truth data with imagery and tephrochemical correlations.

1.2 Geochronological methods

Geochronological results from independent methods proved essential in calibrating the composite and detailed sections presented in Fig. 2. The integration of tephra chemistry, \(^{40}\)Ar/\(^{39}\)Ar, \(^{14}\)C, and the temporal bridging technique of \(^{230}\)Th/U burial dating of OES allowed greatly improved chronological placements for the four key sets of LSA and underlying MSA occurrences reported here. This provides the temporal scale against which ongoing and future studies of key fossils and artifacts will be measured, providing a model approach for other occurrences with similar geochronological challenges and resources. We utilized ostrich eggshells derived from paleoanthropologically rich and stratigraphically associated strata, as well as intercalated tephra, as dating materials. Tephra and obsidian artifacts were used as correlation materials.

1.2.1 \(^{14}\)C methods

Samples of ostrich eggshell (OES) weighing ~30-50 mg were prepared at the Berkeley Geochronology Center (BGC) and sent to be analyzed at the Chrono Lab at Queen’s University (Belfast, Northern Ireland) using established protocols for ratite eggshell. These included reaction with HCl to acid etch ~25% of the sample to reduce modern carbon contamination, similar to other \(^{14}\)C dating studies of ratite eggshells (3, 4). Following etching, samples are rinsed in deionized water and then hydrolyzed with phosphoric acid (\(H_3PO_4\)) in an Exetainer septa seal vial on a heating block at 90\(^\circ\)C to evolve carbon dioxide from the carbonate fraction as described
in ref. (5). The carbonate fraction, and not the organic fraction, was analyzed by accelerator mass
spectrometry (AMS) $^{14}$C, as methods to date eggshell organic fractions have not been developed
for ostrich eggshell yet. AMS $^{13}$C measurements were made directly on graphitized samples to
correct the $^{14}$C data prior to calibration to a calendar age. AMS $^{14}$C analyses were conducted at
the Chrono Lab. All $^{14}$C dates are calibrated using Calib8.2 using a 50/50 mixed calibration
curve combining SHCal20 and IntCal20 (6, 7) due to the near-equatorial position of the Middle
Awash study area. All ages for LSA deposits are presented as kiloyears before present (ka BP),
where present is 1950, as is conventional for $^{14}$C dates. Results are provided in SI Dataset S1A.

1.2.2 U and Th concentrations and $^{230}$Th/U dating

1.2.2.1 U and Th concentrations by Laser Ablation ICP-MS

$^{230}$Th/U analyses were carried out at the BGC using techniques similar to those described
in ref. (8). For laser ablation analyses, each OES fragment was cut to expose the cross-section of
the eggshell on a fresh surface and mounted in epoxy and polished to 3 $\mu$m grit. In situ
concentrations of $^{238}$U and $^{232}$Th in eggshell samples were determined via laser ablation
inductively coupled plasma mass spectrometry (LA-ICP-MS) using a Photon Machines Analyte
II excimer laser attached to a Thermo-Fisher NEPTUNE Plus ICP-MS. Analyses were
normalized to Ca by monitoring $^{43}$Ca and assuming stoichiometric abundance of Ca. Magnetic
field calibrations and signal intensities of $^{238}$U and $^{232}$Th were monitored using NIST 612 (37.38
and 37.79 ppm of U and Th, respectively; ref. 9). Five lines on each OES were first treated with
a pre-ablation step to remove any surface contamination at 10% laser output, 0.68 J/cm$^2$
fluence, 5 Hz rep rate, 65 $\mu$m spot size, and 50 $\mu$m/s scan speed. The following ablation step used 50%
laser output, 3.4 J/cm$^3$ fluence, 5 Hz rep rate, 65 $\mu$m spot size, 5 $\mu$m/s scan speed, all at 4.5 mJ
laser energy set point. $^{238}$U, $^{232}$Th and $^{43}$Ca were measured sequentially using magnetic field
switching in cycles lasting ~7 seconds. $^{238}$U and $^{232}$Th were measured using an ion counter while
$^{43}$Ca was measured on a Faraday detector. Grand Canyon Travertine LC 190.3, a homogeneous
calcite containing 2.52 ppm U (10), was used as an internal calcite standard and to calculate [U]
of eggshells. $^{232}$Th/U ratios were corrected for the reduced sensitivity to $^{232}$Th in the plasma and
ion optics via a relative sensitivity factor determined from contemporaneous analyses of ~0.1 mg
carbonate samples that were dissolved, equilibrated with mixed U-Th spike (see below for
details), desolvated, ionized, and analyzed. Results are presented in SI Dataset S1C.

1.2.2.2 $^{230}$Th/U geochronology

For each OES, two sub-samples of palisade-layer calcite were prepared and analyzed,
each located at different distances from the outer eggshell surface. Preparation included abrasion
with a Dremel tool to remove outer- and inner shell calcite with higher $^{232}$Th/U and/or erratic U
detections as indicated by laser ablation analyses. Sample thickness was monitored at each
stage using a digital caliper allowing the position of the abraded sub-sample relative to the outer
surface to be determined. Weathered edges of OES were also removed by abrasion, as weathered
edges may host additional U and Th taken up at a later stage from chemical weathering and/or
entrainment of detritus (8), and to avoid edge effects that would violate the boundary conditions
for 1-dimensional (1-D) diffusion. Large, radial pores for gas exchange in OES, which
Sub-samples were dated in BGC's U-Daughter Lab via solution analyses using a Thermo-Fisher NEPTUNE Plus multi-collector ICP-MS using methods described in (8). Eggshell tablets were totally dissolved in 7N HNO₃ and equilibrated with a mixed spike containing ²²⁹Th, ²³³U, and ²³⁶U. The spike was calibrated using New Brunswick National Lab CRM-145 uranium solution and solutions prepared from a 69 Ma U ore from Schwartzwalder Mine, Colorado, USA (SCH) that have been demonstrated to yield concordant U-Pb ages (11) and sample-to-sample agreement of ²³⁴U/²³⁸U and ²³⁰Th/²³⁸U ratios. U and Th were separated from sample matrix using two stages of HNO₃–HCl cation exchange chemistry followed by reaction with HNO₃ and HClO₄ to remove any residual organic material.

Samples were introduced into the ICP-MS using an Aridus II desolvator after mixing with dilute HNO₃-HF run solution. Each analysis included a wash step followed by an instrumental blank measurement, the sample measurement, and a measurement of peak tails. Sample measurements were carried out in peak-jumping mode with all masses measured on Faraday collectors during multi-collection except ²³⁰Th and ²³⁴U which were measured with an ion counter. Simultaneous collection of U and Th isotopes included measurement of ²³⁸U to normalize fluctuations in signal intensity. Instrumental backgrounds, Faraday collector baselines, and ion counter dark noise were measured at each U and Th mass while aspirating the same HNO₃-HF run solution used to dilute samples. Sample measurements incorporated within-run mass bias corrections using the known ²³³U/²³⁶U ratio of the spike. Ion counter yield (relative to Faraday collectors) was determined during each sample measurement by cycling ²²⁹Th and ²³³U between the ion counter and the L1 Faraday collector. Peak tails were measured on the ion counter at seven half-mass positions from m/e = 229.5 to 241.5 while aspirating sample solution and the sum of tails from all peaks was modeled and subtracted at each U and Th mass position. Measured peak heights were corrected for Faraday baselines/ion counter dark noise, instrumental backgrounds, Faraday gains, ion counter yields, mass fractionation, peak-tails, procedural blanks, and interfering spike isotopes. The external reproducibility of ²³⁴U/²³⁸U and ²³⁰Th/²³⁸U ratios of SCH solutions measured during analytical sessions was better than 0.17% and 0.21%, respectively. Unknowns were normalized to SCH reference values measured in the same run sequence to correct for run-to-run bias. Procedural blanks for ²³⁸U, ²³²Th, and ²³⁰Th ranged, in pg, from 106.1 to 235.3, from 0.006 to 4.6, and 0.00001 to 0.00008, respectively, and were subtracted from samples. Activity ratios and ages were calculated using the half-lives of (12) for ²³⁸U, (13) for ²³²Th, and (14) for ²³⁰Th and ²³⁴U.

The ²³²Th contents of OES samples were used to correct for unsupported ²³⁰Th (i.e., ²³⁰Th not produced by in situ decay of secondary U). ²³²Th is not a product of U decay and its presence indicates that associated ²³⁰Th is present. ²³²Th has a half-life of 1.405 × 10¹⁰ a (annum) and can be treated as a stable isotope on Quaternary timescales (the timescales at which ²³⁰Th/U dating is applicable). ²³²Th thus serves as an index for the amount of extraneous ²³⁰Th incorporated into the eggshells. By measuring the amount of ²³²Th in a sample and assuming a ²³⁰Th/²³²Th ratio for the unsupported ²³⁰Th, typically that of upper crustal silicates, extraneous ²³⁰Th may be estimated and subtracted from the sample (e.g., ref. 15). To make such a correction for OES, we assume the unsupported ²³⁰Th comes from fine-grained detritus derived from host sediments, which is often visibly present, and that the detritus is at or near secular equilibrium and has a crustal Th/U ratio. Specifically, we assign the following values and uncertainties to the detritus: (²³²Th/²³⁸U) = 1.21 ± 0.5; (²³⁰Th/²³⁸U) = 1 ± 0.1; (²³⁴U/²³⁸U) = 1 ± 0.1, subtract the indicated U and Th from
samples, and propagate resulting uncertainties into the final age errors. Initial (\(^{234}\text{U}/^{238}\text{U}\)) is back calculated from measured (\(^{234}\text{U}/^{238}\text{U}\)) and the corrected \(^{230}\text{Th}/\text{U}\) age.

Ages and uncertainties were calculated using Isoplot 3.75 and do not include uncertainties in decay constants (16, 17). Samples MA15-09-1 and MA15-12 were analyzed on November 20, 2017; MA15-09-2, 15-09-4, and 15-09-5 were analyzed on October 11, 2018, and MA15-09-3 on September 19, 2019. All activity ratios and ages are provided in SI Dataset S1C. Ages for LSA samples are presented as ka BP; MSA deposits are presented as both thousands of years (ka) and ka BP (Table S1).

### 1.2.3 \(^{40}\text{Ar}/^{39}\text{Ar}\) geochronology

Sample MA15-07 of the Didale Glass Shard Tuff was sieved and washed in distilled water in an ultrasonic bath. Anorthoclase crystals were concentrated with the aid of a magnetic separator and heavy liquids, while adhering residues were removed via an ultrasonic bath of dilute (5-7%) hydrofluoric acid. All samples were finally handpicked to purity. Samples were co-irradiated with Alder Creek sanidine (ACs; ref. 18) crystals at Oregon State University TRIGA reactor for 1 hour in the cadmium lined CLICIT facility.

Ar isotopic measurements of single anorthoclase crystals from the sample MA15-07 and the ACs sanidine standard were analysed on a Noblesse 5-collector sector-magnet mass spectrometer, configured with one axial Faraday detector and four off-axis, symmetrically arrayed ETP ion counters. Quasi-uniform heating of each sample (a single grain of anorthoclase or sanidine) was achieved via illumination with a CO\(_2\) laser fitted with a beam-shaping lens to generate a flat energy profile of adjustable diameter, typically 2 mm at the target distance. Individual grains of ACs sanidine were heated for ~30 seconds at progressively increasing power levels (1.4–8 W) until fusion was achieved, typically in 3-4 steps. Evolved gas was exposed for several minutes to an approximately -130° C cryotrap to remove condensable gases such as H\(_2\)O, and to a GP-50 SAES getter to remove reactive gases. Integrated ages of the step-heating results of ACs were used to calculate J values and provide the basis for interpolation of J values for unknowns using a planar fit. J-values determined from the fluence monitor are based on an age of 1.1891 ± 0.0008 Ma, 1σ; (19). Single grains of anorthoclase from sample MA15-07 were totally fused at 6 watts. Five Ar isotopes were measured, with simultaneous measurement of \(^{40}\text{Ar}, ^{37}\text{Ar},\) and \(^{36}\text{Ar}\) on separate ion counters over a period of ~1000 seconds, alternating with peak hopping to position \(^{38}\text{Ar}\) and \(^{39}\text{Ar}\) on the same ion counter as \(^{40}\text{Ar}\). All signals were normalized to the \(^{40}\text{Ar}\) ion counter. \(^{36}\text{Ar}\) signal normalization was achieved through periodic measurement of the \(^{40}\text{Ar}/^{36}\text{Ar}\) ratio of air (=298.56, ref. 20) inlet from an air-reservoir pipetting system. \(^{37}\text{Ar}\) and \(^{38}\text{Ar}\) signal normalizations were achieved through periodic measurement of \(^{40}\text{Ar}\) from a static gas sample on relevant detectors in a round-robin peak-hopping procedure. Procedural blanks, matching sample gas extractions precisely but without firing the laser, were run every four analyses, and were stationary over time. Mean blank values and their standard deviations were used to correct the unknown sample and air pipette data (SI Dataset S1D). For further details of the analytical procedures refer to (21). Interference corrections for all data are after (22) and are included in SI Dataset S1A.

### 1.3 Electron probe micro-analysis (EPMA) methods
Major and minor element analyses of tephra glass by electron probe micro-analysis (EPMA) are here used to provide characterizations and correlations. Tephrochemical correlations are based on evaluation of a full suite of elements, but particular attention is paid to the oxides of Fe, Ti, Mn, Ca, and Al plus Cl.

Grain-specific glass major element analyses of tephra samples were performed by EPMA at Los Alamos National Laboratory (LANL) and at the New Mexico Institute of Mining and Technology (NMT) following established methods previously used for Middle Awash Project tephra (e.g., refs. 23–25). Both polished thin sections and polished grain mounts were used. The LANL Cameca SX50 electron microprobe was operated at 15 kV accelerating voltage, 15 nA beam current, a fixed beam size of 10 µm and 20 s peak count times for glass analyses. The NMT Cameca SX100 electron microprobe is operated at 15 kV accelerating voltage and 10 nA beam current. Depending on grain size, beam sizes of 20, 15, or 10 µm were employed, using the largest allowed by the sample. Peak count times for all reported elements were 20 seconds, except for Na and Cl (40 s). Between 20-30 discrete spots were targeted per unknown sample facilitating the identification of chemical heterogeneities and multiple glass populations. Basalt and rhyolite secondary glass standards were run with all unknowns. Results for the secondary glass standards analyzed during analytical sessions that included tephra from this study are presented in SI Dataset S1E.

Results of individual analyses for each Halibee member tuff sample and sample averaged data are presented in SI Datsets S6 and S7, respectively, and unit average data are reported in Table S2. Raw data for each sample were filtered for obvious rogue analyses of mineral species (not listed) and for those with low analytical totals and/or alkalis (K and Na), unusual Ca and/or Al values, and clear compositional outliers, all of which are included in the dataset but excluded from averages and data plots. In cases where apparent “outlier” compositions are represented by multiple analyses, particularly when these appear in multiple samples, these compositions are considered as either distinct modes or clusters and are indicated as such in the dataset and averages and are displayed on data plots.

1.4 XRF methods

All archaeological samples of obsidian artifacts are analyzed whole. The results presented here are quantitative in that they are derived from "filtered" intensity values ratioed to the appropriate x-ray continuum regions through a least-squares fitting formula rather than plotting the proportions of the net intensities in a ternary system (26, 27). These data (through the analysis of international rock standards) allow for inter-instrument comparison with a predictable degree of certainty (28, 29). Using non-destructive EDXRF the trace elements in the mid-Z region have been found to be the best inter-source discriminators in most regions, including the eastern African Rift Valley (30–32).

All analyses for this study were conducted on a ThermoScientific Quant'X EDXRF spectrometer, located in the Geoarchaeological XRF Laboratory, Albuquerque, New Mexico equipped with a thermoelectrically Peltier cooled solid-state Si (Li) X-ray detector, with a 50 kV, 50 W, ultra-high-flux end window bremsstrahlung, Rh target X-ray tube and a 76 µm (3 mil) beryllium (Be) window (air cooled), that runs on a power supply operating 4-50 kV/0.02-1.0 mA at 0.02 increments. The spectrometer is equipped with a 200 l/min Edwards vacuum pump, allowing for the analysis of lower-atomic-weight elements between sodium (Na) and titanium (Ti). Data acquisition is accomplished with a pulse processor and an analogue-to-digital
Elemental composition is identified with digital filter background removal, least squares empirical peak deconvolution, gross peak intensities and net peak intensities above background.

Multiple Z conditions (conditions of fundamental parameter analysis, e.g., voltage, livetime, filter, and maximum energy) are designed to ameliorate peak overlap identified with digital filter background removal, least squares empirical peak deconvolution, gross peak intensities and net peak intensities above background. Elements of Na, Mg, Al, Si, and P were analyzed under low Zα conditions (6 kV, no filter, 10 keV maximum energy); elements K, Ca, Ti, V, Cr, Mn, Fe under Mid Zβ (32 kV, Pd filter, 40 keV maximum energy); Sn, Sb, Ba, Ag, Cd under high Zβ conditions (50 kV, Cu filter, 40 keV maximum energy); and elements S, Cl, K, and Ca under low Zβ conditions (8 kV, cellulose filter, 10 keV maximum energy). All conditions were run for 100 seconds livetime, under vacuum, and current is automatically set based on the mass absorption coefficient. For the analysis of mid-Z condition elements Ti-Nb (Kα1 lines), Ce (Lα1 lines), the x-ray tube is operated at 30 kV, using a 0.05 mm (medium) Pd primary beam filter in an air path at 100 seconds livetime to generate x-ray intensity Kα1-line data for elements titanium (Ti), manganese (Mn), iron (as Fe2O3), cobalt (Co), nickel (Ni), copper (Cu), zinc (Zn), gallium (Ga), rubidium (Rb), strontium (Sr), yttrium (Y), zirconium (Zr), niobium (Nb), and Lα1-line data for lead (Pb), cerium (Ce), and thorium (Th). Not all these elements are reported since their values in many volcanic rocks are very low and often outside the detection limits. Trace element intensities were converted to concentration estimates employing a linear or quadratic calibration line ratioed to the Compton scatter established for each element from the analysis of international rock standards certified by the National Institute of Standards and Technology (NIST), the U.S. Geological Survey (USGS), Canadian Centre for Mineral and Energy Technology, and the Centre de Recherches Pétrographiques et Géochimiques in France (33). Line fitting is linear (XML) for all elements. When barium (Ba) and cerium (Ce) are analyzed in the high Zβ condition, the Rh tube is operated at 50 kV and up to 1.0 mA, ratioed to the bremsstrahlung region (see refs. (29, 34). Further details concerning the petrological choice of these elements in Southwest obsidians and other volcanic rocks is available in refs. (29, 31, 32, 35–38). Twenty specific pressed powder standards are used for the best fit regression calibration for elements Ti-Nb, Pb, Th, Ba, and Ce include G-2 (granite), AGV-2 (andesite), GSP-2 (granodiorite), SY-2 (syenite), BHVO-2 (hawaiite), STM-1 (syenite), QLO-1 (quartz latite), RGM-1 (ryholite), W-2 (diabase), BIR-1 (basalt), BCR-2 (basalt), SDC-1 (mica schist), TLM-1 (tonalite), SCO-1 (shale), NOD-A-1 and NOD-P-1 (oceanic manganese; all USGS standards), NIST-278 (obsidian), BE-N (basalt) from the Centre de Recherches Pétrographiques et Géochimiques, and JR-1 and JR-2 (obsidian) from the Geological Survey of Japan (33).

Analysis of the major oxides of Na, Mg, Al, Si, Cl (as trace element in ppm), K, Ca, Ti, Mn, and Fe, is performed under the multiple conditions elucidated below. The fundamental parameter analysis (theoretical with standards), while not as accurate as destructive analyses (pressed powder and fusion disks) is usually within a few percent of actual, based on the analysis of the USGS RGM-1 rhyolite standard (see also ref. 29). The fundamental parameters (theoretical) method is run under conditions commensurate with the elements of interest and calibrated with ten USGS standards (RGM-1, rhyolite; AGV-2, andesite; BHVO-1, hawaiite; BIR-1, basalt; G-2, granite; GSP-2, granodiorite; BCR-2, basalt; W-2, diabase; QLO-1, quartz latite; STM-1, syenite), and one Japanese Geological Survey rhyolite standard (JR-1). The oxides are normalized to the RGM-1 USGS recommended versus measured values.
The data from the WinTrace™ software were translated directly into Excel for Windows software for manipulation and on into IGPET ver. 2000 for Windows and JMP ver. 12.0.1 for statistical analyses. In order to evaluate these quantitative determinations, machine data were compared to measurements of known standards during each run. RGM-1 a USGS obsidian standard is analyzed during each sample run of ≤19 for obsidian artifacts to check machine calibration. All data for samples are presented in SI Dataset S1H.

2. Results

2.1 \(^{14}\text{C}\) results and context

2.1.1 Oulen Dorwa Beds

OES from the Oulen Dorwa (OUD) Beds and the Wallia Beds were dated using AMS \(^{14}\text{C}\) (SI Dataset S1A). East of the Awash River are archaeological localities OUD-A1 and -A2 and paleontological locality OUD-VP-1 hosting tephra, archaeology, fauna, and associated OES. The LSA layer is intercalated with two fine grained reworked aphyric tuffs hosting glass shards (Fig. 2.; MA18-11 and 18-13). OES deriving from the archaeologically rich silty clay layer were surface-collected (Samples MA18-01 to 18-05) and \(^{14}\text{C}\)-dated to between 23.9 – 21.4 cal ka BP (95% C.I. for all \(^{14}\text{C}\) ages; Fig. 3; Table S1; SI Dataset S1A). The Oulen Dorwa beds comprise some of the youngest beds of the ~1 km sediment stack in the Middle Awash study area.

2.1.2 Wallia Beds

Fragments of a collapsed whole ostrich egg (MA15-12; Fig. 2; Fig. S25) collected from the Wallia Beds (Fig. S20) were eroding out of gray bedded to partially laminated silty clay ~2.1 meters above sediments hosting LSA archaeology (including gastropods and stone tools; Fig. S17) at locality HAL-A27. In between the OES and the archaeology is a very fine-grained, massive, gray, vitreous, aphyric tuff (MA15-11). This tuff outcrops at HAL-A26 underneath a second, light-medium gray fine aphyric tuff (SEVT; MA15-10) interbedded with medium brown to gray silty clay. At HAL-A27, this intercalated medium brown silty clay bed hosts root casts, carbonate concretions, and medium brown color, indicating the development of a soil profile. The age of the MA15-12 OES is a minimum age for the archaeology underlying it and for tuff MA15-11 at HAL-A26 and HAL-A27. Two fragments were analyzed by laser ablation ICP-MS, one eroding from the surface (MA15-12a) and one \textit{in situ} fragment (MA15-12b). MA 15-12b was \(^{14}\text{C}\) dated to 31.211 – 32.203 cal ka BP (95% C.I.).

The \(^{14}\text{C}\) ages for all OES samples from the Oulen Dorwa and Wallia Beds can be found in Table S1.

2.2 \(^{230}\text{Th}/\text{U}\) dating of ostrich eggshell

2.2.1 Background

Ostrich eggshells (OES) consist of ~2 mm-thick, low-Mg calcite (Fig S23) containing 1-3 wt.% organic matter and are suitable for \(^{230}\text{Th}/\text{U}\) dating, a technique capable of producing accurate, precise ages (~0.1%, 2\(\sigma\)) with a useful limit of >500 ka (e.g., refs. 39, 40). Similar to
other biominerals of higher animals, eggshells contain little to no primary U; accordingly, U in ancient OES is secondary. Until recently, $^{230}\text{Th}/U$ dating of eggshells has been carried out without consideration of the effects of prolonged secondary uptake of U (41, 42). In contrast, “$^{230}\text{Th}/U$ burial dating” of OES, which we employ herein, explicitly accounts for U uptake by OES from soil water after burial (8). Laser ablation profiles across eggshells demonstrate consistent patterns of secondary [U] in most samples that vary with eggshell structures and indicate that uptake of U across most of the shell takes place at the outer surface (Fig. S24; SI Dataset S1B; (8, 43). Three distinct layers of calcite are apparent in the cross-section of an eggshell and they mineralize so rapidly (<24 hours) that it is thought to be the fastest biomineralization process known (44, 45). The inner surface (the surface that defines the interior of an intact egg) is made of mammillary knobs and cones that are relatively rich in organic matter compared to the other calcitic structures in an ostrich eggshell. Interior to the eggshell surfaces are the palisade layer crystals (macroscopic columnar calcite crystal bundles). At the outer ~50um, the vertical crystal layer comprises the outer surface and is covered with a thin layer of nanocrystalline apatite, possibly linked to cessation of eggshell mineralization in the avian oviduct (45, 46). SEM images of eggshells show these calcite structures (e.g., 47), but are likewise visible in polished thin sections (8) and with the naked eye. [U] can be high in the outer vertical crystal layer, presenting a smooth continuous gradient of U into the palisade layer crystals (Figs. S24, S25). Then, in palisade crystals, [U] profiles are flattest, indicating fast uptake of U from the outer surface, until the transition from the palisade- to cone layer occurs, where nano-scale porosity is lowest and where U minima also occur (ref. 48); Figs. S24, S25). U concentrations rise again in the mammillary cone layer and can be erratic (SI Dataset S1B; Figs. S24, S25). A simple diffusion model accounts for the effects of prolonged U uptake on ages measured on OES sub-samples (8). Resulting model ages termed $^{230}\text{Th}/U$ burial ages have been shown to be concordant with $^{14}\text{C}$ ages measured on splits of several latest Pleistocene to Holocene eggshells (8) and in agreement with single-grain optically stimulated luminescence ages at MSA rock shelters (43). Having demonstrated coherent results with other chronometers, $^{230}\text{Th}/U$ burial ages can produce robust independent chronologies for Pleistocene terrestrial strata. This was recently demonstrated at a ~120-113 ka MSA shell midden, Ysterfontein 1, located on the west coast of South Africa, where coherent $^{230}\text{Th}/U$ burial ages provided a high-resolution chronology consistent with stratigraphic and sea-level constraints (43).

Although most uranium in ostrich eggshell is acquired after burial, accurate burial ages can be determined when two or more sub-samples of each OES are dated and the measured $^{230}\text{Th}/U$ ages are interpreted using a simple diffusion model (8). For $^{230}\text{Th}/U$ burial dating, we analyze two sub-samples at precise distances from the outer surface of the eggshell. These two sub-samples are called inner and outer shells, as they are sub-samples that are physically closer to the inner and outer surfaces, respectively, of the original eggshell fragment. We expect that if simple diffusive uptake of U occurred quickly from the outer surface (the diffusive surface) and U remained fixed in place upon uptake, then closed system U-series ages of two sub-samples should present either indistinguishable ages, or outer shell samples closer to the outer diffusive surface should be older than inner shell samples that took up U later. $^{230}\text{Th}/U$ burial dating thus provides reliability criteria inherent to the $^{230}\text{Th}/U$ data. For more details, see refs. (8, 43).

2.2.2 $^{230}\text{Th}/U$ results and context: Wallia and Faro Daba Beds
All laser ablation concentration data, 230Th/U isotope data, measured ages, and burial ages, can be found in SI Datasets S1B and C. U concentration profiles aid in determining optimal positions within OES for preparing inner and outer shell sub-samples. [U] ranges from ~29 to 52 ppb (median = 35.8 ppb; Table S2) in the palisade layer crystals, where profiles are flattest and most consistent across multiple ablation profiles, and where ratios of common 232Th to U are lowest. (SI Dataset S1B; Figs. S24, S25).

U concentrations for all samples indicate uptake of U occurred starting at the outer surface (Figs. S24, S25). U and Th isotope ratios from the inner and outer sub-samples of MA15-12b and three of five fragments of MA15-09 in this study are consistent with diffusive uptake of U at the outer surface (Figs. S25, S26) (8, 43).

Splits of sample MA15-12b from the Wallia Beds were dated by both the 14C and 230Th/U techniques. MA15-12a was not dated. For MA15-12b, the 230Th/U results show the outer shell age (29.8 ± 0.4 ka) is older than the inner shell age (28.4 ± 0.4 ka) of sample MA15-12b (Fig. S25), as expected for a simple diffusive uptake model to apply, so we calculate a burial age after ref. (8) and convert from ka (thousands of years ago) to ka BP (thousands of years before 1950) for direct comparison with the 14C results (Fig. S25; main text Fig. 3). Sample MA15-12b has a 230Th/U burial age of 30.59 ± 0.73 ka BP. 14C results of MA15-12b were not produced or analyzed until after all 230Th/U measurements and age determinations were made. The 230Th/U burial age overlaps within 95% C.I. with the 14C age (32.20 – 31.21 cal ka BP; Table S1). Because the 14C age dates the time of eggshell mineralization, and the 230Th/U burial age estimates the time the eggshell came into contact with soil pore water upon deposition, good agreement between the two results indicates that the eggshell was deposited shortly after the egg was laid, and U uptake began early upon deposition. The 14C and 230Th/U techniques are based on distinct assumptions, employ different isotopic systematics and analytical methods, and are subject to distinct modes of potential failure. Thus, good agreement between the two techniques indicates the ages from each approach are accurate.

The results from OES in the Wallia Beds lend confidence to applying the 230Th/U burial dating approach to older eggshells beyond the ~50 thousand-year 14C dating limit in other open-air sites in the study area, such as the MSA deposits in the Faro Daba Beds. Ostrich eggshell fragments (MA15-09) were recovered eroding out of a medium brown silty clay ~5 meters above the Afcaro Basaltic Tuff (AFBT), a thin (<1 cm) black pumiceous aphyric tephra deposit (main text Fig. 2) found across many MSA localities in the Faro Daba Beds. Archaeology in HAL-VP-9 within the Faro Daba Beds derives from sediments directly above and below the AFBT. Numerous OES fragments were collected at a single position from within a medium to light grayish brown silty clay, likely from a single egg or a larger eggshell fragment that was later broken into smaller pieces. Some were partly eroding out of the deposit and upon scraping into the deposit, more in situ fragments emerged. Thus, we expect these eggshell fragments to all be the same age. Five of these fragments were analyzed with 230Th/U geochronology (Fig. S23). Similar (234U/238U) ratios observed in inner and outer sub-samples of each OES are indistinguishable or overlapping at 2σ uncertainty for most samples (Fig. S26), consistent with rapid uptake of U and subsequent closed evolution of the 230Th/U system after U uptake ceased. MA15-09-1 had an inner shell age < outer shell age, indicating that a simple single-stage diffusive uptake model may appropriately characterize the first time of U uptake at the outer surface of the eggshell. We calculate a burial age of 94.3 ± 2.8 ka using the method of Sharp et al. (8). Two samples had inner shell and outer shell ages that were indistinguishable at 1σ uncertainty, and burial ages are calculated from weighted means for samples MA15-09-2.
(96.90 ± 0.70 ka) and MA15-09-5 (96.11 ± 0.69 ka), as indistinguishable inner and outer shell ages may indicate very rapid U uptake upon deposition.

One sample, MA15-09-4, did not meet our reliability criterion that the inner shell age should be younger than the outer shell age for a 1-D diffusive uptake model to apply. Accordingly, we interpret the inner shell age (96.6 ± 2.2 ka) as a minimum age for the sample. Similarly, MA15-09-3 produced an inner shell age that is older than the corresponding outer shell age at the 1σ level of uncertainty but overlapping at 2σ uncertainty. We also take the inner shell age (93.1 ± 1.5 ka) as a minimum age of this sample. These minimum ages are indistinguishable from burial ages of other MA15-09 samples, but because of the expected relationship of inner shell < outer shell age was not preserved in these two samples (Fig. S26), we conclude that more complex U mobility may have affected these samples, making their ages less reliable. Thus, these minimum ages are excluded from further interpretation of the age of the deposit despite their similarity to other samples. For the three samples that produced burial ages fitting a single-stage model of U uptake upon burial (8), these produce a weighted mean age of 96.4 ± 1.6 ka (95% C.I.). This provides a capping (minimum) age for the paleoanthropological materials underlying MA15-09 in the Faro Daba beds.

2.3 40Ar/39Ar results and context

Challenges of calibrating the embedded fossils and artifacts of the Halibee Member via interbedded volcanic deposits were substantial because the relatively few interbedded distal and fine-grained tephra are not only laterally discontinuous, but nearly all lacked minerals susceptible to 40Ar/39Ar dating of such young rocks. Prior work utilized 40Ar/39Ar and 14C dating to constrain the ages of the Faro Daba Beds at Halibee, including dating of obsidian by 40Ar/39Ar (49). The results did provide accurate ages, but the low precision of the 40Ar/39Ar ages required additional sampling. The MSA-rich Chai Baro Beds of the Halibee Member host The Didale Glass Shard Tuff (DGST). The glass shard dominated DGST also typically contains variable sized feldspar grains.

DGST sample MA15-07 was selected for 40Ar/39Ar work, and of 44 total anorthoclase grains analyzed, 20 were from a coarser fraction (>800 um, sample 37215) and 24 were from a finer fraction (400-800 um, sample 37214; SI Dataset S1D). The coarser fraction comprised 50% xenocrysts while the finer fraction only contained 8% xenocrystic material (main text Fig. 3), thus preserving most of the juvenile population. Of 44 grains, 32 juvenile grains were combined to yield an inverse isochron age of 159.4 ± 11.6 ka. Combining this age with the age determined by ref. (49) provides a weighted mean age of 158.1 ± 11.0 ka for the DGST. This is a minimum age for hominids and MSA archaeology localities in the Faro Daba Beds and a capping age for MSA localities underlying the DGST in the Chai Baro Beds (main text Fig. 2). While typical grain size separations may vary by laboratory from fewer than 100 um to more than 500 um, our results indicate that the separation of smaller ranges of grain sizes may be a way to selectively remove xenocrystic contamination. In this case, xenocrysts dominate the coarser grain-size fraction. We interpret this to suggest that these non-juvenile components were derived from the DGST eruptive system as opposed to being products of post-eruption transport or deposition processes. Moreover, DGST sample MA15-07 is poorly consolidated and fines upwards with up to coarse sized pumice grains at its base, characteristic of a primary fall deposit. Additionally, glass chemistry indicates a predominant compositional mode in agreement with a single eruptive source (SI Datasets S1F and G).
2.4 EPMA Results

The Halibee member tephra present as a fundamentally bimodal mafic-felsic suite (Table S2, SI Datasets S1F and G, Figs. S21, S22), similar to those of other well-characterized Middle Awash suites belonging to, for example, the Sagantole and Adu Asa Formations (23, 24, 50, 51). Within this broad context, individual Halibee member tephra range from compositionally homogeneous (unimodal) to multi- and bi-modal, to those displaying linear arrays through a compositional range (Fig. S21).

Tephra reported here and definitively associated with LSA assemblages are the Seegeri Vitric Tuff (SEVT) of the Wallia Beds and tuffs MA18-11 and MA18-13 of the Oulen Dorwa beds. An additional isolated tuff occurrence of similar antiquity is noted (MA04-16). This tuff and three correlated samples comprising the Seegeri Vitric Tuff are unimodal, forming tight clusters on bivariate plots, with both tuffs occupying unique Ca-Fe space (Fig. S21). In contrast, the Oulen Dorwa tuffs display abbreviated linear compositional arrays (Fig. S22). Additionally, the upper Oulen Dorwa tuff (MA18-13) has two very distinct compositional modes and a subtle additional mode/cluster. The high-Ca mode (5 shards) of MA18-13 is identical to glass from underlying MA18-11, thus likely was inherited from this deposit.

EPMA information also is provided for tephra associated with MSA assemblages of the Faro Daba (Afcaro Basaltic Tuff - AFBT) and Chai Baro (MA15-08, Bartikimber Vitric Tuff - BRVT, Didale Glass Shard Tuff - DGST) beds. Five correlated samples comprising the AFBT carry a distinctive chemical fingerprint characterized by glass capturing a range of differentiation but that can be distilled down into two basalt compositional modes (Table S2, Fig. S22). This, together with its occurrence as one of only two basaltic tuffs in the Halibee area make the AFBT an excellent marker horizon. The other Halibee member tuff with a significant basaltic component is the bimodal mafic-felsic tuff MA15-08. This tuff captures a more abbreviated range of basalt differentiation and also carries two readily distinguishable felsic modes. One of these felsic modes is chemically similar to the stratigraphically lower DGST. The two most prominent and widespread Chai Baro beds tephra are the BRVT and underlying DGST; the latter is exposed extensively throughout the Halibee region. These tephra are markedly different in appearance and chemical signature and pose excellent examples of two common types of tephra glass chemical signatures; one that preserves a pronounced linear array through a compositional range (BRVT; 2 samples), and one that is dominantly homogeneous/unimodal (DGST; 10 samples) (Fig. S21). One sample from each of these tephra units preserves multiple outlier modes/clusters as evidenced from the bivariate plots. Although true compositional modes cannot readily be discerned for the BRVT, we do provide average data for the apparent endmember clusters as well as for the entire array. The compositional space occupied by the main BRVT linear array sets it apart from all other analyzed Halibee member tephra. The very homogenous primary mode (225 of 230 analyses) of the DGST occupies unique Ca-Fe space thereby also readily distinguishing it from other Halibee region tephra (Fig. S21).

A small number of glass shard compositions from what is now known as the DGST were reported by Morgan et al. as sample MA04-9a (49) (equivalent to sample MA04-09b reported here), and this EPMA chemical signature subsequently has been suggested to correlate to the signature preserved by a subset of obsidian artifacts from the archaeology sites at Halibee, Aduma, and Porc Epic (52), which in turn had previously been linked to a likely source at Ayelu
volcano (53). None of the artifacts analyzed as part of the current study preserve this DGST signature.

2.5 Results of X-Ray fluorescence and archaeological obsidian sourcing

The results presented here (SI Dataset S1H) were compared to results from studies in refs. (52–55), all from sites in the Middle Awash and the Main Ethiopian Rift Valley. Obsidian samples in ref. (49) derived from surface contexts in the HAL-A2 locality within the Faro Daba beds, some of which produced an extrusion (maximum) age for Faro Daba fossils and artifacts of ~106 ka. This unknown ~106 ka geological source was represented by three pieces (MA-04-28K, 28O, and 28P) in Morgan et al. (49) and characterized in Negash et al. (52) as Type 10. In this study, 17 pieces were analyzed by EDXRF. These MSA finds were recovered from surface collections (sample ID’s starting with A2-) and from excavations (sample ID’s starting with A2B- or A25B-; SI Dataset S1H). Three pieces, two from surface collections (samples A2-58 and A2-91) and one in situ (sample A25B-E1-367), chemically match the ~106 ka obsidian of ref. (49) (Fig. S27). These confirm that the ~106 ka extrusion age can now be confidently assigned as a maximum age for surface-collected and in situ remains from the Faro Daba beds.

Unlike many eastern African rift rhyolites which are often peralkaline (56), the oxide concentrations indicate the obsidians analyzed in this study are relatively high Si rhyolites, tightly clustered, and uniformly peraluminous (Na2O+K2O<Al2O3; ref. 52). A conundrum in this assemblage is sample A2B-E3-10 with a trace element concentration statistically identical to Adokoma (also written Adukoma) obsidian but with peralkaline composition (Na2O+K2O/Al2O3>1; SI Dataset S1H; see also refs. 30, 57).

While there has been little research in archaeology on the effects of weathering and patination of volcanic rocks including obsidian, it does appear that low temperature alteration of obsidian with the inclusion of water affects Na and K concentrations, possibly an issue for dating. This process actually may be at work in this assemblage where at least the one sample (A2B-E3-10) mentioned above, a piece of patinated obsidian debitage, exhibits much higher Na and K proportions while seemingly not affecting the trace elements. We removed patination from the remainder of artifacts for this study.

Shackley and Dillian (58) found in peraluminous and peralkaline obsidian trace element concentrations are not affected by heating samples to near the melting point (<1000°C). The same seems to be apparent with patination although patination occurs at low temperatures (59). Patination effects, particularly on very old volcanic glass artifacts should be examined in future obsidian studies. It does seem apparent that trace element concentrations, generally better for source assignment than the more variable lighter elements, are not significantly affected by patination or heat.

Finally, the dominance of artifacts produced from Adokoma obsidian makes sense, but as has been discussed elsewhere the number of, as yet, unlocated sources in the Ethiopian and Afar rift regions hinders construction of exchange, group interaction, and social network models in archaeology (52, 55). The work ongoing in Ethiopia is a step in that direction.
Figure S1. Halibee member Overviews.
A. View to the WNW. Far horizon is the western Afar margin; horizontal green tree line mid-frame follows the modern Awash River channel. The upper part of the section west of the river comprises the Halibee member.
B. Aerial view to the NNW. Eroding badlands of the Wallia beds below the aircraft are in the Halibee catchment; the Wallia drainage is in the upper left.
C. Aerial view to the E. Badlands in the southern catchment of the Kada Halibee stream widely expose Halibee member sediments; Wallia beds in the foreground.
D. Aerial view to the E. Kada Halibee and Awash River are indicated by yellow and white arrows, respectively; the Faro Daba beds are best exposed in the right upper part of the frame.

NOTE: Throughout the SI figures, double-headed arrows indicate linked views of the same subject.
Figure S2. Chai Baro bed Overviews.
A. View to N; lower Chai Baro beds in the catchment of the Ounda Halibee. Arrow indicates step trench immediately N. of the HAL-VP-5/16 hominid cranium excavation.
B. View to E; HAL-VP-5/16 cranial excavation; yellow flags indicate erosional surface scatters of cranial fragments.
C. View to SSW; HAL-VP-5/16 cranial excavation; arrow to the far horizon indicates sediments stratigraphically and elevationally above the DGST (obscured by intervening ridge). This ~158 ka tuff overlies this *H. sapiens* cranium by ~9m. Inset figure shows left frontal; yellow flags indicate individual fragments.
Figure S3. Faro Daba bed Overviews.

A. View to E; lower Faro Daba beds platformed and eroding atop the DMCC (white arrows) W. of the incised Awash River, whose current channel is below the yellow arrow’s tip. Horizontal sediments beyond the Awash River belong to underlying members of the Dawaitoli Formation.

B. View to W; lower Faro Daba beds, Afar Rift western margin is far skyline. Camels standing on the DMCC, downthrown to the west by a small normal fault (blue arrows).

C. Artifacts and fossils eroding from the soft silts of the lower Faro Daba beds immediately atop the DMCC (white arrow). Reddish clay fragments (yellow arrow) are associated with an eroding burned tree stump.

D. Lithics and fossils selected from a 2m x 2m square to illustrate diverse raw materials and excellent preservation. Note adhering carbonate on the manuport and cobble pick.
Figure S4. Wallia bed Overviews.
A. View to SSW; lower Wallia beds in the S. catchment of the Kada Halibee. Tuff SEVT being sampled at far left (white arrow).
B. View to NNW; lower Wallia beds in the S. catchment of the Kada Halibee.
C. View to W; lower Wallia beds in the S. catchment of the Kada Halibee; western Afar Rift margin on skyline.
Figure S5. Overviews of Oulen Dorwa.
A. View to SSW; lava flows of the Name Koma hill (white arrow) blocked the upper catchment of the Messalou tributary (yellow arrow) of the Awash River, creating a small basin in which Late Pleistocene sediments accumulated unconformably atop eroded Pliocene deposits (cobble mantled dark surface in foreground).
B. View to ESE; LSA archaeological locality OUD-A1 main concentration centered around the members of the field crew.
C. Closer view to ESE, excavation marked by the group on the right; differential GPS surface collection by the group to the left.
D. View to ENE; Artifacts and manuports eroding from the OUD-A1 main concentration.
Figure S6. Chai Baro bed marker horizons.

A. View to NNE; the ~158 ka DGST is the prominent, resistant tuff (white arrow) interbedded within Chai Baro bed sediments in the southern Faro Daba area.

B. View to NNE; another outcrop of DGST in the southern Faro Daba area.

C. View to W; the base of the Chai Baro beds (and the Halibee member itself) is the top of the DMCC (white arrow), here exposed about 100m E of the HAL-VP-5/16 hominid.

D. View to W; the deep cut into the DMCC shows its thickness and resistance, forming an undulating platform upon which the fossiliferous silts of the lower Chai Baro beds were emplaced at the base of an abruptly fining-upward sequence in which clay-dominated sedimentation is marked by the overlying dark beds on the skyline.

E. View to NNW; another cross section of the DMCC, showing its basal unconformity with dark silty clays below.
Figure S7. Faro Daba bed marker horizons.
A. View to N; basal Faro Daba bed sediments eroding atop the resistant top of the DMCC (white arrow) are rich in artifacts and fossils.
B. View to N; a burned tree stump in the lower Faro Daba sediments provided charcoal that showed an infinite radiocarbon age.
C. Sampling the charcoal.
D. View to E; the AFBT basaltic tuff is the dark band exposed in a step trench through the lower Faro Daba beds.
E. View to NNE; the AFBT basaltic tuff thickness varies, increasing in channel fills (above photographer’s shadow) in the lower Faro Daba beds, in the southern catchment of the Kada Halibee.
Figure S8. Inverted channels cut into the Chai Baro beds.

A. Satellite imagery of the southern Faro Daba area reveals sinuous paleochannels (yellow arrows) cutting into the Chai Baro beds at the level of the DGST (red arrows).

B. Same imagery, enhanced to highlight vegetation and pebble gravels. Blue dots correspond to *Acacia* bushes that preferentially anchor in well-drained beds of modern streams (white arrows) and inverted paleochannels (purple arrows), rather than the soft, deflating silts flanking these features. The *Acacia* in photo C is indicated by the green arrow.

C. View to the S; the DGST (white arrow) is seen as a horizontal, white, resistant band in the distant outcrop. Person to the left is standing on a segment of the inverted channel near location (D).

D. View to WNW; pebbles and angulation mark the contact of the now inverted channel that cut into the Chai Baro silty clays to the left of frame. The figure on the horizon shows where this channel cut the white DGST (white arrow).

E. Section of the inverted channel showing clasts of silicified DGST (white arrows) incorporated when the paleochannel cut into the tuff.

F. Closeup of the section.
Figure S9. Inverted channel cut into the Faro Daba beds.

A. Satellite imagery of the southern Faro Daba area reveals a segment of now inverted paleo channels cut into Chai Baro sediments (older indicated by blue arrow) and Faro Daba sediments (younger channel indicated by yellow arrow).

B. Same imagery enhanced to show the paleo channels and spectral contrast between currently eroding Chai Baro (left upper quadrant) and Faro Daba sediments (right lower half of frame), both sitting atop the DMCC (lower border and lower right corner featuring dense *Acacia)*.

C. View to the W; the Afar Rift western margin is the skyline to the left. The DGST is the white horizontal stripe below it (red arrow), within the Chai Baro beds (see Text Figure 2). The hill behind the people is a segment of a now-inverted paleochannel whose bed load is more resistant to erosion than the softer silts of the Faro Daba beds that underlie it, and that it was cut into. Foreground: DGPS collection of fossils (blue flags) being exposed by erosion of the lower Faro Daba bed silts.
Figure S10. Emergent inverted paleo-channels flanked by lower Faro Daba bed sediments.

A. View to NW; expansive outcrops of the lower Faro Daba bed silts in the southeast catchment of the Kada Halibee drainage. Person holding DGPS antenna (same position as yellow dot in imagery (B) and photo (C) examines an outcrop of AFBT basaltic tuff south of the emergent paleo-channel (sandstone boulders).

B. Satellite imagery showing traces of E/W paleo-inverted channels running perpendicular to, and now being cut by modern drainage channels flowing north into the Kada Halibee.

C. View to E; sandstone boulders mark the course of the paleo-channel flanked by softer Faro Daba bed silts banked against it to its north, where the person stands. The channel feature is now in the process of re-inversion as the softer lower Faro Daba bed silts are deflated by wind and water erosion. Person stands between the underlying DMCC (white arrow) and the AFBT basaltic tuff.
Figure S11. Inverted paleo-channel in the Wallia sediments.

A. and B. are geo-matched panchromatic and multispectral satellite images illustrating the surface of the DMCC at the base of the Chai Baro beds (white arrow), overlain by reflective silts (blue arrow) exposed in the southern catchment of the Ounda Halibee drainage. Above these silts a dark band of clay-rich sediment is present above the DGST (orange arrow). The overlying reflective silts of the Wallia beds contrast with a sinuous paleo-channel cut into them (yellow arrows), now emerging as an inverted channel that is orthogonally crossed by tributaries in the modern Wallia catchment that currently drain to the south. This paleo-channeling, when combined with similar features cut into underlying strata—and now emerging as inverted channels—demonstrates that shifting deposition, erosion, drainage patterns, and channel inversion persisted through the entirety of the Halibee member in this area.
Figure S12. Chai Baro bed stone artifacts and vertebrate fossils.
A. Fresh basalt flakes from a single cobble eroding from the rootcast silty clay.
B. Excavation and DGPS recording of an *in situ* bovine cranium in the lower Chai Baro beds.
C. Fossil felid mandible.
D. Fossil rodent skull.
E. Fossil cercopithecine cranium.
Figure S13. Faro Daba bed MSA points.
All frames. Size and typo-technological ranges of geologically contemporaneous MSA points found eroding from the lower Faro Daba beds. A wide range shapes and sizes is present, and a variety of raw materials was used, including non-local obsidian and chalcedonies, in addition to fine-grained basalt. These ranges are mirrored in the smaller sample of excavated points. Field photographs; relative sizes approximate.
Figure S14. Freshly eroded sets of conjoining MSA stone artifacts from the Faro Daba beds. A, B, and C are three sets of freshly exposed conjoining sets of flaking debris and cores in the lower Faro Daba beds. (B) shows the proximity of one dense concentration of flakes to burned clay associated with a burned tree stump (yellow arrow). The timing of such spatial co-occurrences is currently indeterminable.
Figure S15. Faro Daba bed MSA core tools.
Large elongate cobbles modified into cores, cobble-butt picks, and core axes are found alongside finely-made points in the MSA-bearing lower Faro Daba beds. These were made on basalts and ignimbrite. (A) shows a large core embedded in a calcium-carbonate concretion with fossil wood; (B-D) are matched views of surface artifacts.
Figure S16. Faro Daba bed vertebrate fossils.

A. A colobine monkey skeleton eroding from silts of the lower Faro Daba beds. View is to the E. in the SE catchment of the Kada Halibee tributary of the Awash River.
B. Cercopithecine cranium freshly eroded from the lower Faro Daba beds. View is to the E.
C. Colobine cranium.
D. Rhinoceros cranium in situ.
E. Concentration of microfaunal remains cemented in decomposing calcium carbonate and representing fossilized owl pellets.
F. Right femur of the partial Homo sapiens skeleton. Recovered parts of this large male skeleton indicated by grey shading.
Figure S17. Wallia bed LSA flaked and ground stone artifacts.
A, C, D, E. The HAL-A27 locality lithic assemblage includes a wide range of raw material types.
B. The HAL-A26 locality shows a blade based assemblage, mainly made on obsidian raw material.
Figure S18. Oulen Dorwa LSA lithic artifacts.
The Oulen Dorwa OUD-A1 surface assemblage is richly artifactual, with a wide range of raw materials, primarily chalcedony, jasper, and obsidian.
A. Part of a freshly eroded meter square to illustrate artifact density.
B. A selection of raw material and tool types arranged on a manuported lava cobble.
Figure S19. Oulen Dorwa LSA artifacts.
The Oulen Dorwa surface assemblage includes finished and in-process ostrich eggshell beads (G), and small cores (A,B), retouched blade (C), points (D,E) and grinding stones (F).
Figure S20. **Wallia beds, ostrich eggshell sampling.**

A. View to SE. Overview of the geological setting of the lower Wallia beds. Sample location is in the center of the photo.

B. Ostrich eggshell *in situ*.

C. Closeup of sampling the MA15-12a specimen that was used to obtain independent $^{230}$Th/U and radiocarbon results.
Figure S21: Selected EPMA glass element-element plots for felsic tephra reported as averages in Table S2 and SI Dataset S1F and as individual spot analyses in SI Dataset S1G. Note the range from compositionally homogeneous (unimodal; MA04-16, SEVT, DGST) to multi- and bi-modal (MA08-13, BRVT, MA15-08), to those displaying linear arrays through a compositional range; most notably the BRVT (also with “outlier” clusters) and to lesser extents the two OUD tuffs. Data plotted as measured and expressed as weight % with total Fe as Fe$^{+3}$. 
Figure S22: Selected EPMA glass element-element plots for felsic tephra reported as averages in Table S2 and SI Dataset S1F and as individual spot analyses in SI Dataset S1G. Plot A includes all tephra illustrating the total compositional range and bimodal mafic-felsic nature of the suite. Plots B and C include only the distinct mafic AFBT marker horizon and the mafic mode of the predominantly mafic bimodal MA15-08 unit. Data plotted as measured and expressed as weight % with total Fe as Fe$^{3+}$. 
Figure S23: Ostrich eggshell fragments of sample MA15-09 that were analyzed in this study.
A. Sample MA15-09-1 after cutting sample – smaller fragment was mounted in epoxy and analyzed by LA-ICP-MS; B. All other fragments of MA15-09 analyzed in this study. Scale bar is 1 cm.
Figure S24: $^{238}$U concentration and $^{232}$Th/$^{238}$U ratio profiles from LA-ICP-MS fragments of eggshell sample MA15-09.

Each sample is plotted as a mean (points) and $1\sigma$ standard deviation (shaded regions) of 5 lines starting at the outer surface (normalized distance = 0). Sub-samples for dating derive from sub-samples between a normalized distance of ~0.2 to 0.6, where $[^{238}\text{U}]$ is consistent and $^{232}\text{Th}/^{238}\text{U}$ ratios are lowest.
Figure S25: Sample recovery and all U-Th results for sample MA15-12.
A. Recovery of sample MA15-12 in the field, shown directly eroding out of the sediment and likely from a whole crushed egg. Relative scale from ice axe handle, pointing to the highest fragments and location of in situ material. B. $^{238}$U concentration and $^{232}$Th/$^{238}$U ratio profiles from LA-ICP-MS for fragments of eggshell sample MA15-12a (derived from lagged out fragments) and MA15-12b (in situ fragment). Laser ablation profiles are similar between samples, particularly internal to eggshell surfaces (between normalized distance ~0.2 to 0.6), indicating that brief exposure at the surface in an arid climate does not impact U retention. C. $^{230}$Th/U burial age plot of subsamples from the in situ fragment selected for paired $^{230}$Th/U and $^{14}$C dating; measured ages are shown as 2σ error ellipses. See comparison to $^{14}$C result in Table S1 and Figure 3.
Figure S26: $^{230}$Th/U evolution diagram of all sub-samples of eggshell fragments from sample MA15-09.
Initial $^{234}$U/$^{238}$U$_0$ activity ratios are plotted in blue lines and age isochrons (in ka) are plotted in black lines. Data ellipses (colored circles) encompass 2σ uncertainty. Samples whose outer shell subsample (O) plot to the right of the inner shell (I) are suitable for burial age calculations (MA15-09-1, -2, -5); samples with the opposite relation on the evolution diagram provide minimum ages only from the inner shell subsample (MA15-09-3, -4).
Figure S27: Selected trace elements used for obsidian source identification from Faro Daba beds, Halibee member.
Concentrations in parts per million (ppm). Confidence ellipses at 95%. Source “Type 10” after ref. (52) (red circles) characterizes three samples that match the ~106 ka obsidian from ref. (49).
Table S1: Summary of ages presented in this study. For analytical data, see SI Datasets S1A-D. Correlate: ¹Morgan et al.

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<th>Sample name</th>
<th>Sample Material</th>
<th>Sample Group ID</th>
<th>(^{14})C Age (cal ka BP; 95% C.I.)</th>
<th>(^{230})Th/U Burial Age or Minimum Age ((^{9})ka BP or ka ± 2σ)</th>
<th>(^{40})Ar/(^{39})Ar age (ka ± 2σ)</th>
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Table S2: Averages of EPMA major/minor element data expressed in weight % with total Fe as Fe$^{+3}$. S column is number of samples and N column is number of individual points used in averages. Full details of the EPMA data are contained in SI Datasets S1F-G and illustrated in the bivariate plots of Figs. S21 and S22.

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54. A. Negash, M. Steven Shackley, M. Alene, Source provenance of obsidian artifacts from


