Findings, context, and significance of combustion at the late Early Pleistocene Palaeolithic site of Cueva Negra del Estrecho del Río Quípar (Caravaca de la Cruz, Murcia, Spain)

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Abstract

Evidence of combustion inside a large rock-shelter in southeastern Spain is afforded by Palaeolithic finds excavated in a deep 0.8Ma (million years ago) closed sedimentary deposit. The principal findings are of abundant charred and calcined fragments of bone and numerous fragments of thermally altered chert; in both cases some fragments were excavated in conjoinable apposition, strongly suggesting in situ combustion and minimal post-depositional disturbance. Palaeotemperature estimates indicate heating in antiquity at 400-600ºC, determined on samples subjected to analyses by thermoluminescence (TL), Fourier Transform infrarred spectroscopy (FTIR), and electron spin resonance (ESR). Taphonomical investigations of bone discolouration, together with scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDX) of samples, indicate findings that are not typical of post-depositional mineral staining but are compatible with combustion. Macroscopical inspection of the sediment is suggestive of combustion and the demonstration in samples of hydroxyapatite is compatible with the degradation of bone, although micromorphological study of thin-sections of sediment has not yet provided incontrovertible evidence of combustion. The findings are discussed in the context of Early Palaeolithic fire and its likely significance for human evolution.

Introduction, context, and background

When are the first traces of combustion found in Palaeolithic sites in middle latitudes? What can be inferred from them about cognitive evolution in early *Homo*, especially in the context of dispersal in Eurasia. It has been argued that only after *c.*0.5-0.4Ma are there clear signs of fire at European sites (Roebroeks & Villa, 2011a), though probably this should be modified by a qualification that it seems to be the case where archaeological features point to control of the *heat* of a fire (*e.g.* hearths). Evidence from *c.*0.8Ma is presented of combustion in the absence of such features in a large rock-shelter in Murcia, Spain. It is proposed that cognitive versatility at least enabled advantage to be taken, opportunistically, of natural fires outside such that fire could be tended inside.

Evidence of fire at *c.*0.8Ma has been uncovered in the form of thermally-affected fragments of both bone and Palaeolithic chert artifacts, nodules, fragments, and spalls, excavated in a deeply-lying sedimentary deposit (at top of unit VI) inside the southeastern Spanish rock-shelter of Cueva Negra del Estrecho del Río Quípar which lies at 740m above sea level 10km south of Caravaca de la Cruz, Murcia (lat. 38.0375; long. -1.8846). Systematic excavation began in 1990 of the 5m depth of Pleistocene sediment lying on bed-rock. It has provided an abundant assemblage of small Palaeolithic artifacts, on chert and other raw materials (see tables in Walker *et al*. 2013, 140, Figs. 4, 5), which shows noteworthy consistency throughout the stratigraphical sequence and includes flakes removed by repetitive centripetal striking of small, sometimes discoidal, cores, several fragments and flakes with steeply retouched edges, and, notably, several keeled forms and pieces with fine points (*cf*. Debénath & Dibble 1994, 99 Figs. 7.22-7.27, 108-109 Figs. 8.29-8-37), as well as a bifacially-flaked (Acheulian) limestone hand-axe (Walker *et al*. 2013). There is no chert in the rock walls of the cave which are of Upper Miocene (Tortonian) biocalcarenite. Most chert at the site came from a slightly older Tortonian conglomerate bed that outcrops 0.8km east of the cave. Some chert that has been excavated may have been obtained up to 30km away from the site according to comparative trace-element analyses by laser-ablation inductively-coupled plasma mass-spectrometry (Zack *et al*. 2013).

Magnetostratigraphy indicates that all of the cave sediment falls within the Matuyama chron and is therefore >0.78Ma (Scott & Gibert 2009). Preliminary TL-dating (D.R., unpublished new data) of heated flint is in agreement with single-grain OSL analysis (J-L.S., unpublished new data) pointing to an antiquity of >0.5Ma though small sample number and signal saturation precludes further precision, respectively; previously, the less accurate method of multiple-grain OSL analysis had suggested 0.3-0.5Ma (Walker *et al*. 2006). Regrettably, $^{26}Al/^{10}Be$ analysis provided estimates far older than are plausible (Braucher, personal communication).

Biostratigraphical analysis of micro- and macro-mammalian remains is consistent with assigning the deposits to a period of <1->0.7Ma (Walker *et al*. 2013; Walker *et al*. submitted). Table 1 of excavated teeth shows that extinct Arvicolid Rodents of the Iberian late Early Pleistocene occur from the bottom to the top of the sedimentary sequence (*N.B.* the higher units II, III and IV have been excavated over a larger area to date than the deep units V and VI). Whereas a decade ago it was wondered whether these taxa might not be faunal atavisms at *c*.0.5Ma (Walker *et al*., 2006) the now larger sample is seen to be wholly comparable to late Early Pleistocene samples from sites at Atapuerca and in the Guadix-Orce Basin (Walker *et al*. submitted). Furthermore, revision of the large mammals by Dr. J. van der Made (ibidem) shows presence even in high units of late Early Pleistocene taxa (*e.g. Dama vallonnetensis*) and has corrected several misguided designations (*e.g*. *Stephanorhinus hemitoechus* which should be either *S. etruscus* or perhaps *S. hundsheimensis*) in early publications (*e.g.* Walker *et al*. 2004) that were unduly influenced by the pioneering consideration (Martínez-Andreu *et al*. 1989) of the deposits as "*Mousterian*" *i.e.* early Late Pleistocene (or, at most, late Middle Pleistocene).

Table 1

Taxa identified	Excavated lithostratigraphical units				
	unit II	unit III	unit IV	unit V	unit VI
Pliomys episcopalis	1	9			1
Mimomys savini	7	17	4	1	5
Microtus (Iberomys) huescarensis	21	101	42	1	26
Microtus (Stenocranius) gregaloides		3			
Microtus (Terricola) arvalidens		2			
Microtus(Allophaiomys/ Victoriamys) chalinei	40	45	49	8	13
Allocricetus bursae	9	3	2		
Apodemus cf. *sylvaticus*	13	13	16	1	3
Eliomys quercinus	1	1	2		
Crocidura sp.	8	39	27	1	13
Neomys sp.	2	11	4		
Erinaceus europaeus	43	29	6		
Oryctolagus cf. *giberti*	87	84	24	4	30
Prolagus calpensis	12	21	20		11
Numbers refer to finds identified for each species: for Arvicolidae the numbers refer to mandibular first molars; for other Rodentia and Insectivora they refer to maxillary and mandibular molars; for *Oryctolagus* they refer to mandibular third premolars; for *Prolagus* they refer to different molars of that taxon.					

The sedimentary sequence inside the rock-shelter comprises near-horizontally bedded, laminated, and cross-bedded, bands or lenses of fine (silt- and sand-size) particles of litharenite, micritic limestone, and quartz, with remarkably few coarser components. Macroscopical inspection and micromorphological analysis reveal several cycles of alluvial deposition (Angelucci *et al*. 2013) during a time when the cave mouth lay close to the river (before neotectonic activity and riverine incision in the Middle or Upper Pleistocene led to their present vertical separation of 40m) which encroached on it intermittently whenever the water level rose. Throughout the sequence there is ample evidence of Palaeolithic activity, no doubt during dry periods or seasons. Only one of these periods, in the upper part of the sedimentary sequence, was sufficient to have given rise to an incipient buried soil (with traces of bioturbation), the upper surface of which was partly truncated later on by an erosive surface (ibidem); otherwise significant stratigraphical discontinuity is conspicuous by its absence (*pace* Jiménez-Arenas *et al*. 2011) and the sediments remained undisturbed save for a few small pits, 1-2m deep, dug into them *c.*1940. Pollen from the sediments attests to mild, humid conditions with gallery woodland nearby (Carrión *et al*. 2003) and presence of waterfowl (Walker *et al*. 2004) is consistent with erstwhile presence of a lake close by. It now seems likely that the sediments were laid down during MIS21, towards the end of the Early Pleistocene (whereas publications before that of Scott & Gibert 2009 had interpreted them as Middle Pleistocene).

Excavation and macroscopical on-site consideration of thermally affected bone and chert

Findings that fire had affected both bone fragments and Palaeolithic chert came to light in 2011 during excavation in 1m^2 of sediment *c.*0.1m thick (at the top of unit VI, Walker et al., 2013), 4.5m beneath the surface of the sedimentary sequence 6-7m behind the cave mouth (Figure 1). Hitherto, lithics showing thermal alteration were unknown among >3,000 pieces excavated since 1990, and barely a score of possibly burnt bone fragments were scattered among >40,000 thousand faunal items uncovered between 1990 and 2011 of which barely 15% belong to larger mammals (>5kg live weight). From the topmost

0.1m of unit VI the 2011 excavation of $1m^2$ recovered >165 thermally-altered chert items, many being only 5.0-0.5mm in size owing to shattering by combustion, as well as 10 of limestone, 5 of quartzite, and (in 2012) one of radiolarite (a “scraper” that has edge retouch). There were also numerous charred fragments of bone and several white calcined fragments (Figure 2), including some which show conjoined lengthwise long-bone spalling (typical of circumferential shrinkage after thermal volatilization of organic components at 800-900ºC, cf. Uberlaker, 1999 [2004]: 35-38). Still undergoing analysis are many more burnt fragments of chert and bone that were excavated between 2012 and 2015 in a further 1.5 m^2 of the $5m^2$ area over which the surface of the thermally-altered sediment now has been identified between 5 and 7m behind the cave mouth, and the rear section indicates further inward extension below 4.5m of overburden.

One excavated thermally-altered chert piece is a nodule (Figure 3 Top) split open by heat (so-called “thermal shock”) with several minute razor-sharp splinters still in place together, and a split surface showing oval or circular shallow depressions typical of thermal alteration; such shallow pock-marks often accompany “pot-lid” fracture surfaces caused by heating chert or flint (Richter 2007; *cf*. Schön 2012, 104 Abb. 4) though we have not detected “pot-lid” surfaces, doubtless because most burnt chert excavated consists of small splinters, spalls or chips). In 2013 an artificially-struck flake (Figure 3 Bottom) was excavated with sharp conjoinable fragments still in place. Presence of splinters, spalls and fragments found in conjoinable apposition at excavation implies that following thermal alteration the finds hardly can have undergone displacement of more than a few centimetres. Unfortunately, discernment of any possible impingement owing perhaps to fluviatile activity must await several more excavation campaigns in order that the deep layer can be exposed outwards towards the cave mouth, because the abundance of Palaeolithic and palaeontological remains in all of the 4.5m of overlying sediments requires these to be excavated carefully and washed on 2mm mesh sieves (each 0.45m in diameter), thereby imposing a severe brake on the pace of excavation and hence the rate at which it is able to expose the deep layer.

Effects of combustion on chert are well-documented, albeit far from uniform owing to the variety and complexity of cherts: thus, whereas in some cherts temperatures of 250-300ºC can lead to changes in colour, lustre, or even cause heat damage or recrystallization of quartz, in other cherts higher temperatures up to 500ºC are needed for heat damage or recrystallization, depending on the chemical and crystalline properties of both the quartz itself and impurities present in the chert, such as calcium carbonate or even water (Clemente Conte 1997; Luedtke 1992). Hence combustion temperatures cannot be inferred with accuracy from visual inspection alone of the state of either burnt bone or burnt chert.

Supplementary analytical procedures and specific studies were called for. However, because at 700-800ºC chert tends to shatter into splinters, spalls and chips that are far too small for application of laboratory techniques, the larger burnt fragments to which these can be applied are unlikely to have been heated in antiquity to such temperatures, and therefore a possibility cannot be excluded that laboratory determinations of palaeotemperature may underestimate temperatures reached by an erstwhile fire. In this connexion, it should be borne in mind that our excavation of the deep sediment has yielded up very many splinters, spalls and chips, several of which afford clear microscopical indications of thermal alteration.

Analytical procedures, specific studies, and summary of principal conclusions

It was considered appropriate to supplement the observations made at excavation with some analytical procedures and specific studies, as follows.

(a) Thermoluminescence, TL, analysis of a fragment of excavated burnt chert was undertaken. The high temperature of the main TL-peak, the strong signal increase and the presence of a very well developed heating plateau is clear evidence of past heating above 400℃ (Figure 4).

(b) Fourier Transform infrared spectroscopy, FTIR, of an excavated bone fragment found characteristic sharpening of the phosphate absorptions at 1032-

1091 and hydroxyl bands that appear when bone mineral is heated above 400-450ºC (Figure 5), and some residual carbonate absorptions which show that calcination of the specimen was incomplete, suggesting that the temperature at which it had been heated was below 700-800ºC.

(c) Comparison was undertaken of the electron spin resonance spectra of three excavated bone fragments, one of which, without ostensive signs of previous burning, was heated as a control, whereas the other two appeared to have undergone burning in antiquity. This palaeothermometer methodology which had been applied at Swartkrans (Skinner *et al*. 2004) involves identification using ESR of any residual carbon fragments containing "soot" radicals, resulting from radiation damage to the bone matrix (causing thermal fragmentation of collagen mainly) in samples of bone in which oxidation of manganese has been attained at 400-500ºC. Modern bones have so much carbon in them that, on heating, the peak due to pure carbon (basically soot) is so wide that it conceals the other peaks. When fossil bones are available that have lost most, but not all, of their organic carbon, the ESR palaeothermometer methodology may be able to estimate temperatures to which heated fossil bones had been subjected at the site in antiquity. One of the two apparently burnt fragments from Cueva Negra afforded an organic radical signal in addition to that of manganese, indicating an ancient heating temperature of 400-450ºC (Figure 6 Centre). It should be mentioned that several other bone samples submitted from the site had too little carbon left to show any effects on heating, which is unsurprising because bones, being porous, both lose material over time and absorb material from the environment; moreover, organic carbon will break down during fossilization, aided by presence of bacteria, and the resulting fragments can be leached from the bone by ground water. All the bones were cleaned of surface dirt and then powdered to a grain size of approximately 0.1mm. ESR measurements were taken at room temperature on a JEOL RE1X spectrometer, with sample sizes of approximately 100mg.

Any fossil bone is likely to show a 'dating peak' due to radiation damage to the carbonate in the bone matrix. A bone heated in antiquity will show this, superimposed on other spectral features. It should be noted that bones cannot be dated from this peak because the environmental radiation dose, especially internal to the bone, is incalculable. Radioisotopes, largely uranium, may have leached in and out of the bone during its burial history. Whereas this dating signal is extraordinarily stable, it does decrease on heating to about 300ºC for several hours. Thus the pattern sought on artificial heating of a fossil bone is the disappearance of the "dating signal" and its replacement by structure attributable to carbon fragments, with a central peak due to carbon (soot) radicals. At about 400-500ºC peaks due to manganese appear, representing oxidation of manganese by heat. By 600ºC essentially everything disappears except perhaps some residual carbon radical intensity: this pattern is shown in Figure 6 Top.

(d) Thermal discolouration of excavated bone is supported by taphonomical analysis combined with scanning electron microscopy and energy-dispersive spectroscopy that enabled sporadical isolated deposits on bone surfaces of oxides of manganese or iron to be distinguished from more widespread discolouration that can be attributed mainly to thermal alteration (Figure 7). A statistically significant contrast was found between the proportion of bone fragments of small mammals (<5kg live weight) showing noteworthy change in colour, consistent with exposure to heat, as against those showing less change, when samples from the deeply-lying sediment at the top of unit VI containing burnt chert and bone were compared with samples from sediment (unit V) immediately above and even deeper unit VI sediment below. In a taphonomical analysis of almost 2,300 fragments of small mammal bone, identified as such among *c*.4,400 microfaunal fragments from those sedimentary units (see tables and taphonomical information in Rhodes *et al*., submitted), 25% showed evidence of thermal alteration as discolouration of the bone surface; of these, ~70% showed either light coloured isolated spots (Category 1) or dark gradient discolouration (Cat 2), whereas ~30% exhibited either carbonation (Cat 3), or calcination to grey-white (Cat 4) or pure white colour (Cat 5), and, moreover, these specimens were more fragile than those in Categories 1 and 2, with more cracking and less root etching (ibidem; Rhodes 2014; Rhodes *et al*., 2014). The deeply-lying sediment provided approximately 95% of all of those small mammal bone specimens inspected from the site which corresponded to Categories 3-5 (Figure 8); furthermore, in that sediment bones from different anatomical regions were affected alike, which is compatible with in situ exposure to high temperature. Although excavation of the deep sediment recovered fragments of large mammals (over 80, of which 25% showed signs of thermal alteration) and tortoise, the taphonomical study was devised with the particular methodological purpose to compare and contrast remains of small mammals from different parts of the site and to consider their source, which is most likely to have been predation by owls, lynxes or foxes, doubtless during periods of absence by humans who perhaps might have burnt unhealthy rubbish on their return and maybe roasted foodstuff also.

(e) Detailed examination of the deeply-lying sediment containing burnt chert and bone (Figure 9) reported that "distinct layers were observed of materials resembling ash, sometimes resting on reddened belts" (Angelucci *et al*. 2013), although incontrovertible high-resolution microscopical evidence of combustion, such as in situ reddening, presence of wood ash, or charcoal fragments, could not be detected in the thin sections on which sediment micromorphology was undertaken.

(f) Chemical and mineral investigation compared the deep reddened sediment with sediment above and below by thermogravimetrical analysis with mass spectrometry, granulometry (of the <2mm fraction) using laser diffraction, and X-ray fluorescence, XRF, and X-ray diffraction, XRD, studies. Hydroxyapatite was present (2.5%) in the reddened sediment and (1%) in the sediment immediately below it, which is compatible with degradation of bone; and it was found also (0.2%) in an excavated burnt chert fragment.

Samples: A sediment sample was analyzed from reddish layer *TA-U6* and another from the underlying layer *TA-U7*. They were compared to a sample from overlying sediment. A burnt chert fragment was analyzed also.

Principal findings: Sediment samples from *TA-U6* and *TA-U7* contained $CaCO_3$ inclusions as microscopical clumps and fine powder. Organic content: 1.45-1.8%. Organic CO_2: 20-21.5%. *TA-U6* and *TA-U7* consist mainly (~90.5%) of $CaCO_3$, and (~2%) hydroxyapatite $Ca_{10}(PO_4)_6(OH)_2$ (*TA-U6* 2.5%P; *TA-U7* 1%P) which is compatible with degradation products of bone.

Elements present at >1%: O (48%), Ca (20-24%), Si (10-12%), C (6%), Al (4-4.5%), Fe (2-2.5%), P (1-2.5%), K (1.6-1.8%), Mg (1%).

Elements present at <1%: Na, S, Cl, Ti, V, Cr, Mn, Ni, Cu, Zn, Br, Rb, Y, Zr, Ba.

Mineral species identified (%ages*)*:

	calcite	quartz	muscovite	(?)sanidine(?)	clinochlore	hydroxyapatite	illite	haematite
overlying sediment	71%	22.3%	2.1%	0	2.2%	1.2%	0.7%	0.3%
TA-U6 sample	57.8%	27.6%	7.6%	1.9%	1.9%	2.2%	0.5%	0.5%
TA-U7 sample	50.6%	29.4%	7.8%	6.8%*	2.8%	1.9%	0.4%	0.2%

(g) Microscopy reveals that grey hues predominate on surfaces of chert excavated in the sediment affected by combustion. Vertical and oblique fractures are frequent (giving rise to tiny spalls), as are conspicuous oval or circular shallow depressions caused by thermal alteration of chert surfaces. Cracks and reticulate crazing are widespread, particularly on surfaces showing rubefaction. White opaque or translucid patination is frequent, as also is shiny thermal lustre that can seem slightly "greasy". Both macroscopical and microscopical observations at Cueva Negra are in line with both experimental findings and others from archaeological sites with evidence of combustion (cf. Clemente Conte 1997; Luedtke 1992) Photomicrographs show thermal alteration and surface patination of thermally-altered Cueva Negra chert specimens (Figure 10).

Early fire and mid-Quaternary human evolution

Given that the results appear to be consistent with a plausible inference of presence of combustion *c*.0.8Ma at a European Palaeolithic cave site, it is fitting to consider the possible significance of such presence, whilst paying due regard to the necessary caution with which claims of early Palaeolithic fire must be treated always. It is therefore appropriate first to draw attention to some problems of interpretation at Cueva Negra and other early sites.

Evidence of fire from ancient cave sites often seems quite convincing (James 1989) but warning bells are sounded by much-discussed difficulties of interpretation at some well-known sites. For that reason, inconclusive comments in an earlier article (Walker *et al*. 2006) were limited with prudence to cursory mention of small bones with "signs of burning" excavated in higher sediments at Cueva Negra del Estrecho del Río Quípar, and, in relation to a 1m^2 test pit dug in 2004, to "loose sediment flecked with carbon (unit V = layer and spits 5a through 5g). It passes into unit VI, which is half-a-metre thick, and is distinguished by zones of very dark, loose soil, suggestive of burning (unit VI = layer and spits 6a through 6i)" though post-depositional decalcification could be responsible by diagenesis. The small test pit reached bed-rock in 2004 but its vertical profiles and its aforementioned sediment, lacking burnt chert and burnt bone, do not reproduce clearly the sedimentary sequence of the adjacent 2m^2 that have provided incontrovertibly burnt fragments of bone and chert. In these squares the bed-rock slopes slightly downwards towards the test-pit square, to which drainage seems to take place, and in which its aforementioned deeply-lying sediment (of units V and VI = layers 5 and 6 = Complex 3-2 of Angelucci *et al*. 2013) contains organic residues, doubtless derived from the adjacent area, albeit lacking both pollen and microscopical traces of charcoal. Six more excavation campaigns were to pass before the adjacent area could be exposed, because of the time-consuming methodological requirement to remove overlying sediments and wash them over 2mm mesh sieves. The prudent caution expressed in 2006 was inspired by a possibility that burnt material might have been blown into the cave from bush fires sweeping past the cave mouth. Such an interpretation may account indeed for traces of burnt material reported from 1.2Ma sediments at the Sima del Elefante (in the Sierra de Atapuerca in northern Spain), as follows: "L'abondance de micro-charbons associés à des composés organo-minéraux exogènes atteste de la récurrence d'incendies naturels dont le déclenchement semble être lié à des évènements exceptionnels d'origine cosmique" (Carbonell *et al*. 2010).

Roebroeks & Villa (2011b) wrote, "…heated flints in a cave site are unlikely to be the result of natural wild fires and may be considered a reliable indicator of anthropogenic fire if (i) there is no evidence of reworking of sediments, slope wash, or debris flow entering the cave; (ii) the excavator noted a localized concentration of heated flint and bones; and (iii) only a small proportion of heated flint occurs at the site. This combination of evidence suggests a good probability of localized fire." It is a combination present at Cueva Negra del Estrecho del Río Quípar.

"Anthropogenic fire" carries an unfortunate overtone tending to direct attention to how fire was *generated* in the Palaeolithic. Although at the Murcian cave hot sparks given off by a wood hand-drill or when pyrite is struck with chert could have ignited carefully prepared tinder, this begs a question of how cognitive appreciation arose of a useful possibility of making fire that were to be afforded by bringing together two different kinds of technical behaviour, namely, selecting and preparing different kinds of wood (*e.g.* mullein and clematis) or suitable stones for striking sparks and selecting and preparing suitable tinder for sparks to set alight (whereas pyrite occurs in some rock strata near Caravaca none has been excavated in the cave). Prerequisites may have included advantages gained opportunistically from *tending* fire, itself a likely consequence of a *reduction* in innate fear of fire (induced by burns to the skin). Evidence of fire inside an early Palaeolithic cave carries very important implications for understanding cognitive evolution. The argument is set out briefly as follows.

First, it is unlikely that sparks from a bush fire outside, perhaps caused by lightning, could set alight a chance accumulation of brushwood inside such as to bring about a roaring blaze within, causing high temperatures. Moreover, the river and its swamp lay in front of the cave, where gallery woodland flourished in a damp environment, not a dry one. Furthermore, quite likely the cave roof then extended outwards further than nowadays (because it may well have undergone some erosive reduction); if so, then the signs of fire we have uncovered would have been still further back inside than at 5-7m today. Maybe smouldering brands or embers left behind by bush fires nearby were carried inside so that fire could be *tended* where rain or wind would not put it out. No fire-pit or hearth stones have been found, therefore evidence is lacking of ability to control the *heat* of a tended fire. Nevertheless, from the standpoint of cognitive evolution in later Early Pleistocene *Homo* it is worth contemplating the likelihood that the denizens of the cave would have been less afraid of fire outside than were animals they saw fleeing before it. That could have led them to meddle with fire in order to drive animals towards natural death-traps, such as swamps, where they could be dismembered.

A tended fire in a cave could serve several purposes at the same time, such as providing warmth, roasting food, and deterring approach by fierce animals. There are compelling physiological arguments for considering that cooking may have played a significant part in human evolution from at least 1.5Ma. Wrangham (2009: 88-90) wrote that archaeological “hints from the Lower Paleolithic tell us only that… the control of fire was a possibility, not a certainty” and “The inability of the archaeological evidence to tell us when humans first controlled fire directs us to biology… At some time our ancestors’ anatomy changed to accommodate a cooked diet”. With regard to the evolution of human anatomy, following attainment *c*.1.6Ma of more-or-less modern stature, it is a plausible conjecture that subsequently widespread noteworthy increases in cerebral volume in *Homo erectus* and *H. heidelbergensis*, and, eventually, *H. neanderthalensis* and *H. sapiens*, were enabled by the enhanced digestion and absorption of nutrients which cooking afforded to pregnant women, lactating mothers, and growing infants and children (*cf*. Fonseca-Azevedo & Herculano-Houzel 2012). Outcomes of cognitive evolution surely are reflected in the extensive material record of Palaeolithic technologies (including fire) from Middle and Late Pleistocene times, to which can be added the late Early Pleistocene at Cueva Negra del Estrecho del Río Quípar. The bifacial flaking of its hand-axe is matched in Mediterranean Spain by that of a cleaver excavated in an even earlier deposit at Barranc de la Boella (Vallverdú *et al*. 2014), and an assemblage of small artifacts at Vallparadís (Martínez *et al*. 2010) shares several features with those from our Murcian site.

Because the Cueva Negra del Estrecho del Río Quípar excavation has provided both an Acheulian bifacially-flaked hand-axe and abundant flakes made by repetitive centripetal flaking of small, sometimes discoidal, cores for producing retouched small tools, the site exemplifies the ability of those who frequented it to select and carry out two different self-determining or self-constraining Palaeolithic chains of sequential behavioural activities (Walker 2009; Walker *et al*. 2013; Walker *et al*. submitted; Zack *et al*. 2013). Survival of early humans in middle latitudes laid heavy evolutionary demands on their cognitive versatility and manual dexterity, which are attested to by the diversity of their Cueva Negra Palaeolithic artifacts, so it is unsurprising they may have tended fire. More than one palaeospecies of late Early Pleistocene *Homo* may have engaged, opportunistically, in behaviour with fire. The skillfullness manifested by Acheulian stone tools at several sites with traces of fire implies evolution of cognitive versatility sufficient for such behaviour.

At the threshold of Europe, fire at the Acheulian site of Gesher Benot Ya'akov in Israel (Alperson-Afil & Goren-Inbar 2010; Goren-Inbar *et al.* 2004) corresponds to an early time within the Brunhes chron that commenced *c.*0.78Ma. B**a**rely 140km south-west of Cueva Negra magnetostratigraphy has identified the "Matuyama/Brunhes boundary only a few metres below the fossil/tool-bearing levels" that belong to *c.*0.76Ma (Scott & Gibert 2009) at the open site of Solana del Zamborino where excavation uncovered five stones around a small area containing burnt bone and "carbón", and also from the site there are two bifacially-flaked hand-axes and small retouched artifacts (Botella *et al*. 1976).

Fire was present at the South African Wonderwerk Cave with Acheulian artifacts during the Jaramillo subchron *c*.1.07-0.99Ma (Berna *et al*. 2012). Although other late Early Pleistocene sites with evidence of combustion are known in Africa from *c*.1.5Ma onwards (Gowlett *et al*. 1981; Rowlett 2000), most are open sites where bush-fires might have been responsible (Berna *et al*. 2012, who do not exempt Gesher Benot Ya'akov in that regard, contra Alperson-Afil 2012; Richter *et al*. 2011). At Swartkrans cave, the evidence for combustion (Skinner *et al*. 2004) from Member 3, which contains Acheulian artifacts, has been subject to uncertainty about the integrity and age of the member with dates ranging from 1.4 to 0.6 Ma (Berna *et al.* 2012; Herries *et al*. 2009) although a $^{26}Al/^{10}Be$ estimate from it of 0.96±0.09Ma (Gibbon et al. 2014) and another by U-Pb of 0.83±21Ma (Balter et al. 2008) seem plausible.

Acheulian artifacts are unknown at Zhoukoudian Locality 1 where six $^{26}Al/^{10}Be$ estimates of *c.*0.77±0.08Ma (Shen *et al*. 2009) come from levels 7-10 that also have 17 estimates ranging from 0.35 to 0.55Ma obtained by $^{230}Th/^{234}U$, TL, ESR and fission-track methods (Goldberg *et al.* 2001). Layer 8 is correlated with the laterally separate "quartz horizon 2" where "ash" was reported (Black *et al*., 1933; Pei, 1932; Teilhard & Pei, 1932). Chemical signs of combustion exist in later levels 4-6 (Zhong *et al*. 2013) notwithstanding micromorphological demonstration of post-depositional alteration of their sediments by diagenesis. This also took place in the deeper layers 7-10, leading to mistaken identification of "ash" features; burnt bone found slightly above them is incompatible with situ combustion (Goldberg *et al*. 2001).

In England, excavation at the site with Acheulian artifacts of Beeches Pit *c*.0.42-0.37Ma uncovered features interpreted as hearths "indicating controlled fire-use" (Gowlett *et al*. 2005) and "occurrence of bones burned to grey or white… implies more intense combustion than is usual for a natural fire, which often results in only partial and superficial burning (David, 1990)" (Preece *et al*. 2006). At *c*.0.3Ma hearth features at Qesem Cave in Israel (Karkanas *et al*. 2007; Shahack-Gross *et al*. 2014) and *c*.0.78Ma at Gesher Benot Ya'aqov (Alperson-Afil 2012; Alperson-Afil & Goren-Inbar 2010; Goren-Inbar *et al*. 2004; Richter *et al*. 2011) are plausible early instances of repeated use of fire in controlled and (at Qesem Cave) restricted spaces.

From the standpoint of mid-Quaternary human evolution, it is intriguing that in Africa, Israel, and now at Cueva Negra del Estrecho del Río Quípar, convincing signs of combustion occur at several sites where Palaeolithic assemblages include bifacially-flaked stone artifacts. A tempting surmise is that the conjunction reflects the cognitive versatility and technical ability of early humans that undoubtedly played a part in enabling their dispersal into middle latitudes.

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Figure 1

The Cueva Negra del Estrecho del Río Quípar excavation. The deeply-lying deposit containing burnt remains is the deepest part of the excavation in the two left-hand views, and close-up views are shown on the right.

Figure 2

Excavated thermally altered bone fragments. The left-hand photograph shows longitudinal spalling (see text).

Figure 3

Top: Excavated thermally-altered chert nodule. On the right is shown the rippled surface of a large fragment of the same nodule that covered the fragments on the left which include several small splinters (the difference in colours between the two images is more apparent than real and owes to different lighting effects).

Bottom: Excavated Palaeolithic flint flake that was found in three fragments in situ. Red part of scale = 25mm.

Figure 4

Thermoluminescence (TL) analysis of excavated burnt chert. The constant ratio (heating plateau) of natural/(natural+dose) TL signals indicates heating above 400ºC.

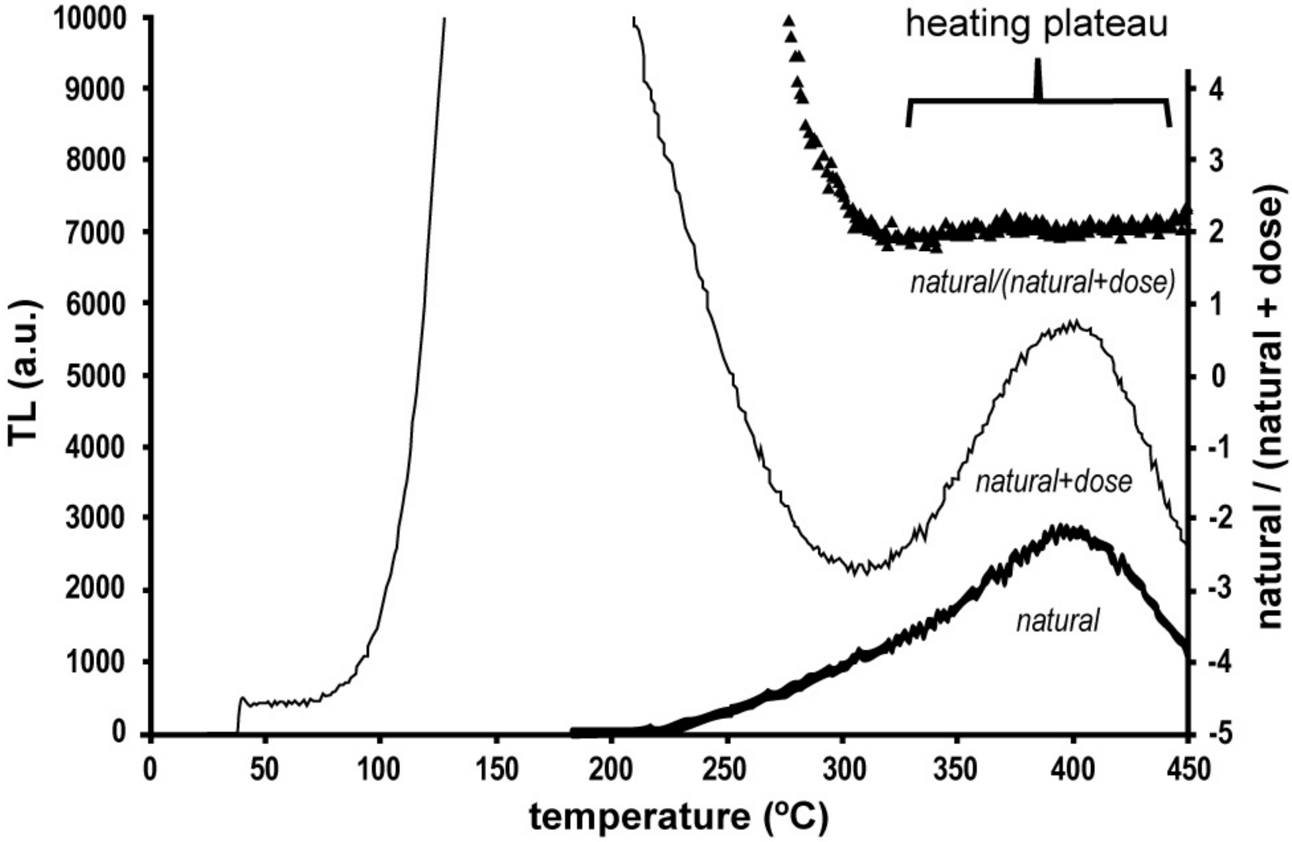


Figure 5

Fourier Transform infrared (FTIR) spectroscopical analysis of excavated burnt bone. The spectra of the sample (shown in the photograph) demonstrate characteristic sharpening of the phosphate absoprtions at 1032-1091 and hydroxyl bands that appear when bone mineral is heated above 400-450ºC, though residual carbonate absoprtions indicate an incomplete calcination pocess implying a temperature under 700-800ºC.

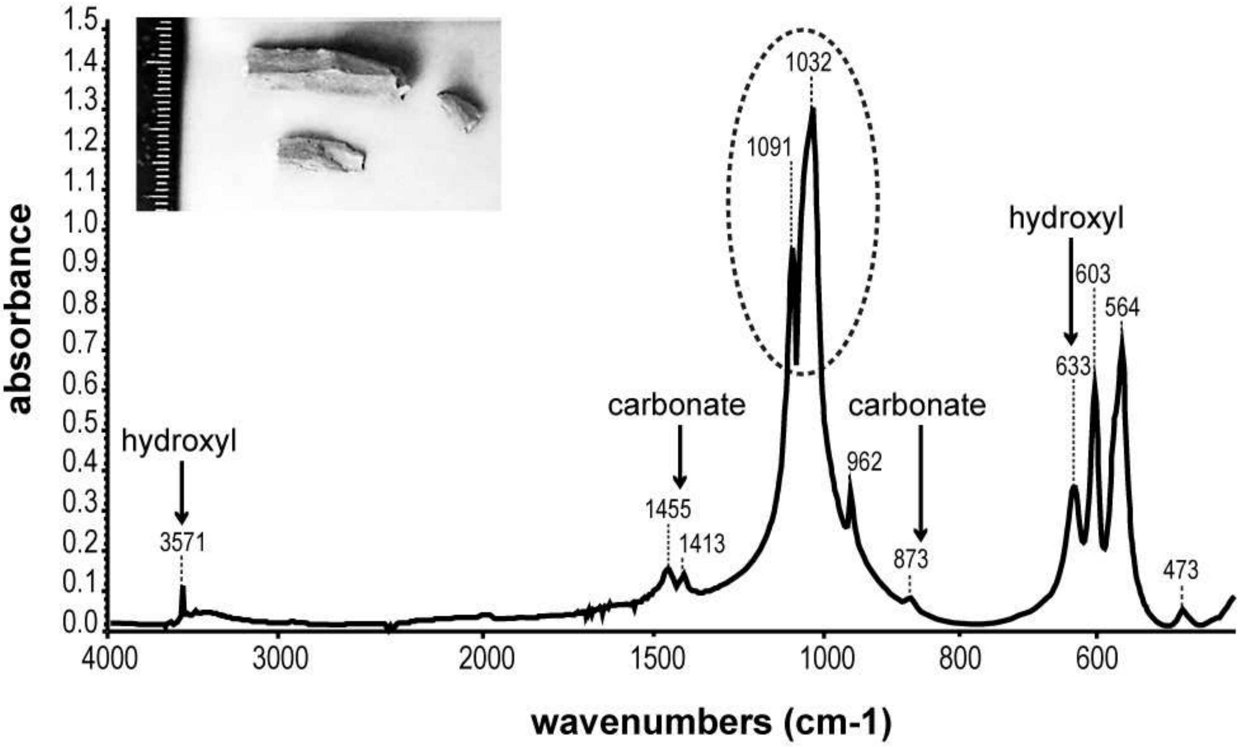


Figure 6

Electron spin resonance (ESR) analyses of bone.

Note: Signal intensity is in arbitrary units; the spectra curves within each graph are drawn to the same scale but are separated vertically for ease of interpretation by readers.

Top: Fossil bone from Cueva Negra, used as control.

1 unheated, showing “dating peak”;

2 heated to 300ºC, dating peak gone and replaced by carbon radical peak;

3 heated to 450ºC, peaks on the wings due to Mn, small peak due to ethyl-type radicals, can be seen on low field side, other peaks concealed by wide carbon radical peak;

4 heated to 600ºC, most structure gone.

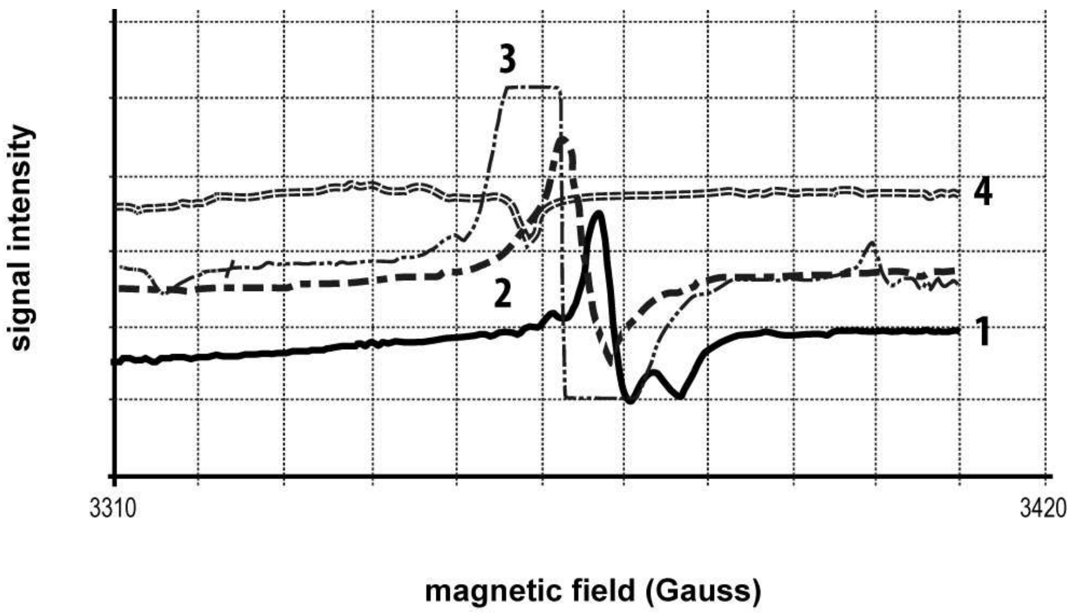
Centre: Two fragments of a fossil bone from Cueva Negra, apparently heated in antiquity; both showed Mn peaks as well as organic radicals. Best estimate of heating temperature: 400-450ºC.

1 fragment a, small Mn and ethyl-type peaks;

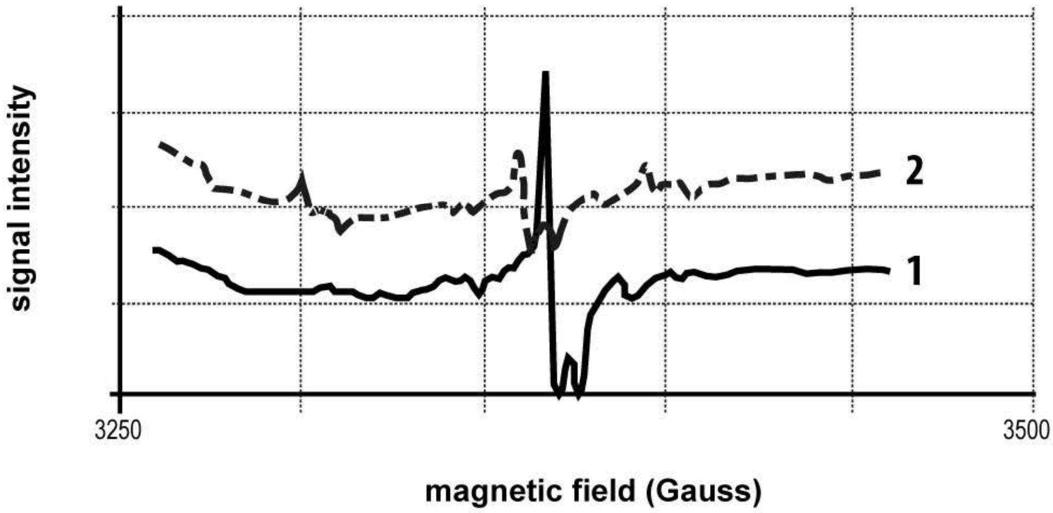
2 fragment b, the spectra show clear examples of organic radicals (the 3350 G peak is the expected doublet; the small 3352 peak represents methyl-type radicals) as well as Mn.

Bottom: A fragment of fossil bone from Cueva Negra, described as “calcined”.

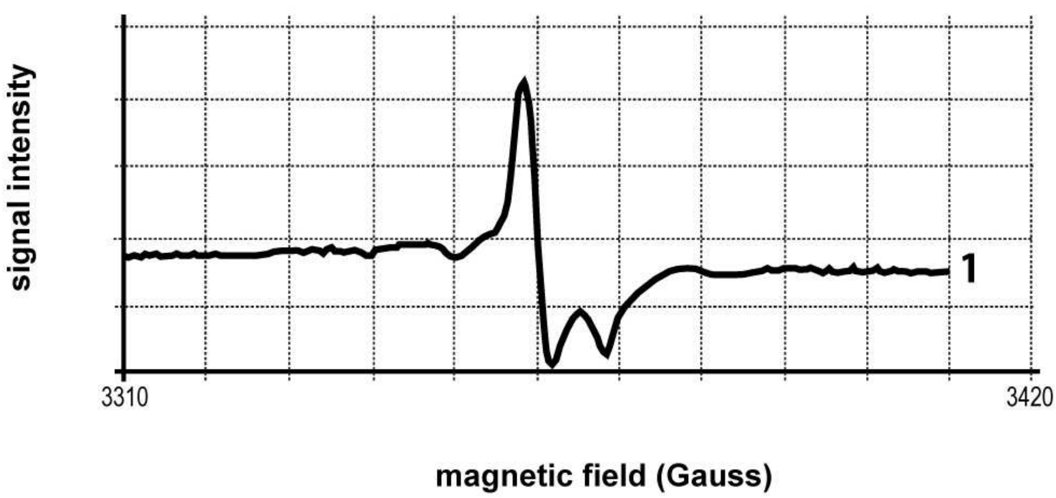
1 The ESR spectrum is not helpful as it shows no significant radical peaks but the “dating peak” would probably cover the carbon radical; there may be some structure at the field positions around 3350 and 3380 G, which would suggest heating to <600ºC. In other words, notwithstanding weak indications of heating to the same temperature as the two fragments of above, the bone could have experienced a temperature that has destroyed all soot and manganese signals; that would imply heating to <600ºC.



signal intensity
3
4
2
1
3310
3420
magnetic field (Gauss)



signal intensity
2
1
3250
3500
magnetic field (Gauss)



signal intensity
1
3310
3420
magnetic field (Gauss)

Figure 7 Scanning electron microscopy and energy dispersive spectroscopy were undertaken and the plates highlight the contrast between a charred rodent femur (Left) and a heavily oxide-stained rodent metapodia (Right). Note the heavy Mn and Fe deposits on the metapodia, which follow the pattern of discolouration recognizable macroscopically (with little deposit on non-discoloured surfaces). Conversely, the femur, visibly identified as charred, shows minimal Mn adherence and Fe deposit which do not follow the pattern of discolouration on the bone surface (whereas oxide-staining of bone usually presents as deposits over noteworthy areas of bone surface). Proportions of Mn and Fe are low on both oxide-stained and burnt specimens throughout sample, but lower proportions occur on those identified as burnt based on discolouration morphology.

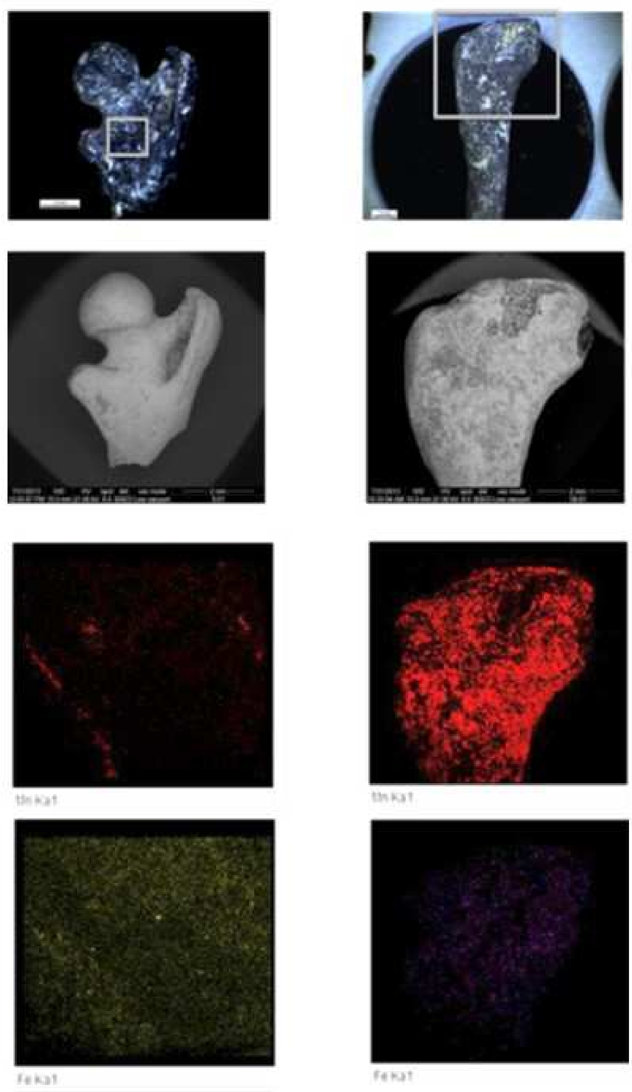


Figure 8 In a comparative study of ≈2,300 small-mammal bone fragments excavated in 2011 from (a) a 0.3m thickness of sediment covering the "ash" layer, (b) the 0.1m-thick "ash" layer, and (c) and a 0.15m depth of sediment underneath the "ash" layer, 25% showed evidence of thermal alteration as discolouration of the bone surface. 5 categories of discolouration were defined: most specimens (>70%) were Category 1 (light coloured isolated spots) or Cat. 2 (dark gradient discolouration); the remaining ~30% showed either carbonation (Cat. 3) or calcination to grey-white (Cat. 4) or pure white (Cat. 5) colour; they were more fragile than those in Cat. 1 and 2, with more cracking and less root etching. Approximately 95% of all charred and calcined bone came from the deeply-lying "ash" layer. Statistically significant differences in proportion of heavily burnt bone between different layers excavated at the site suggest exposure to fire of those from the "ash" layer ($\chi^2 = 169.2$; $p < 0.001$).

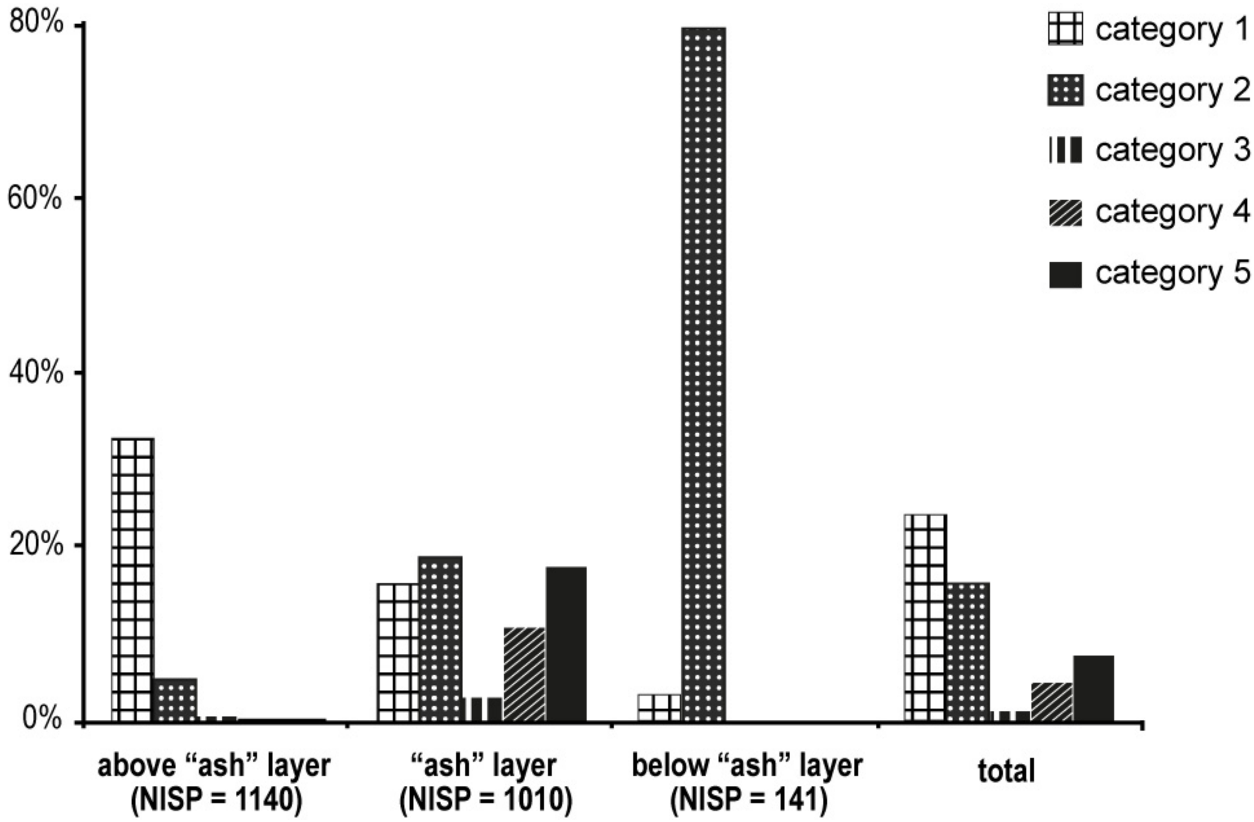


Figure 9

Photographs and stratigraphy of deeply-lying sedimentary layers with burnt remains in metre-square C2d during excavation in 2012. Adapted from the Supplementary Information published in Angelucci *et al*., 2013 (http://dx.doi.org/10.1016/j.quascirev.2013.09.009)

TA = possible thermal alteration. Numbered boxes refer to samples taken for sediment micromorphology.

CN-TA-U1: laminated silt with very fine sand, overconsolidated, 7.5YR 4.5/6, ca. 8 cm – corresponds to unit CN-TA-U1; lower boundary sharp (possible minor discontinuity);

CN-TA-U2: thin, dark (10YR 3/3), organic (and phosphatic?) layer, almost loose, with possible excremental features, ca. 2 cm thick, rather discontinuous laterally;

CN-TA-U3 ("yellowish ash"): silt with limestone fragments, rather disorganized; 2.5Y 6/4, with heterogeneous (1-15 cm) fragments of limestone, often shattered (in situ) by physical weathering; massive, locally soft and locally irregularly cemented by calcium carbonate and probably phosphate, giving a sort of nodular structure; lower boundary clear;

CN-TA-U4 ("whitish ash"): layer of sandy silt, irregular, 2.5Y 7/2.5 (dry) and 2.5Y 6/3 (humid); massive, locally poorly laminated (north section); partially (and irregularly) cemented by carbonate concretions in south section; partially reworked, it contains rare very small charcoal fragments and occasional fine (mm) angular fragments of limestone; lower boundary clear;

CN-TA-U5 ("greyish ash"): layer of silt, 2.5Y 6/4 (dry) and 2.5Y 5/4 (humid); it contains charcoal and limestone fragments as in unit U4; lower boundary clear;

CN-TA-U6: reddened layer, mostly silt, 7.5YR 4/6 (humid), irregularly cemented;

CN-TA-U7: thin discontinuous organic layer, silt, 10YR 3/3, with poorly developed prismatic structure, common organic matter finely dispersed in matrix, no stones, lower boundary clear;

CN-TA-U8: clayey silt with scarce stones (locally common: limestone fragments 0.5-15 cm with larger stones sometimes horizontal and small limestone fragments slightly weathered); 7.5YR 5.5/6; low porosity with small channels sometimes containing partly calcified rootlets; not cemented; lower boundary gradual towards very fine sand layer with scarce limestone fragments, 10YR 5/6, resting on bedrock.

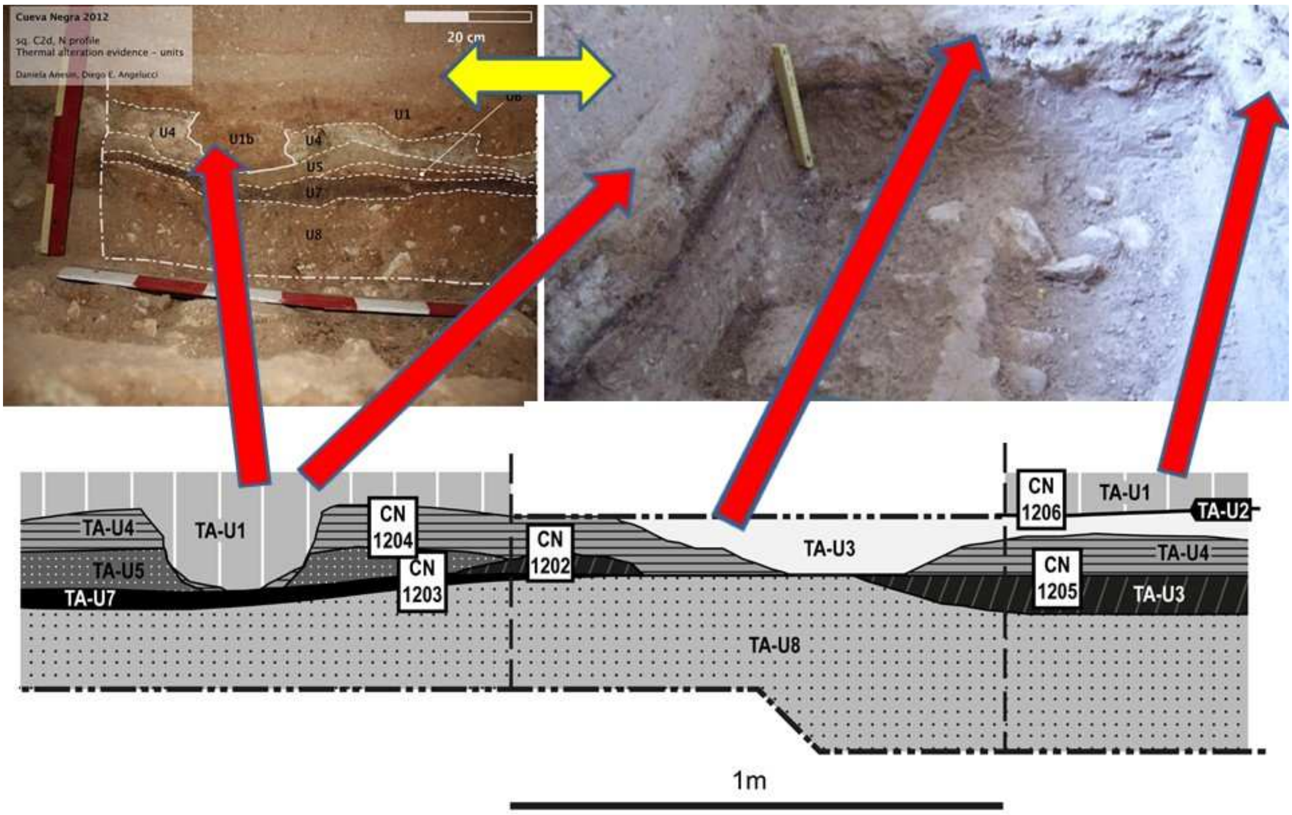


Figure 10

Microscopy of chert fragments shows thermal alteration (Top) and thermal patination (Bottom)