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

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*Control of fire was a hallmark of developing human cognition and an essential technology for the colonisation of cooler latitudes. In Europe, the earliest evidence comes from recent work at the site of Cueva Negra del Estrecho del Río Quípar in south-eastern Spain. Charred and calcined bone and thermally altered chert were*

*recovered from a deep 0.8-million-year-old sedimentary deposit. A combination of analyses indicated that these had been heated to 400–600ºC, compatible with burning. Inspection of the sediment and hydroxyapatite also suggests combustion and degradation of the bone. The results provide new insight into Early Palaeolithic use of fire and its significance for human evolution.*

*Keywords*: Early Pleistocene, Palaeolithic, Acheulean, combustion, cognitive evolution

*Supplementary material*: Figures 6–9 with more detailed captions.

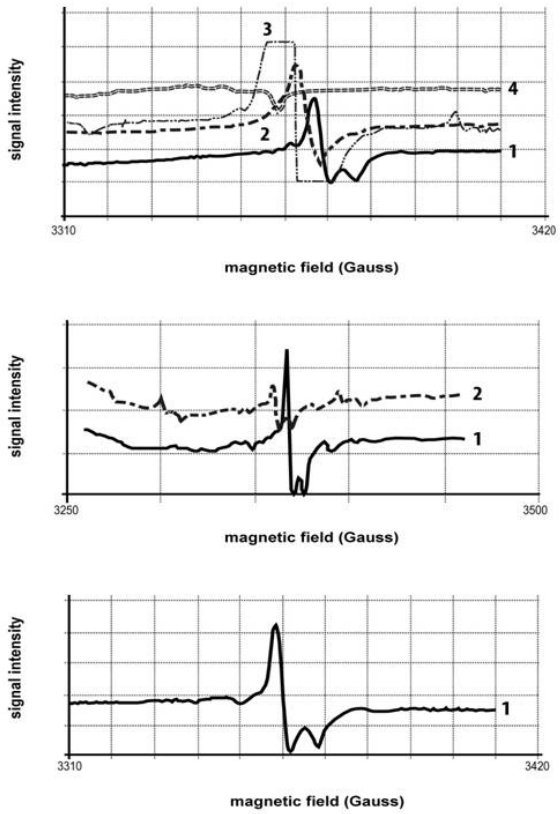


Figure 6. Electron spin resonance (ESR) analyses of bone.

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

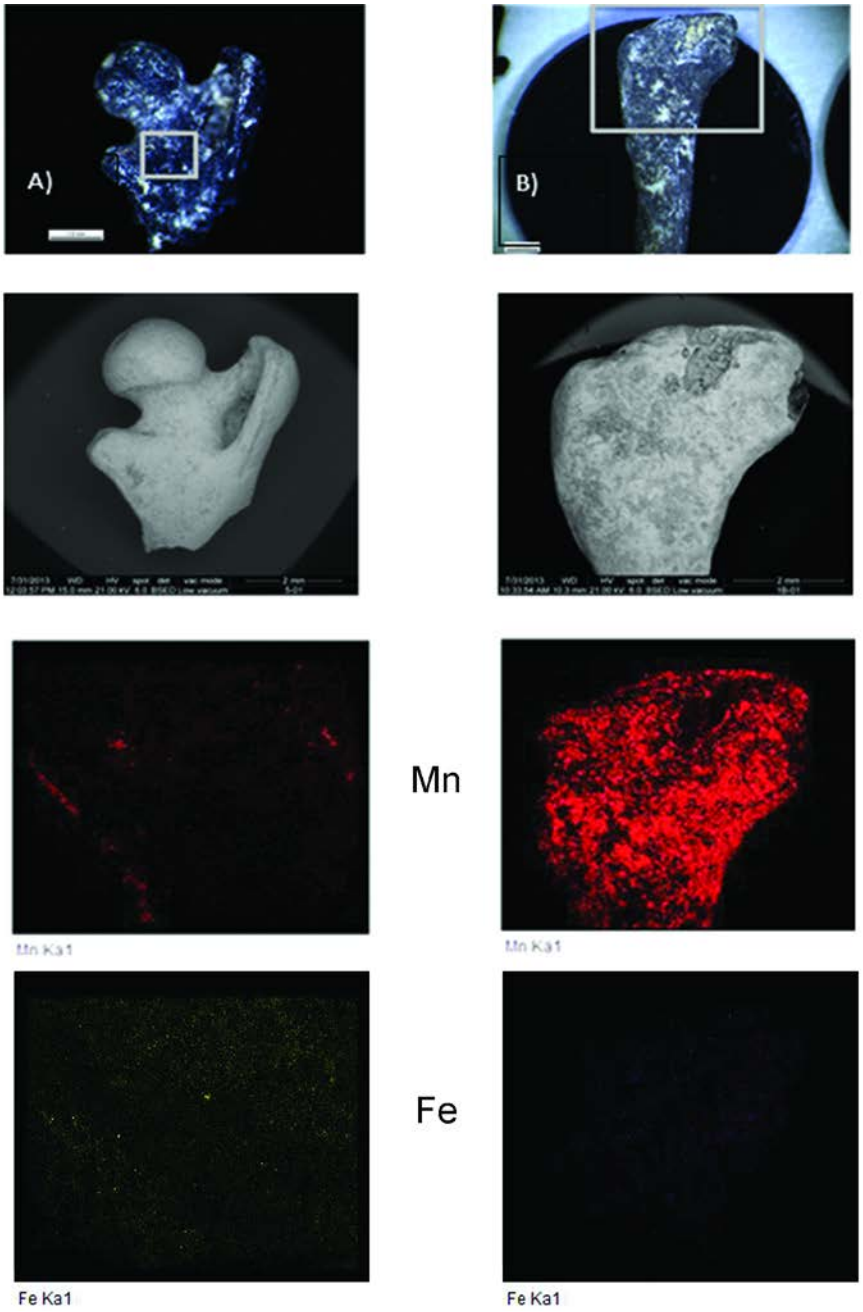


Figure 7. Scanning electron microscopy and energy dispersive spectroscopy were undertaken and the plates highlight the contrast between a charred rodent femur (Left) and a heavily oxide-stained rodent metapodia (Right). Note the heavy Mn and Fe deposits on the metapodia, which follow the pattern of discolouration 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.

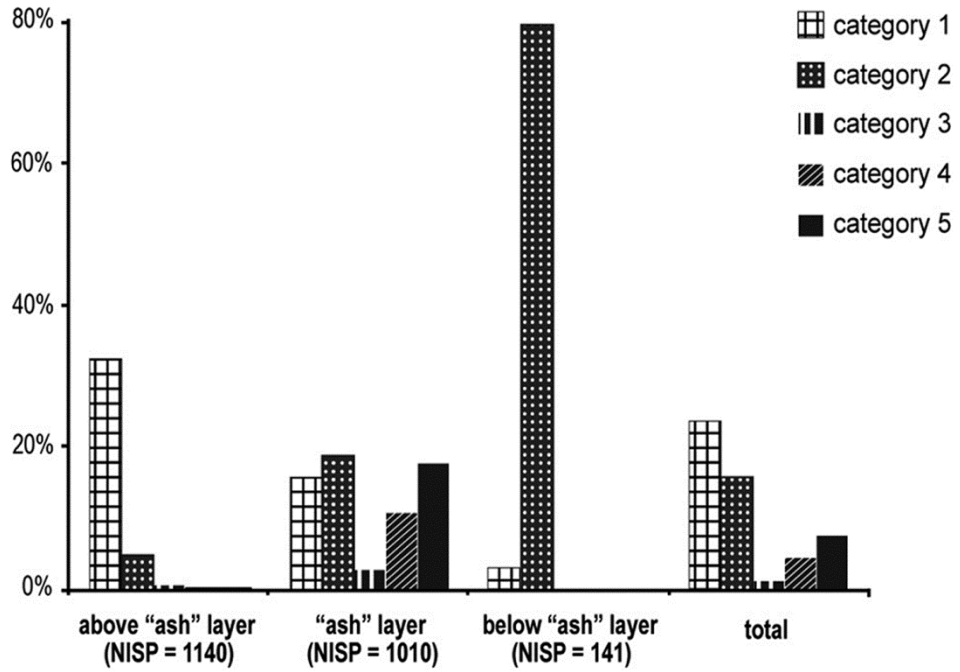


Figure 8. In a comparative study of ≈2,300 small-mammal bone fragments excavated in 2011 from (a) a 0.3m thickness of sediment covering the "ash" layer, (b) the 0.1m-thick "ash" layer, and (c) and a 0.15m depth of sediment underneath the "ash" layer, 25% showed evidence of thermal alteration as discolouration 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 ($\chi^2 = 169.2$; $p < 0.001$).

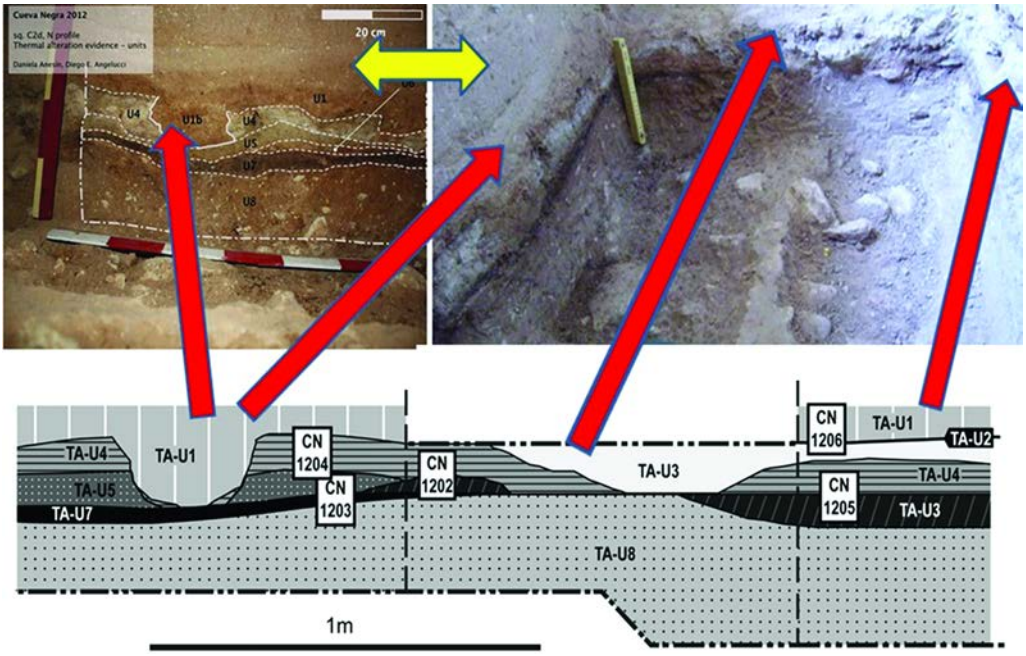


Figure 9. Photographs and stratigraphy of deeply-lying sedimentary layers with burnt remains in metre-square C2d during excavation in 2012. Adapted from the Supplementary Information published in Angelucci et al., 2013 (http://dx.doi.org/10.1016/j.quascirev.2013.09.009)

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.