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eprints@whiterose.ac.uk https://eprints.whiterose.ac.uk/ 1 Evidence from burial sediments for prehistoric burial practice and ritual in

- 2 Monte Claro chambered tombs: micromorphology, mineralogy and 3 geochemistry
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19 Highlights

- Signatures present in burial sediments can aid interpretation of human burials
- CaCO₃ depletion in sediments underlying disarticulated remains indicative of primary burial
- Reddish deposit identified as being ochre applied to the grave
- The silicified remains of a sedge torch reveal how the burial chamber was illuminated

¹ Deceased 13 May 2018 (MRU) and 26 September 2016 (DRB)

24 Abstract

25 The burial matrix in archaeological graves is seldom subject to detailed examination for evidence of 26 its potential interaction with the interred remains. Sediments adjacent to skeletal remains resting on 27 a marl platform in a subterranean chambered burial dated to the third millennium BC were 28 micromorphological, archaeobotanical, and chemical methods. investigated using 29 Micromorphological features in the tombs, typical of CaCO₃ depletion, appeared more abundant in 30 positions close to the skeletal remains than in positions further away, suggesting that the carbonate 31 dissolution was caused by acidification resulting from the body decay as well as from environmental 32 factors. Organic signatures around the remains were dominated by background sedimentary organic 33 matter of the marl, attesting to the exposed style of burial being unfavourable for the preservation of 34 organic remains. Red concentrates present in a distinct region of the resting platform were 35 distinguishable from similar coloured deposits formed by debris from the vault. They were identified 36 as haematite (ochre) deposits, matching features observed in other prehistoric sites, including those 37 of the same culture, and attesting to ritual associated with the treatment of the dead. Greyish fibrous contexts found on the burial resting platforms were identified as silicified sedge, probably remnants 38 39 of the combustible parts of torches. The position and nature of the sedge-derived material suggests 40 their use for illuminating the graves at depth and/or as a possible votive ritual. The detailed scientific 41 examination of the burial matrix can reveal unique perspectives and add interpretative value to the 42 archaeological investigation of graves.

43 Keywords: Eneolithic chambered burials; geoarchaeology and micromorphology; organic
 44 geochemistry; ochre; ritual burning.

45 1 Introduction and site description

46 The InterArChive project considered the extent to which the burial matrix preserves evidence that is 47 currently unexploited within the archaeological investigation of human burials (Usai et al. 2014). 48 Soils/sediments originating from a diverse range of temporal and geographical contexts were 49 examined using systematic sampling and a multidisciplinary analytical approach (Burns et al. 2017; 50 Pickering et al. 2014, 2018; Usai et al. 2014). One of the sites examined, Gannì, Sardinia, belongs to 51 the Eneolithic Culture of Monte Claro, a culture that displays intriguing signs of ritual burial practices 52 (Manunza 2010a, 2010b; Ugas 1993). The culture was named after discovery, in 1906, of an Eneolithic 53 burial in the Monte Claro area within the city of Cagliari. The burial contained a stone bench as a resting platform for the dead, and various grave goods including a situlate vessel and a monoansato 54 55 (single handled) bowl. In this, and other Monte Claro tombs discovered later, the primary deposition 56 of the dead was in a crouched position on their left side with features ascribed to ochre in some of the graves. The practice of adorning ancient graves with ochre has been described for archaeological
sites as far back as the Palaeolithic (Roebroeks et al. 2012), the deep red colouration in burials from
various sites and periods being attributed to haematite (Gialanella et al. 2011; Roebroeks et al. 2012).

60 Two underground chambered Copper Age burials of the Eneolithic Culture of Monte Claro, at the 61 archaeological site of Gannì (Fig. 1a, b), presented a unique opportunity to examine the evidence for 62 ritual burial practices within this culture. The vaults, carved into the massive Miocene bedrock 63 (Carmignani et al. 2012) using prehistoric implements (Fig. 2a), extend to c. 3 m underground. Tomb 64 I contained two burial chambers (T1 and T2) whereas Tomb II was constructed with a small entrance vestibule connected to a single burial chamber (Fig. 2b). Tomb II contained human skeletal remains 65 66 radiocarbon dated to 2469-2293 BC (Manunza 2013). Tomb II and chamber T2 of Tomb I were found 67 fully sealed and unviolated, and hence with all architectural elements in place. The archaeological 68 features of the tombs and their cultural implications have been described by Manunza (2013).



69

Fig. 1. a) Location of the site in Sardinia (black rectangle); b) inset corresponding to the black rectangle

in a): site location is indicated by the star; c) close-up of the burial chamber of Tomb II with position

72 of skeletal remains indicated by the letters Sk.

The Gannì graves feature sub-vertical access tunnels leading from the land surface to underground burial chambers delimited by small stone walls and stone benches employed as resting platforms for the dead (Fig. 1c). Such architecture was frequent in Monte Claro burials in Sardinia and also among coeval cultural groups of Aegean-Anatolian background in central-southern Italy. The former include sites some distance from Gannì as well as others that are closer to the site (Table S1).

78 At the time of sampling, the skeletal remains in Chamber T1 of Tomb I at Gannì were disarticulated, 79 having been disturbed by thieves. Prior to the disturbance, Chamber T1 contained disarticulated and 80 damaged bone as well as the skeletons of a male and a female aged between 18 and 25 years and two 81 males aged 30-45 and 20-30 years resting on stone benches. In the second, unviolated chamber, T2, 82 the remains of two children rested in a niche in the wall and, directly on the floor below, were the 83 skeletons of a male and female couple (aged 18-25 years) next to a child aged 9-10 years. Tomb II, 84 fully sealed and unviolated, contained poorly-preserved skeletal remains of an adult female aged 35-85 50 years in anatomical position, lying crouched on the left side and resting on a bench carved in the marl (Fig. 3). Archaeological interpretation suggested that the burials date to the Monte Claro culture 86 87 (Manunza 2013). The absence of bone lesions excluded interpersonal violence as causes of death, 88 suggesting a local epidemic or, more likely given the patterns of articulation, that these were sites of 89 collective burials (Manunza 2013).



90

Fig. 2. a) Internal vault of Tomb II carved with prehistoric implements during the Copper Age; b) front
vestibule of Tomb II connecting to burial chamber through a small stone wall (see Fig. 1c for close-up).

The positioning of individuals either on stone-carved benches or in direct contact with the floor at Gannì, and also in the Cagliari, Selargius, Settimo and Serdiana sites, has been interpreted to relate to social status – tombs where additional efforts were expended to fashion stone benches were postulated to have been destined for people of higher status (Manunza 2013). An emergent group of high status within the local community was also suggested by the exceptional quality of some of the

98 pottery present in Tomb I (Manunza 2013). This included bowls decorated by incisions, plates, vases 99 with polished surfaces, cups, beakers, situlate vessels and fragments of vases. The pottery was similar 100 to that found at many other Monte Claro sites, the *situlae* being very similar to those found in sites 101 South of the S'Imbirizzi Lake and in Cagliari. Vessels and monoansato bowls from Ganni were also very 102 similar to those of the first Monte Claro burial excavated in Cagliari, particularly in the shape of the 103 rims and identical decoration of the bowls. Other fragments of ceramics had identical decorations to 104 those of a site near Villasor. A stone axe, three-pointed stone tool and a pointed copper implement 105 were also found. The grave goods were all from Tomb I, no grave goods were present in Tomb II, and 106 their features were not sufficient to determine whether they were part of any particular burial practice 107 or ritual at the site.



108

Fig. 3. Plan of Tomb II resting platform, with context and sample distribution (notations in Table S2;
height expressed in metres above sea level). The skeletal remains (green in colour image) were
orientated NW-SE with the skull to the south east.

The vault marl, of dominant colour in the yellow-red range (2.5YR8-7/1-2) (Munsell 2009), contained variable amounts of orange-coloured deposition nodules. Much of the bone in Tomb II had been lost due to decay and the skeletal remains and the platform on which they rested (Fig. 3) were covered in places by sparse sediment deposits, tentatively interpreted on site as debris fallen from the vault. The debris displayed several colours in the yellow-red range (2.5YR7-6/3-4) (Munsell 2009) and rare fine bio-voids and cracks were visible to the naked eye. Fine rock fragments and poorly developed soil aggregates (peds), barely visible at field scale, suggested a low degree of pedogenesis. Two visually distinct areas of the resting platform, one red-coloured (Context 32) and the other grey (Context 28),
were evident (Fig. 3).

121 1.1 Deep red concentrates

122 Concentrates of very deep red colour, varying in the range 2.5YR 8-5/3-4 (Munsell 2009), were situated 123 in the vicinity of the pelvis and thighs of the skeleton in Tomb II (Context 32; Fig. 3). The material 124 appeared as a covering over part of the resting platform and as deposits with sharp or diffuse 125 boundaries over bone fragments and the local rock/sediment components (Fig. 4). Preliminarily on-126 site interpretation was that the material could be ochre (Manunza 2013), as observed in other ancient 127 burials (Roebroeks et al. 2012).



128

Fig. 4. a) Photograph of Context 32, comprising a deep red material (area marked by 'r' and enclosed by white outline); b) detail of the red material showing the intense red feature indicated with 'r' in a).

131 1.2 Fibrous grey contexts

Loose grey material was found near the presumed position of the feet in Tomb II (Context 28 in Fig. 3), forming a thin, irregular, sub-circular lens over the resting platform (Fig. S1). A visually similar material was present in a clump to the side of and between the two burial platforms in Chamber T1 of Tomb I (Context 13). Visual inspection revealed the material to include fine fibrous filaments; its nature and possible ritual implications was unclear from field and macro-scale observations.

- 137 The nature, chemical and mineralogical compositions of two coloured pedofeatures in Context 32 and 138 of the grey fibrous material of Contexts 13 and 28 were examined to determine their compositions 139 and significance in relation to the grave sediments, burial practices and effects of body decomposition
- 140 on the burial matrices that had been adjacent to and beneath the skeletal remains.

141 2 Methods

142 2.1 Sampling

143 The sampling protocols for the InterArChive project (Usai et al. 2014) were followed as closely as 144 possible given the limitations of the incomplete nature of the skeletal remains (Fig. 3, Table S2). 145 Undisturbed sediment samples were collected with Kubiena tins or hand lifting, each with a loose 146 replicate. Site controls were collected from the marl surrounding the grave: from the sides, $C1(\alpha)$, and 147 from a marl bed equivalent to the resting platform from the vault in Tomb II, $C1(\beta)$ (Carmignani et al. 2012). Control C4 was collected from the thin layer deposited on top of the skeletal remains in Tomb 148 149 II. The sediments from the skull and pelvic regions were collected from the upper part of the resting platform under the skeletal remains. Loose sediment samples for organic chemical analysis were 150 151 collected from numerous positions around the skeletal remains (Table S2) and from the red and grey 152 contexts. Thin sections were analysed from the block-lifted undisturbed samples. They were 153 examined by light microscopy and scanning electron microscopy-energy dispersive X-ray spectroscopy 154 (SEM-EDS). Loose sediment samples were dried, sieved and aliquots of selected samples subjected to 155 X-ray diffraction (XRD). Aliquots were also subjected to total organic carbon analysis and extraction 156 with organic solvent to recover organic residues. The organic residues were derivatised and analysed 157 by gas chromatography-mass spectrometry (GC-MS). Full experimental details are given in SI.

158 3 Results and discussion

159 3.1 Micromorphology and inorganic chemistry

160 3.1.1 Grave and site controls

161 The material overlying the skeleton (Control C4) was visually and compositionally similar to the marl 162 of the vault, though with some evidence of pedogenesis, consistent with debris originating from the chamber vault rather than being from allochthonous material that was intentionally deposited on the 163 164 skeleton. Thus, the C1 controls, representing the marl from which the vault was excavated, exhibited 165 largely uniform groundmass with c. 50% grey and crystallitic fine material ($\leq 2 \mu m$) and a coarser 166 fraction (mainly \leq 50 µm grains but occasionally as grains and composite silt agglomerates of up to 200 µm). The orange-coloured nodules observed on-site in the grave vault were not observed in the 167 168 thin sections from $C1(\beta)$, probably as a result of local variability. Voids in the controls were mostly 169 planar, randomly oriented and typical of rock fracturing. Quartz and calcite were both very well represented in XRD analysis of the vault control C1(β) (Fig. S2). Control C4 showed evidence of pedogenesis through the presence of 2 - 200 μ m granular peds (80% of slide area), sub-angular blocky peds and small randomly orientated biovoids. Orange-coloured nodules observed in thin section reflect material derived from the vault.

174 3.1.2 Debris on resting plane (Contexts 32, 37 and 38)

The samples from the resting platform, while exhibiting compositional features consistent with derivation from the marl, showed limited evidence of pedogenesis and alteration. Thus, within the upper *c*. 10 mm the deposits under the pelvis (Context 38) and the red area adjacent to it (Context 32), rare, very small and poorly developed peds were observed (Fig. S3). Voids with sub-parallel orientation to the resting platform and cloudy crystallitic *b*-fabric only in the regions under the skull (Context 37) and pelvis (Context 38) reflect pedogenic processes in areas contiguous to the skeletal remains, *i.e.* in the upper *c*. 10 mm of areas of the resting platform below the skeleton.

182 3.1.2.1 Pedofeatures T and R

183 The debris coating the upper 10 mm of the surface of the resting platform contained two distinct 184 pedofeatures, designated T and R. The debris was disturbed beneath the thigh (Context 32) and pelvic 185 regions (Context 38). Pedofeature T was compositionally similar in both areas and dominated the samples. Its chemical composition and anatomical location are consistent with decalcified marl 186 187 formed by leaching due to generation of acidic fluids during decomposition of the body. Specifically, pedofeature T, beige (PPL) and frequently isotropic or dark grey in XPL exhibited lower crystallinity 188 189 than the groundmass (Fig. 5). Abundance levels were \geq 50% of the thin section areas throughout the 190 upper 8 mm of the resting platform, with a sharp decrease to almost 0% at 8 mm depth but 191 occasionally extending further down to 20 mm depth (Fig. 5). SEM-EDS revealed that pedofeature T 192 in the thin sections from Contexts 32 and 38 showed no significant difference in elemental 193 composition, suggesting similar origins or processes of formation in both contexts. Pedofeature T was 194 dominated by Si and Al together with Fe, K, and trace amounts of Na, Mg, P and S (Table S3). The Fe 195 contents were higher, and Ca substantially lower, than in the adjacent grey matrix in the thin section and in the C1(β) control (Table S3). XRD analysis indicated that Context 38 exhibited significantly 196 197 depleted calcite levels relative to the marl of the vault. Given that the micromorphology and depth 198 profiles of pedofeature T suggest that it formed as an alteration product rather than as a surface 199 deposit, the chemical composition was compared with that of the unaltered grey fabric of the resting 200 platform (Table S3). As with the vault control, the grey fabric was dominated by Ca, the levels being 201 much higher and Si, Al and Fe being lower than in pedofeature T (Table S3). To account for 202 decalcification, the data were normalised to the Si content, enabling direct comparison of pedofeature

T and the grey matrix (Fig. 6). The significantly lower Ca and Mg contents and slightly higher, but statistically significant, Fe content is consistent with pedofeature T being formed by depletion of the grey matrix. The prevalence of pedofeature T in the deposit directly under the pelvis hints at the depletion being caused by generation of acidic fluids during decomposition of the body, consistent also with the lower prevalence under the skull.



Fig. 5. Mosaic image comprising all adjacent fields of view from the thin section from Context 32: a) PPL, pedofeature R comprises deep to very light red opaque material and b) XPL. Pedofeature T appears black or very dark. The bulk of pedofeature T appears as a disturbed zone of lower crystallinity situated under the sub-horizontal resting plane (toward the upper part of the image). A small cavity within the sub-horizontal resting plane is denoted C. The grey matrix (G) appears mostly in the lowermost part of the section (as also observed in Context 38).

Pedofeature R, deep to very light red opaque material (Figs 3 and S4), was generally adjacent to 215 216 pedofeature T (Fig. 5). The abundance levels dropped sharply from c. 10% area in the uppermost 7-217 11 mm of the resting plane in Context 32 to only occasional occurrences below 7-11 mm depth. Context 38 contained a few fragments of pedofeature R below the pelvic bone. The material differs 218 219 chemically from the marl of the vault and was identified as haematite, consistent with ochre deposits 220 added to the surface of the resting plane. Specifically, the material of the reddened area of the resting 221 platform (Context 32) contained isolated micro-patches of Fe oxides of variable colour intensity in thin 222 section (Fig. S4). The accumulations (ranging from 10 to 150 μ m) were interpreted as weakly to 223 strongly impregnated nodules and occasionally as fragmented typic- or hypo-coatings. Pedofeature R 224 in Context 32 exhibited an appreciable content of silica as revealed by SEM-EDS analysis (Si = 10.2%) 225 and the Fe contents of the red particles were highly variable (Table S3). The low concentrations of Al, 226 Ca and Mg suggest relatively low levels of aluminosilicate and carbonate components by comparison 227 with the vault control and grey matrix in the undisturbed samples from the resting platform (Table 228 S3). XRD analysis indicated these deep red components to be composed mainly of haematite,

229 consistent with ochre deposits: goethite was not detected in any of the samples (Fig. S2). The similar 230 levels of quartz and calcite in the host materials (Fig. S2) suggest similar mineral types, sizes and 231 orientations. Hence, it is likely that the intensity of the secondary XRD radiation correlates with 232 differences in amounts of mineral species present. Thus, the depth distribution, disturbed nature, 233 chemistry and morphology of the pedofeature is consistent with an allochthonous material deposited 234 on the surface of the resting platform and incorporated into the disturbed/debris layer to a depth of 235 *c*. 11 mm.



Fig. 6: Comparison of relative element abundances in beige pedofeature T and grey grave *b*-fabric.
Significance threshold = 0.05.

240 3.2 Grey fibrous material (Contexts 13 and 28)

241 Microscopy revealed the dominant material of the grey fibrous deposit of Context 13 to contain 242 recognisable remains of plant material. When concentrated by acid dissolution of carbonate, characteristic features of Poales (grasses and sedges) could be recognised. The plant remains were 243 244 preserved mainly as silica, consistent with the material having been burnt in situ. Specifically, under 245 low-power microscopy, the grey fibrous material of Context 13 appeared to include two components: 246 a minor one comprising tubular structures (c. 15 mm × 2 mm), possibly parts of recent roots, and a 247 major component comprising finely divided amorphous granules and powdery material. The latter, 248 also present in Context 28, contained bright white spicules with maximum dimension up to about 4 × 249 0.5 mm but mostly about 2 × 0.25 mm. Treatment of unconsolidated replicates of Context 13 with hydrochloric acid revealed that the granules and powder were highly calcareous whereas the spicules 250 251 remained undissolved. At ×400 magnification the spicules were recognisable as fragments of plant 252 epidermis in the form of silica skeletons, where the original cell walls had been replaced by silica. Most 253 specimens bore typical epidermal features of Poales (grasses and sedges) in the form of long and short cells, the former with characteristic finely sinuous walls (Fig. 7a). Many fragments bore small 'prickles' 254 255 (micro-hairs) on their surfaces (Fig. 7a) and some bore more obvious and guite substantial marginal

teeth (Fig. 7b, c). Some fragments bore rows of stomata exhibiting the morphological characteristics
(elongated dumbbell shaped structures) associated with grasses and sedges (Fig. 7b). In other cases
the fragments appeared to be more or less cylindrical, with prominent teeth (Fig. 7d), and were
presumably from a bristle or awn from the tip of a leaf or from a spikelet within a flowering head.



260

Fig. 7. Optical microscope (×400 magnification) images of silicified plant remains from Context 13. Epidermis with a) long cells, short cells (white arrows) and prickles (black arrows). The single 'spiny' structure at the bottom centre is a detached epidermal cell; b) marginal tooth (black arrow) and a row of stomata (white arrows); c) sharp marginal teeth indicated by arrows; d) awn fragment with whorls of teeth.

Further identification of the grey fibrous material was not possible in the absence of relevant reference material though, on the basis of the tissue anatomy observed, only one or two taxa are likely candidates. The size is rather small for one of the major cultivated grasses (*i.e.* cereals). Preservation by silicification² has been observed occasionally in archaeological occupation deposits where it was inferred that large quantities of silica-rich plant material had been burnt under strongly oxidizing conditions, generating temperatures sufficient for the silica in the tissues to be fused, but low enough

² Silicification is a term used in palaeobotany to denote permineralisation of tissues by silica within an aqueous burial environment, and this differs as a mechanism from what is presumed to have occurred in these archaeological specimens.

272 to prevent loss of cell structure (Robinson and Straker 1991). Such silicified material was attributed 273 to rapid accumulation or burial of plant materials, protecting the delicate cellular structure from 274 mechanical damage (Hall and Huntley 2007). Material of this kind in several prehistoric and medieval 275 sites in England represented concentrations of cereal chaff (Robinson and Straker 1991) and, in several 276 sites in Northern England, material other than cereal chaff was sometimes recorded (Hall and Huntley 277 2007). SEM-EDS analysis of the fibrous material in Context 28 confirms its siliceous nature (Table S3), 278 consistent with silicified plant epidermis as suggested from palaeobotany. The XRD analysis indicated 279 that calcite and quartz were scarce in the grey fibrous material of Context 28, contrasting with the 280 high level of Si detected by SEM-EDS (Table S3) and calcareous component indicated by dissolution 281 with acid. The apparent discrepancies are consistent with dominance of poorly ordered carbonate 282 and silica (Drees et al. 1992) formed by a process, such as burning, that did not induce crystallisation. Hence, the components of Contexts 13 and 28 represent remains of leaf and flower parts from non-283 284 cultivated herbaceous materials that underwent silicification induced by fire. Given the delicate 285 nature of the material and its form, the mineralisation must have occurred in situ.

286 3.3 Organic chemical analysis

With the exception of the grey material (Context 28) the organic carbon contents of the sediment 287 288 samples from around the skeletal remains were not distinguishable from those of the controls 289 suggesting there to be no significant contribution from the human remains. The absence of organic 290 material associated with the coloured pedofeatures in Context 32 is consistent with its colour being 291 due to haematite rather than to an organic dye. By contrast, Context 28 contained molecular 292 signatures consistent with an origin from sedge. Specifically, total organic carbon (TOC) analysis 293 revealed the majority of sediment samples from around the skeletal remains to be very similar to the 294 controls, containing low levels of organic carbon (TOC values << 1%; Table S4) and with the majority 295 of carbon being in the form of inorganic carbonate. By contrast, the grey material (Context 28) 296 exhibited the highest TOC content (0.77%). GC-MS analysis of the total solvent extractable organic 297 matter revealed the samples to contain organic compounds including C_{16:0} and C_{18:0} fatty acids, 298 monoacylgycerols (MAGs), *n*-alkanols (C_{18:0} and C₂₄-C₃₀), *n*-alkanes (C₂₉ and C₃₁), β-sitosterol and 299 cholesterol (Fig. 8), all present in low concentrations, consistent with the low organic matter contents 300 indicated by elemental analysis. For the most part, little variation was evident between the lipid 301 profiles of the controls (e.g. $C1(\beta)$) and samples taken from around the skeletal remains (e.g. Fig. 8a) 302 suggesting they contain predominantly background sediment organic matter with little contribution 303 from the human remains.

304 By contrast, the extract from the grey deposit (Context 28) contained appreciable amounts of C22-C32 305 *n*-alkanoic acids, C₂₄-C₃₀ *n*-alkanols and β-sitosterol (Fig. 8b) together representing 6136 ng/g sediment, *cf*. 383 ng/g sediment for C1(β). The higher levels of those components in the grey deposit 306 307 is indicative of a significant higher plant input (Eglinton and Hamilton 1967; Jambu et al. 1993). In 308 addition, the profile includes two prominent peaks at retention times ($t_{\rm R}$) 53.5 and 57.5 min (Fig. 8b), 309 which do not feature in the other extracts and are comparable in abundance to β-sitosterol. GC-MS 310 revealed their molecular ions (M^{+}) at m/z 548 and 576 and the same principal fragment ions: m/z 268 311 (base peak) accompanied by low abundance ions at m/z 267, 281 and 310 (e.g. Fig. 9). The EI mass 312 spectra are characteristic of long chain alkylresorcinols (Avsejs et al. 2002). The ratio of m/z 268:267 of approximately 5:1 is consistent with the attachment of the alkyl chain at position 5 on the aromatic 313 ring (Zarnowski and Kozubek 1999). Accordingly, the two components were assigned as 5-n-314 315 alkylresorcinols having C₂₁ and C₂₃ alkyl chains, respectively. The presence of a minor C₁₉ alkyl homologue (M⁺ = m/z 520) at t_R = 50.5 min (Fig. 8b) was revealed in the m/z 268 extracted ion 316 317 chromatogram.



Fig. 8. Partial FID gas chromatograms of the total lipid extracts from a) the abdomen, Context 38, and b) the grey material, Context 28. Triangles = n-alkanoic acids, squares = alcohols (mainly n-alkanols unless labelled otherwise), diamonds = n-alkanes, stars = 5-n-alkylresorcinols, MAG = monoacylglycerols. Numbers denote carbon chain length. * = plasticisers arising from plastic bags used for sample storage.

324 Previous reports of 5-n-alkylresorcinols possessing alkyl chains in the range C13-C27 and exhibiting a 325 prevalence of odd-chain members indicate a range of natural sources including higher plants, algae, 326 fungi and bacteria (Kozubek and Tyman 1999). Their biological function is largely uncertain although 327 they are believed to fulfil roles including microbial resistance, growth regulation and membrane 328 modulation (Kozubek and Tyman 1999). Cereals such as wheat and rye contain substantial amounts 329 of alkylresorcinols. Previous studies, involving pollen analysis or examination of the contents of burial 330 pots, have indicated foodstuffs, including cereals, as occasional items placed in human burials (Lagerås 331 2000). The 5-*n*-alkylresorcinol distributions of cereals, typically C₁₇-C₂₅ and dominated by the C₁₉ and C₂₁ components (Chen et al. 2004; Mattila et al. 2005; Ross et al. 2003), do not match that of the grey 332 333 context, whereas dominance of the C_{21} and C_{23} 5-*n*-alkylresorcinol homologues for the sedge 334 Rhynchospora alba (Avsejs et al. 2002) represents a close match. The resorcinols and silicified remains 335 indicate that the grey material associated with the burial is plant-derived, consistent with the visual 336 description of a fibrous plant like material overlying the area from which the sample was collected. 337 The survival of such clear indicators of plant remains after 4500 years of burial is remarkable, especially 338 given their exposure to air and to combustion. The apparent recalcitrance of the 5-n-alkylresorcinols 339 in that material could be due in part to their antimicrobial properties and may have extended 340 protection to other biomarkers such as β -sitosterol.



341



As with the samples in close association with the skeletal remains, the total lipid extract from the red material (Context 32) is extremely similar to those of the controls. The solvent extract itself was colourless and no components which would explain the red appearance of the sediment sample could be identified by GC-MS. In order to rule out the possibility of the substance responsible for the colour of the sample being physically or chemically bound to sediment matrix, for example via ester linkages between acid residues and free hydroxyl groups present on mineral surfaces, the sediment sample was re-extracted with methanolic sulfuric acid which can disrupt such associations and release the bound molecules as methyl esters (Huseby and Ocampo 1997). The acid extraction did not produce a coloured extract. Thus, on the basis of its inconspicuous extractable lipid profile it appears that the red material owes its colour to an inorganic mineral colourant rather than an organic chromophore.

353 4 Summary and conclusions

Analysis of the sediments from the tomb provided intriguing information relating to the burial practice and ritual of the Monte Claro culture burial at Gannì. Furthermore, the impact of body decay on the matrix adjacent to the human remains was evident through alteration of the sediments.

357 4.1 Pedogenesis associated with the human remains

358 Consistent with the tentative on-site archaeological interpretation, the lithified debris lying on the 359 bone surface is attributed to material that had fallen from the vault of the chamber rather than being 360 brought from elsewhere and lain on the skeleton for ritual or other purposes. Pedogenesis was 361 pronounced only in the material that directly interacted with the organic matter from the body, both 362 in the debris covering the remains and within the upper c. 10 mm of the resting plane under the skeletal remains. The similar organic carbon contents and compositions of these samples and the marl 363 364 indicates that the organic components of the body tissues have degraded to below detectable limits 365 within the burial chamber where the remains were exposed to the air. Thus, despite the complete 366 degradation of the organic remains, the pedogenic alteration of the sediment adjacent to the skeletal 367 remains provides evidence of *in situ* decomposition of the body.

368 4.2 Calcium carbonate depletion from body decomposition

369 Pedofeature T, in the thigh and pelvic regions and in Context 32 containing the red concentrates, is a 370 depletion pedofeature characterized by a significant loss of CaCO₃, small decrease in Mg and 371 consequent relative increase in Fe levels compared with the surrounding grey matrix. Although 372 sporadic patches of carbonate depletion occur in most samples, depletion features were considerably 373 more abundant beneath the thigh and pelvic regions reaching \geq 50% area within the uppermost 10 374 mm of the resting platform. The decalcification is attributed to localised acidification resulting from 375 the generation of organic acids during body decomposition (Dent et al. 2004). Subject to the sediment 376 matrix containing carbonates, such evidence could be of value in recognising primary burials in cases 377 where skeletal articulation can not be established.

378 4.3 Ochre coloured context (Context 32)

379 The red haematite concentrates in Context 32, representing c. 10% by area of the disturbed/debris 380 layer at the surface of the resting platform, were mixed with deposits that otherwise have very similar 381 chemical compositions (with the exception of P and S) to the marl of the vault. The low organic matter 382 content confirms the inorganic nature of Context 32. The extreme Fe concentrations in some regions, 383 and high variability, reflect the heterogeneity observed by micromorphology. The Fe concentrations 384 are well within the range recorded elsewhere for ochre type deposits (Attard Montalto et al. 2012; 385 Gialanella et al. 2011; Iriarte et al. 2009; Popelka-Filcoff et al. 2008; Ramos et al. 2008). The 386 heterogeneous mixture of haematite, silicates, clays and carbonates is also consistent with the previous analyses of archaeological ochre pigments (Darchuk et al. 2010; Gialanella et al. 2011). At 387 388 other sites in Sardinia (Monte Claro culture: Sibiola, Su Fraigu (S. Sperate) and Sa Costa) and a Neolithic site at Ozieri (Manunza 2010a, 2010b) ochre was used for grave paintings and to adorn the dead. At 389 390 Sibiola (16 km N of Ganni), ochre was used for single inhumations of elderly women, in tombs of 391 identical architecture to those at Gannì. Thus, the identification of ochre deposits at Gannì, applied 392 to a specific area of the grave as part of the burial ritual, complements and extends previous 393 interpretations.

394 4.4 Grey fibrous material: torches from sedge

395 The grey fibrous material of the thin sub-circular lens of Tomb II and the clump to the side of the 396 skeleton in Tomb I contained plant remains from the leaf and flower parts of non-cultivated 397 herbaceous materials of the order Poales. Though almost entirely silicified, the material showed 398 poorly ordered crystal form. The grey material from Tomb II (Context 28) contained plant-derived 399 lipid components including a series of 5-n-alkylresorcinols. This supports the evidence from 400 archaeobotanical analysis that the deposit relates to degraded plant material and suggests that it was 401 deliberately placed in the tomb, probably as part of the burial ritual. In combination with the 402 archaeobotanical evidence, the 5-n-alkylresorcinols provide compelling evidence for the material 403 being from sedge. Analogous to elsewhere, it is suggested that silicification was induced during 404 combustion. The rapid accumulation of the material *in situ* strongly suggests it was burnt in the burial 405 chamber, though combustion was incomplete enabling some organic signatures to preserve. Given 406 that the only access to the tombs at the time of burial was through a c. 3 m long vertical tunnel, it is 407 likely that the grey fibrous materials derive from remains of torches fabricated from sedge, incomplete 408 combustion possibly being associated with a tightly bound area. No other comparable contexts or 409 other evidence for the use of torches exist in burials of the Monte Claro culture, nor is it known how 410 these people illuminated their dwellings and caves.

- The use of torches within the tombs could have been either for practical purposes or as part of a burial 411 412 ritual. There is scant evidence of votive or ritual burning at other sites of the Monte Claro culture. 413 Hypotheses of ritual burning for the dead were proposed on the basis of the presence of pieces of burnt wood found among grave goods adjacent to the dead in a cist (Gemiliano site) and the presence 414 415 of some 5-8 cm vessels, considered to be either ointment holders or lamps, found near cremated or 416 semi-combusted human bones in a disturbed cave (Tanì site). No information or further evidence to 417 support the hypotheses is available (Lilliu 1998). In both cases, it is likely that those who sculpted the vaults and buried the dead needed light to illuminate the tombs during their passage, to carry the 418 419 dead into the deep (3 m) and dark underground chambers and to prepare the burials with ochre and 420 grave offerings for the dead. It is reasonable to assume that as the living withdrew from the tombs
- 421 they left the torches in place, most likely as a votive offering to provide light for the dead.

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