Soil geochemical methods in archaeo-geophysics: 1

exploring a combined approach at sites in Scotland 2

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7 ABSTRACT

8 This paper explores the application of geophysical and soil geochemical methods to 9 detect archaeological features in three traditionally 'difficult' survey environments in 10 Scotland: wind-blown sands (Bay of Skaill, Orkney), clay (Chesterhall Parks Farm, 11 Lanarkshire) and glacial drift deposits (Forteviot, Perthshire). The results presented here 12 are part of the first research project that systematically tested a combined approach 13 using geophysical and soil characterisation to understand the proxy responses of known 14 archaeological features. 15

16 First, a range of geophysical techniques (earth resistance, magnetometry, FDEM and 17 GPR) was employed over archaeological targets. Second, the different geophysical 18 results were considered with respect to soil chemical concentrations (total phosphate 19 and multi-element analysis), texture, pH, conductivity, organic matter content and 20 magnetic susceptibility from archaeological deposits, topsoil and subsoil samples.

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22 This study demonstrates that, by focusing on the responses of single archaeological 23 features and assessing their physical and chemical signatures, soil composition and 24 processes involved in the 'history' of buried features are of importance in improving our 25 understanding of the reasons behind their detection with geophysical means. For example, at the cropmark site at Forteviot, chemical transformations can be triggered by 26 27 organic matter accumulation and increased water retention within prehistoric ditch 28 deposits and can have an effect on the type of magnetic contrast. In addition, chemical 29 concentrations revealing anthropogenic organic materials can explain the enhanced 30 conductivity of theoretically impervious features, as illustrated at the Bay of Skaill site.

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32 The study also provide insights into how a particular soil environment may affect 33 different geophysical techniques such as the masking effects of heterogeneous glacial

- 34 drift deposits or deep windblown sands. These types of survey environments
- 35 characterise many archaeologically rich areas in Scotland, where integrated strategies,
- 36 such as the one used in this investigation, are the best option to maximise detection of 37 subsurface features and provide confident and augmented interpretations.
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- 39 Key words (6): multi-technique geophysical survey, soil chemical analysis, soil
- 40 magnetic susceptibility, soil physical characterisation, archaeological prospection,
- 41 Scottish archaeology

42 1. INTRODUCTION

43 Geophysical and soil geochemical methods have been widely used to locate and 44 interpret subsurface evidence of past human occupation in a non-destructive and 45 minimally invasive manner (Clark, 1990; Sarris & Jones, 2000; Scollar, Tabbagh, 46 Hesse, & Herzog, 1990). The discipline of archaeo-geophysics has played an important 47 role in discovery and characterisation of cultural heritage assets, from single features 48 and sites to entire archaeolandscapes (Becker & Fassbinder, 2001; Campana & Piro, 49 2009; Gaffney & Gater, 2003; Powlesland & Lyall, 1996; Apostolos Sarris, 2015). 50 Geochemical methods, although adopted on a smaller scale, have also yielded valuable 51 information on the location and extent of archaeological sites as well as investigations 52 into past land use and activity areas (Aston, Martin, & Jackson, 1998; Bintliff, Gaffney, 53 Waters, Davies & Snodgrass, 1990; Entwistle, Abrahams, & Dodgshon, 1998; Jones et 54 al., 2010; Middleton & Price, 1996; Terry, Fernández, Parnell, & Inomata, 2004; Wells, 55 2004; Wilson, Davidson, & Cresser, 2009).

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Geophysical and geochemical prospecting methods are based on very different detection
and analytical principles. However, a fundamental and common requisite for their
successful application is the existence of a physico-chemical contrast between the
buried objects or deposits (e.g., archaeological features) and the surrounding soil
environment. If sufficient contrast exists, these methods are able to map 'anomalous'

variations of the physical properties of the ground or soil geochemical concentrations,
 caused by past human activity (Figure 1). Factors linking soil physical, chemical and
 biological properties, soil processes and their dynamics with different types of
 archaeological features are fundamental in controlling this contrast.

66

67 The effects of these soil factors on the variable results provided by geophysical and 68 geochemical methods are still not fully understood (Armstrong, Cuenca-García, & 69 Moffat, 2015; Oonk, Slomp, & Huisman, 2009). Some consequences of this gap in 70 knowledge are the limitations in extracting all the information available from these 71 datasets, constrained or vague interpretations and disappointment in the application of 72 these methods.

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74 This paper presents the findings of a study that explores the potential of a combined 75 survey strategy, integrating a range of soil geochemical analyses as part of multi-76 technique geophysical surveys to detect archaeological features. The combined 77 approach was systematically applied at three contrasting archaeological sites in 78 Scotland, each one presenting specific challenges to archaeological prospection and 79 conducted over known archaeological features. The aim of the study was to assess 80 possible effects of soil and site setting on the results provided by the different methods 81 in order to improve the existing understanding of the factors involved in the genesis of 82 contrast and type of detected signatures.

2. MATERIALS AND METHODS 83

2.1. Study sites 84

85 Archaeological sites and targets were selected on the basis that they contained well-86 documented archaeological features, covered different environmental settings and 87 presented specific challenges relating to archaeological prospection (Figure 2) (Table 88 1).

89

90 Bay of Skaill (Orkney): a Viking longhouse was discovered on the summit of the 'East 91 Mound', situated near another other important focus of Viking activity, the mound of 92 Snusgar (Griffiths, 2006). Previous gradiometer surveys at the area characterised these 93 mounds as clusters of strong magnetic responses (Griffiths, 2011). Test trench 94 excavations exploring a magnetically quiet area at the 'East Mound' uncovered the 95 longhouse. A linear stone structure revealed near the longhouse during the test trenching

- 96 was selected as the target for this study (Figure 2, a).
- 97

98 Chesterhall Parks Farm (South Lanarkshire): this site contains a group of Iron Age 99 ditched enclosures that were first identified by aerial photography as cropmarks. 100 Previous gradiometer and earth resistance surveys at the site produced inconclusive 101 results in terms of resolving the enclosures revealed by the cropmarks (Sharpe, 2004, 102 181). Test trench excavations confirmed the presence of deep ditches, and they were 103 exposed close to the surface. Finds retrieved included large amounts of cremated bone 104 from floor layers, lead slag, ore, and in situ burning with some burnt wood (Sharpe, 105 2004, 186). Chemical analysis of bulk soil samples collected during the excavation 106 showed enrichment of Zn, S, Ca and P and other depletions within the ditches as well as 107 enhanced Ca concentration in floor samples. The targets selected at this site were the 108 ditches of two enclosures identified by aerial photography (Figure 2, c).

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110 Forteviot (Perthshire): cropmarks identified by aerial photography revealed a 111 concentration of Neolithic to Bronze Age hengiform monuments (Alcock & Alcock, 112 1992). These included a massive palisaded enclosure containing an earth henge 113 monument and other enclosures outside the palisade. The results of previous 114 gradiometer surveys yielded very faint negative responses indicating the ditches. The 115 results from former earth resistance surveys were similarly inconclusive. This case 116 study targeted the ditches of an enclosure located NW of the palisade (Figure 2, b).

Research Methods 2.2. 117

118 A research strategy combining a range of routine geophysical surveys and soil analytical 119 methods was systematically applied at the three archaeological sites (Figure 3). 120 Gradiometer, ground penetrating radar (GPR), frequency domain electromagnetics 121 (FDEM) and/or earth resistance surveys were carried out in order to detect and 122 characterise the geophysical response of the archaeological targets (**Table 2**). The 123 geophysical data collected for this investigation was minimally processed in order to 124 maintain the data as 'real' as possible. Geoplot, ReflexW & Surfer software were used 125 for data processing and map production.

127 Topsoil samples were collected over the targets along a line, generally using 1 m or 0.5128 m sampling intervals (depending on the size of the targeted feature). Soil was extracted 129 at different depths according to the expected depth of the archaeological remains and 130 topsoil interface. Open sections were carefully cleaned to take fresh samples from all 131 visible deposits. The sampling interval was sometimes smaller than 0.5 m in order to 132 sample small deposits visible in open sections. Soil was collected from the bottom of 133 the section upwards to avoid contamination, and sampling tools were cleaned after 134 collecting each sample. After removal of live organic material, soil samples were sealed in air-free plastic bags, stored in a dark environment and transferred as quickly as 135 136 possible to a cold store. Sub-samples were air dried at room temperature to reduce the 137 effects of possible chemical alterations. Air-dried soils were gently ground using a 138 mortar and pestle and then sieved using a mechanical shaker for 5 minutes. The 139 different soil analyses were carried out following the sequence shown in Figure 4. 140 141 Chemical analyses were carried out to characterise the chemical signature of the general 142 site (topsoil samples) and archaeological deposits (open-section samples) (Table 3).

143 Analyses included the determination of total (inorganic) phosphate and multi-element 144 concentrations using an X-ray fluorescence analyser (XRF). In addition, some samples 145 were analysed using inductively coupled plasma optical emission spectrometry (ICP-146 OES) to obtain further information about those elements outside the limits of detection 147 of the XRF technique and to act as a control of the XRF results. Magnetic susceptibility 148 (MS) was measured to identify possible anthropogenic deposits as well as to 149 complement the geophysical information (e.g., gradiometer results). Soil texture, 150 organic matter content (loss on ignition-LOI) and pH (and related electrical 151 conductivity-EC) were performed during the fieldwork and at laboratory premises. The purpose of these measurements was to obtain additional information about physico-152 153 chemical properties related to the archaeological deposits and overall soil environment. 154 The results of the different measurements on soil samples were analysed using 155 elementary statistics. From the geochemical data, relevant enhancements and depletions 156 were established by assessing their particular concentrations in the samples taken inside 157 and over the features (topsoil). The results were compared to off-site controls and general backgrounds found in the literature and available databases. Relevant 158 159 enhancements and/or depletions from the chemical data and other associations 160 established from the different soil analyses were spatially compared with the

161 geophysical responses using a GIS platform.

162 3. RESULTS

163 3.1. Bay of Skaill

164 The GPR and EM38 surveys carried out at this site confirmed the location of the 165 targeted stone structure, showing that these techniques can detect relatively small linear 166 targets in similar environments. Single GPR traverses using 450 MHz frequency 167 antennas detected the kerbed-wall as two high amplitude reflections, sometimes with a 168 third middle reflection, possibly caused by the infill deposits (**Figure 5**). These GPR results also provided a view of the underlying stratigraphic sequence of layered and
dipping deposits.

172 The vertical quadrature results of the FDEM were unexpected since the stone target 173 was, in principle, an impervious target. A fairly weak linear anomaly with a N-S 174 orientation correlated with the location of the targeted structure (Figure 5). During the 175 soil sampling, clayey and silty sand deposits were found within the stone structure. Soil 176 analyses showed an enrichment of K, Fe, Ti (possibly as a proxy for higher clay mineral 177 content) and general increases in MS, LOI, total phosphate and Mn, P, Rb, Ba, Cr, Li 178 (Figure 6). Similar enhanced concentrations and correlations have been reported in 179 association with organic archaeological deposits (Holliday and Gartner 2007; Wilson et 180 al. 2007). The higher water retention capacity and related higher conductivity of these 181 deposits seems to explain the conductive anomaly detected with the FDEM survey. The 182 on-going interpretation is that such deposits, in association with the linear structure, 183 may have been derived from the decomposition of an ephemeral organic wall or 184 enclosure, probably made of peat or turf and other materials used to hold the organic 185 blocks.

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187 Thin layered sands and dark organic deposits including a weakly cemented iron-pan 188 were visible in the exposed sections (**Figure 6**), extending westwards of the targeted 189 structure as observed in the GPR results. These midden-type deposits may have been 190 the origin of the strong magnetic anomalies recorded at the west of the target. Whilst 191 these deposits possibly contributed to mask the targeted structure for the gradiometer 192 survey, they also helped to reveal the presence of the archaeological area in the first 193 instance.

194 3.2. Chesterhall Parks Farm

195 The gradiometer survey satisfactorily resolved the enclosures using a 0.5 m traverse 196 spacing and parallel mode. The targeted enclosures were detected as two and three ring-197 like weak positive magnetic anomalies showing magnetically enhanced ditches (Figure 198 7). This survey also revealed other internal anomalies that may be related to structures 199 or functional areas within enclosure 1. In any case, these magnetic anomalies were still 200 fairly weak. Given the general low MS values of the clay matrix, frequent waterlogging 201 of the area, and zones with strong geological magnetic responses (possibly caused by 202 localised accumulation of varied glacial deposits), it is possible that the magnetic 203 contrast was partially masked by these mixed effects. The slopes of Tinto Hill are 204 mantled by frost-weathered detritus that has been extensively soliflucted (Ballantyne, 205 1993). The magnetic noise distinguishable in the north of the survey area may be caused 206 by this soliflucted material. In spite of the conductive clay soils, the 225 MHz frequency 207 GPR survey provided useful information relating to the total depth of the ditches (up to 208 1.20 m). The results of the 450 MHz frequency antenna survey were heavily affected by 209 signal attenuation, but the survey managed to receive some reflections produced by the 210 uppermost fill deposits of the ditches (~0.25 m). The quadrature component of the 211 FDEM survey in vertical dipole showed potential to detect the ditches (Figure 7). The 212 thickness and soil physical composition of these ditch deposits may explain their 213 detection by the quadrature component (conductivity) of the FDEM survey.

215 Despite the landowner's restrictions on opening a trench to expose and sample the 216 ditches, consent was granted for localised soil sampling using an auger up to 0.5 m 217 depth. Taking into account that this site was rarely ploughed, the shallow archaeological 218 deposits, and other metallurgical activities previously documented at the site, it was 219 expected that the results of the soil analysis would be fairly informative. However, the 220 results yielded only a slightly enhanced concentration of Pb and P (in agreement with 221 the previous soil analysis) and a high variability in the concentration of other elements 222 in the samples taken above the ditches (Figure 8). The pattern observed in the later 223 results seems to be produced by the truncation of the natural deposits by the ditches. 224 Whilst there was a slight MS enrichment approaching the centre of enclosure 2, it is not 225 completely certain that this enhancement would have been able to pinpoint the location 226 of the enclosure in a more extensive survey (Figure 9).

227 3.2.1. Forteviot

228 The gradiometer survey resolved only the enclosure when the traverse spacing was 229 reduced to 0.25 m in parallel mode. This technique revealed two concentric negative 230 magnetic anomalies: an outer ditch and a segmented, less coherent inner ditch (Figure 231 **10**). After topsoil stripping, this survey was repeated and revealed sharper anomalies 232 due to the enhanced magnetic contrast, which had been slightly masked by the topsoil (Figure 10). The topsoil contained magnetic stony material derived from intense 233 234 ploughing of the underlying glacial parent material. This resulted in 'noisy' datasets that 235 obscured the contrast between the ditch deposits and surrounding soil.

236

237 There was no MS enhancement or enrichment of common anthropogenic trace elements 238 in the ditch deposits, which partially explains the characteristic negative magnetic 239 response instead of the more usual positive response (Figure 11 & 12). The ditches at 240 Forteviot were associated with a ritual site, so their deposits were not subjected to 241 continuous anthropogenic input as at settlement sites. Soil samples from the outer ditch 242 showed slight general depletions of major elements (e.g., Fe, Figure 11), as well as a 243 discrete Mn depletion and LOI peak (deposit 1, Figure 12). These results point to 244 mineralogical changes inside the ditch that led to the relatively low MS values of the 245 ditch deposits compared to the higher MS of the topsoil and the even higher MS of the 246 subsoil (hence negative contrast), thereby contributing to their negative magnetic 247 responses.

248

249 The high percolation rate of the sandy top/subsoil at Forteviot and the general dry 250 conditions hampered several attempts to conduct resistance surveys during this 251 investigation and previous surveys. Soil saturation conditions were necessary to 252 successfully complete the survey and reveal a single concentric low resistance anomaly (outer ditch) and a central pit-like low resistance anomaly (Figure 10). The latter 253 254 proved to be an unusual triple cist burial. A higher capacity of these features to retain 255 water explains their detection. This may have been triggered by a higher organic matter 256 content inside these features.

257

In the case of the outer ditch, a discrete LOI peak of the uppermost ditch deposits

(deposit 1, **Figure 12**) may reflect an increased biomass of crops roots at this depth,

260 hence the higher moisture retention. This seems coherent with the depleted total P, K,

and Ca values of ditch samples collected at the same depth (Figure 11), as result of a
higher uptake by crop roots at this point.

263

264 The quadrature response of the FDEM survey produced a similar anomaly to the earth 265 resistance technique: a subtle single concentric conductivity trend (the outer ditch) and a central pit-like anomaly (Figure 10). The higher ground moisture content after the 266 heavy rain before the survey could have increased the contrast in the same manner as in 267 268 the earth resistance survey. The in-phase response was also very weak and the trend 269 again showed a single circular anomaly. Although the responses of both components 270 were fairly weak, the quadrature component in vertical mode of the FDEM survey 271 demonstrated potential in identifying archaeological features expected at c. 0.5–1 m, 272 since the noise created by the plough layer was outside the maximum sensitivity range 273 of the instrument.

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275 The GPR survey produced the most informative results, as it gave depth estimation, 276 high resolution mapping, and an approximate truncation level of the ditches. The survey 277 revealed two concentric high amplitude anomalies (outer and inner ditches), both visible 278 in the reflection profiles and the time-slices (Figure 10). The outer ditch showed strong 279 reflections towards the base and innermost side of the ditch cut. Here, the cut of the 280 ditch lies at a greater angle, and the sands and gravels were more cemented or indurated. 281 The strong high amplitude reflections of the outer ditch correlates with this sudden 282 change in soil texture. The samples taken over the outer ditch showed a greater variation 283 in chemical composition, particularly at the innermost side of the ditch. This type of 284 response may reflect a mineralogical change given the presence of the ditch, and a

similar response was also observed in Chesterhall Parks.

286 4. DISCUSSION

From the results of this combined approach at the three study sites, some general
observations relating to specific effects of the soil environment on the geophysical
results are considered in this section.

290

291 Heterogeneous glacial drift deposits

292 Many of the superficial deposits that form the parent material of contemporary Scottish 293 soils are characterised by Quaternary glacial drift deposits. These contain, inter alia, 294 mixtures of sands, gravels, silts and weathered rocks carried by glaciers and dropped as 295 the ice sheet advanced or receded. These extremely heterogeneous deposits also reflect 296 the great diversity of rock types that characterise the geology in Scotland. The sites 297 studied in this investigation lie over sedimentary rock belonging to different groups of 298 the Old Red Sandstone formation (ORS), which is widespread in Scotland, some 299 containing volcanic rocks between their varied components. At these sites, the bedrock 300 did not directly hamper the geophysical detection of the targeted features. However, the 301 glacial drift superficial deposits of Chesterhall Parks and Forteviot seem to have a more 302 significant effect on the different datasets, particularly those from the gradiometer 303 surveys. These very heterogeneous deposits may contain a great variety of rock debris 304 that may add background noise, mask the contrast for magnetic techniques or contribute 305 to the detection of negative magnetic anomalies (e.g., Forteviot). 306

307 **Table 4** shows a summary of the results of some of the soil topsoil analyses carried out 308 at the study sites. The brown earth and clay soil from Forteviot and Chesterhall 309 respectively are developed from reddish and coarse sands and gravels with variable 310 amounts of sands, silts and clay. ORS rock types are characterised by hematite (α Fe₂O₃) 311 as the main Fe-oxide, which gives the rocks their characteristic red colour (Wilson, 312 1971). The diversity of igneous and metamorphic rock components that ORS 313 formations may contain also contributes to the high variability in MS of the parent 314 material from which soil develops in different areas of Scotland. From the means taken 315 at the case study sites, the highest MS values are from the coarse and acidic brown earth 316 from Forteviot. Such coarse and freely draining texture and acidic pH are characteristic 317 of Scottish soils. However, the clay soil at Chesterhall Parks Farm and the alkaline soils 318 at the Bay of Skaill were both characterised by fairly low MS values (Error! Reference 319 source not found.).

320

321 Glacial geomorphology

322 The glacial past also had an important role in shaping the present day Scottish landscape 323 by developing intricate combinations of erosional and depositional geomorphological 324 features and landforms. This is illustrated at the sites surveyed in this study: outwash 325 terraces in Forteviot and hillslope solifluction processes in Chesterhall Parks Farm 326 (Table 1). These landforms may have an effect on the results of the surveys by 327 introducing clusters of magnetic noise (as in the case of Chesterhall Parks Farm) or 328 adding a high signal variability in the datasets. Geomorphological features associated 329 with glacier landscapes (e.g., kames, eskers, drumlins or ice wedges) may be taken into 330 consideration when interpreting geophysical datasets of Scottish sites. Solutions to 331 distinguish these landscape features from those of archaeological interest could include 332 soil chemical characterisation, in a similar combined manner as used in this study but at 333 a larger scale.

334

335 Soil additions and translocations

336 Archaeologically rich coastal areas in Scotland are often characterised by sites 337 concealed under high-energy deposits of fine-grained sands developed by aeolian 338 processes, as illustrated at the Bay of Skaill. These sands are expected to have high 339 concentration of Ca due to the calcium carbonate of the shells from which they were 340 partially derived and the generally low MS values (Table 4). The low MS values are 341 due to the main constituents of the sands, silica (SiO2) in the form of quartz, and 342 calcium carbonate (CaCO3), which are diamagnetic materials (Maher & Hounslow, 343 1999; Moskowitz, 1991). Cut features presenting enhanced MS fill deposits may be detected, for example, with a gradiometer survey. However, archaeological features 344 345 covered under thick aeolian deposits may be out of reach for this technique. The surveys 346 carried out at the Bay of Skaill demonstrate the potential of GPR and FDEM survey to 347 detect structural features in relatively deep wind-blown sands. Although the gradiometer 348 did not detect any structural features at the site, it proved useful in identifying the 349 midden deposits located near the Viking long-house. Whilst natural mounds may show 350 homogenous and magnetically quiet sand deposits, anthropogenic mounds may be 351 expected to be magnetically noisy. Therefore, this technique can still be useful in 352 exploring mounds of potential archaeological importance by identifying magnetically 353 noisy areas associated with anthropogenic deposits and thus revealing former human 354 occupation.

- 355
- 356 Soil processes may develop localised hill/terrace slope deposits, such as the soliflucted

fine-grained clays and colluvial volcaniclastic material at Chesterhall Parks Farm, that may not correspond to the deposits indicated in soil maps or available databases. These deposits are the result of the gradual accumulation of fine weathered material that moved slowly downslope from the nearby Tinto Hill, induced by gravitational forces on saturated sediments and as a result of Periglacial/Postglacial conditions. The deep clay at this site explains the frequent waterlogged conditions at a site mapped as freely

- draining brown earth in the soil maps. Despite the high conductivity of clay soils and
 the magnetic noise introduced by the igneous material, the targeted enclosures at the site
 were detected to a relative degree by geophysical means.
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367 Soil texture & related water content capacity

Coarse sandy soils drain water more quickly than finer clayey soils. Arable coarse soils with a thin litter layer overlying a freely draining sand or gravel subsoil can produce very high resistance values, which may not be suitable for earth resistance surveys during the Scottish summer time, as was the case at Forteviot. Soil textural variations in the cementation of the sands and gravels of the innermost cut of the ditch may have also contributed to the strong GPR reflections detected in Forteviot.

374

The higher number of micropores in clay soils cause retention of water, whilst the lack of macropores limit water infiltration rates, causing waterlogged conditions (Schaetz & Anderson, 2005). Although such conditions can saturate and mask soil conductivity contrast, the GPR (particularly with the low frequency antenna) and FDEM survey (quadrature in vertical mode) carried out at Chesterhall Parks Farm provided some results. This was possible given the thickness and shallow buried ditches.

381

382 Fe-content & related MS

383 Determination of the iron content in soil, if combined with MS analysis, may give an 384 idea of the magnetic background and state of the iron oxides present at the survey 385 environment. Table 4 shows the relatively high Fe concentrations in topsoil samples in 386 the case study of Forteviot and Chesterhall Parks Farm sites. This may reflect the impact of the acidic parent material from which these soils develop (Phillips, 2007). 387 388 However, high Fe concentration in soil does not always correlate with high MS; at 389 Chesterhall Parks Farm, despite high Fe contents, the MS values are fairly low due to 390 the frequently waterlogged conditions at this site. However, more important is the 391 determination of Fe and MS of topsoil, subsoil and archaeological deposits because this 392 helps to elucidate the complex factors of contrast and detection of archaeological 393 features at the sites. For example, these analyses were fundamental in understanding the

- 394 magnetic reversal of the ditches detected at Forteviot.
- 395

396 Organic matter & related water content capacity

Variation in water retention was the main factor for contrasts in conductivity properties
 of the targeted features and their consequent detection with the earth resistance, FDEM

and GPR surveys at the three sites. At Forteviot and Bay of Skaill, the capability of the

400 targeted features to retain water correlates with their higher organic content. In soils,

- 401 water retention increases with increasing clay content and organic matter because of the
- 402 affinities of water for those solids (Schaetz & Anderson, 2005).

404 **Organic matter, water content & related chemical transformations**

405 Although the chemical analytical techniques used in this investigation (pXRF and ICP-406 OES) did not identify chemical forms, they allow an estimate of soil mineralogical 407 transformations involved in the contrast and type of geophysical response detected at 408 several sites. Chemical transformations can be triggered by organic matter accumulation 409 and water retention in soils. For example, reductive dissolution of Mn and Fe oxides can 410 occur due to the decomposition of organic matter and contribute to the release of metals 411 and magnetisation of sediments (Lovley, 1991; Orgeira & Compagnucci, 2006). 412 According to Weston (2002), prolonged waterlogged conditions can transform ferric 413 material to less magnetic and more dissolvable ferrous forms. The effect of reductive 414 dissolution of these oxides has also been suggested as the cause of Fe and Mn 415 depletions in archaeological floor deposits (Oonk, Slomp, Huisman, & Vriend, 2009). 416 The depleted values in Mn, Fe and MS seen in this study in association with ditch 417 features in Forteviot are likely to be the results of reduction dissolution processes and to 418 have contributed to the detection of negative magnetic anomalies. The higher organic 419 content and related higher capacity of the outer ditch to retain water, seen also in the 420 depletion of Mn and in the LOI peak (Figure 11), may represent a discrete reductive 421 zone that may favour dissolution of Fe-and Mn oxides and consequent lower 422 susceptibilities.

423 5. CONCLUSIONS

424 The study demonstrates that, by applying both geophysical and geochemical methods, 425 'traditionally difficult' survey environments for some geophysical techniques can be 426 prospected in a satisfactory manner. This combined approach can be very valuable in 427 order to provide a more reflective analysis about how local geological and pedological 428 settings influence the geophysical results. For example, many archaeological survey 429 environments in Scotland are characterised by highly variable drift soil deposits that 430 originated from the effect of past glacial processes, weathering a great diversity of rock 431 types. In such conditions, surveys based on a single-technique (e.g., magnetometer 432 surveys) have a high chance of being disappointing given the subtle contrast of 433 magnetic properties and unanticipated magnetic signatures as seen in Forteviot. In the 434 case of aeolian environments, these sand deposits characterise many archaeologically 435 rich coastal areas in Scotland. This investigation shows that anthropogenic organic 436 deposits related to past human occupation can be detectable by sometimes unexpected 437 geophysical techniques, as in the case of the Bay of Skaill.

438

439 The study shows that a detailed analysis of particular archaeological features can help to 440 identify specific soil properties and processes, inside or outside the features, which are 441 behind the contrast detected (or not) by the different geophysical techniques. Successful 442 or failed detection of the targets by geophysical means involves 'case specific' variables 443 such as soil properties, ground surface and temporal climatic variations, and depth of 444 burial of archaeological features. The investigation showed that different types of 445 archaeological features (a wall foundation or a ditch) can provide unpredicted 446 geophysical signatures given the effect of complex contrast dynamics and post-447 depositional processes that develop inside features. The effects of soil materials and 448 properties, such as texture (grain size and cementation), and particularly organic matter

- 449 content and its related water retention capabilities, were fundamental to resolve the
- 450 targets with electrical and electromagnetic methods. The composition of soil parent
- 451 material and chemical transformations triggered by organic matter accumulation (e.g.,
- 452 reduction dissolution of Fe- and Mn-oxides) can influence and be fundamental in
- 453 understanding the results of magnetic methods.
- 454

The study also allowed a detailed observation of the geochemical signatures of shallow
anthropogenic targets from topsoil samples and fill deposits. Some of these signatures
included rather unusual responses, such as general depletions of typical crop
macronutrients (e.g., P, K, Ca) and a high variability of the chemical concentrations of
major elements obtained from the ditch features.

460

461 To summarise, the integration of geochemical soil analysis as part of geophysical
462 prospection can contribute to an understanding of how soil settings of a site may affect
463 the results of different geophysical techniques and thus allow improved interpretations.
464 The drawback of such combined approach is that it is time-consuming and demands
465 expertise.

466

467 In the current framework of fast-moving geophysical surveys, research efforts leading to 468 technique reappraisal and improved data interpretation should encompass large-scale 469 surveys and data production. To do this, well-thought-out soil sampling strategies 470 should be coordinated within geophysical surveys taking into account the time and 471 skilled personnel required to sample in an accurate manner. Additionally, the correct 472 storage of soil samples and soil processing before analysis should be carefully planned. 473 Features exposed during archaeological excavations provide a unique opportunity not 474 only to validate the geophysical results but also to explore particular responses through 475 soil analysis in a similar manner as shown in this study. In return, the results can 476 contribute to enhanced archaeological interpretation of such features. Therefore, closer 477 collaboration among archaeologists should be sought in order to gain access to exposed 478 features for soil sampling and measurements.

479

480 In the wide variety of soil systems, there is not a single factor that can explain the481 detection of geophysical anomalies of archaeological significance, nor is there a

- 482 "magic" technique able to detect them regardless of the soil environment. Soil materials,
- 483 properties and post-burial soil formation processes are inherently related to
- 484 archaeological features and their proxy detection, and they have to be considered
- 485 simultaneously. Despite the extra effort in acquiring and analysing complementary soil
- 486 data, it is crucial to continue developing strategies to incorporate soil analysis within
- 487 large scale geophysical surveys in order to improve data interpretation and advance in
- 488 the discipline of archaeological geophysics in a balanced manner.

489 ACKNOWLEDGEMENTS

490 This research was funded by the Natural Environment Research Council awarded to the

- 491 author as part of a PhD study carried out between 2008-2012 at the University of
- 492 Glasgow. The fieldwork was supported by an equipment loan from the NERC
- 493 Geophysical Equipment Facility (loan number 912). The author appreciates the
- 494 comments provided by the anonymous reviewers.

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590 Tables

591 Table 1: Archaeological targets and general settings at the study sites

Site	Challenge	Target	Bedrock	Superficia l Deposits	Soil	Geomo rpholo gy	Fie ld Us e
Bay of Skaill (Orkne y)	 Viking longhouse concealed under deep windblown sands. Gradiometer surveys did not detect any structural anomaly indicative of the longhouse. 	 Linear Stone structure. N-S direction. ~1 m wide. Deep: revealed at least 1 m deep during trial trenching. 	Sedimentary (Lower Stromness Flagstone-Old Red Sandstone, mid- Devonian): laminated, carbonate-rich siltstones and shales with fine-grained and thinly bedded sandstones	Pale brown and fine- grained windblow n sands (Quaternar y)	Well drained and deep calcareo us Regosols (Fraserb urgh series)	Mound of free sand	Pas tur e
Cheste rhall Parks Farm (South Lanar kshire)	 Grassmarks evidence of Iron Age ditch enclosures buried in highly conductive soil environmen t. The results of gradiometer and earth resistance survey were inconclusiv e in resolving 	 Concentri c ditched enclosures Deep ditches (~up to 1 m deep. Shallow: uppermost fill deposits were revealed to be fairly shallow during trial trenching. 	Sedimentary (Wiston Grey Volcaniclastic Sandstone-Old Red Sandstone, early Devonian): fine-to coarse- grained volcaniclastic sandstones of greenish-grey ashy colour (Browne et al. 2002)	Clyde Valley fluvioglaci al deposits (Quaternar y)	Deep and poorly drained heavy clay (confirm ed by test trench excavati ons (Sharpe, 2004)	Outwas h and river terrace	Pas tur e and ver y occ asi ona lly plo ug hed (la nd ow ner co m me nt)

	the enclosures.						
Fortevi ot (Perths hire)	- Cropmarks evidence of Neolithic to the Bronze Age henge enclosures.	- Shallow	Sedimentary (Arbuthnott- Garvock Group-Old Red Sandstone): sandstones, conglomerates, shales, mudstones and volcanic rocks (Browne et al. 2002)	Glacio- fluvial sand and gravel deposits (Quaternar y)	Well drained reddish- brown earths (Gleneag les series)	Solifluc tion terrace	Ar abl e (ba rle y cro ps at the tim e of this stu dy)

Table 2: Summary of the geophysical techniques and surveys parameters used at the study sites

		Sampling			
Technique	Instrument	Bay of Skaill	Chesterhall Parks	Forteviot	
Gradiometry	Bartington Grad 601-2	0.5 m traverse & 0.125 m in- line (parallel mode)	0.5 m traverse & 0.125 m in- line (parallel mode)	 -0.5 m traverse & 0.125 m in-line (parallel mode). -0.25 m traverse & 0.125 m in-line (parallel). 	
Ground Penetrating Radar (GPR)	Sensors & Software PulseEKKO 1000	Single GPR lines (450 and 225 MHz): 0.05 m and 0.10 m in-line, time- window=80 ns and 120 ns	Single GPR lines (450 and 225 MHz): 0.05 m and 0.10 m in- line, time- window=60 ns and 100 ns	 High resolution survey (450 MHz): 0.25 m traverse & 0.05 m in-line, parallel & step mode, time window=150 ns, stacks=16, samples=200 ps. Single GPR lines using 450 MHz: 0.05 in-line, time-window=60 ns. 	
Frequency Domain Electromagnetics (FDEM)	Geonics EM 38 (+GPS)	1 m traverse (parallel, vertical dipole, in- phase & quadrature component)	1 m traverse (parallel, vertical dipole, in- phase & quadrature component)	1 m traverse spacing (parallel, vertical dipole, in-phase & quadrature component)	

Earth Resistance	Geoscan RM15	0.5 m traverse & 0.5 m in- line (1 m probe spacing) (zigzag mode)	0.5 m traverse & 0.5 m in- line (0.5 probe spacing, zigzag mode)	0.5 m traverse & 0.5 m in- line (0.5 & 1 m probe spacing, zigzag mode)
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Table 3: Geochemical methods used to analyse the soil samples collected at the study sites.

Analysis	Digestion	Procedure	Instrument
Total (Inorganic) Phosphate	Ignition- HCl	 Molybdenum blue colorimetric method (SASSA) 	Fisherbrand colorimeter (Archaeology, University of Glasgow)
XRF	Not required	 Multi-element concentration analysis of 33 elements: Mo, Zr, Sr, U, Rb, Th, Pb, Se, As, Hg, Au, Zn, W, Cu, Ni, Co, Fe, Mn, Cr, V, Ti, Sc, Ca, K, S, Ba, Cs, Te, Sb, Sn, Cd, Ag, Pd Soil samples were transferred into a plastic cup and covered with polypropylene X-ray film TF-240 FLUXANA. A shielded lead box stand was used for protection. A helium purge device was used to increase instrument sensitivity to lighter elements. Each sample was measured for <i>c</i>.2 minutes. Measurements were repeated after slightly rotating the sample cups and were averaged. Calibration checks were done using two international standards (TILL-4 and NIST 2780), a random sample and a blank sample. The accuracy test gave generally good results for the majority of elements except for S, V and Rb. The concentrations of V and Rb were found to be slightly overestimated. 	Thermo Scientific Niton XL3t GOLDD (Archaeology, University of Glasgow)
ICP-OES	HF/HCl/HN O3 mixture using a CEM Mars Xpress microwave digestion system	 Multi-element concentration analysis of 18 elements: Al, Fe, Na, K, Ca, Mg, Ti, P, Mn, Ba, Sr, Pb, Cu, Zn, Cr, Ni, Li, and Co Measurements were evaluated using an external calibration and scandium as an internal standard. Certified reference material (MESS-3) was used to monitor the quality of the analytical process. 	Varian Vista Pro (Scottish Alliance for Geosciences, Environment and Society- SAGES facility,

		 With each batch of samples, two procedural blanks were run to control blank levels and variability. 	University of Edinburgh)
Magnetic Susceptibili ty	Not required	 The samples were transferred into pre-weight 10 cm3 plastic cups. Samples were weighed in order to calculate mass-specific (χ) MS. Before the measurements, the instrument was left to stabilise. Samples were measured at low frequency (χlf=0.46 kHz) and at high frequency (χhf=4.6 kHz) to calculate frequency-dependent mass susceptibly (χfd) in percentage. MultiSus software was used to take the measurements. Instrument calibration and accuracy was assessed by measuring two standard samples, ST1 (ferrimagnetic) and H₂O. 	Bartington MS2B (University of Glasgow)

Table 4: Summary of the mean results of the physical and chemical analysis of the topsoil samples collected at the three case study sites. Most of the results show the in-site and off-site (controls) values. A * signifies not analysed.

		Bay of Skaill		Chesterhall Parks Farm		Forteviot	
s	lite	Viking Settlement (positive/impervious feature)		Prehistoric Enclosure (cropmark)		Prehistoric Enclosure (cropmark)	
S	boil	Calcareou	s Regosol	Clay		Brown earth	
S	oil Texture	Fine-grain with som fragm	ne shell	Clay with some gravels		Stony sandy loam	
р	H (In-site)	9 (Strong a	alkalinity)	6 (Medium acidity) to 5 (Strong acidity)		6.5 (Very slight acidity)	
F	EC (µs) (In-site)	10	00	107		54	
L	LOI (%) (In-site)	0.0	61	0.031		0.029	
I	n/Off-site	In	Off	In	Off	In	Off
Т	fotal P (µg/kg)	155		99	125	243	271
pXRF (mg/kg)	Fe	11 933	*	23 995	22 182	26 416	26 015
шg	Mn	308	*	287	478	791	749
F (j	Ti	1 456	*	4 944	4 747	5 653	5 336
XR	K	13 955	*	10 133	12 545	13 189	12 608
	Ca	14 8313	*	7 573	5 256	9 416	8 518
Ν	$MS (10^{-8} \text{m}^3 \text{kg}^{-1})$	7	*	21	48	144	151

600 Figures

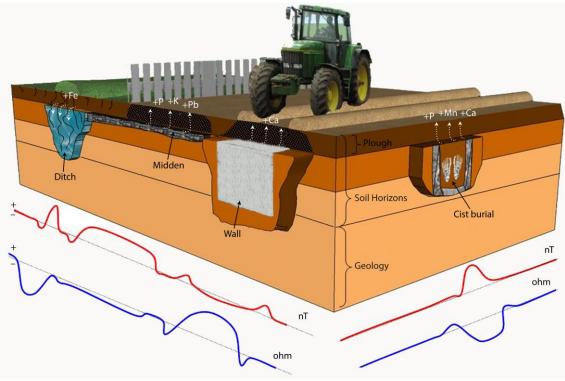


Figure 1: Diagram showing idealised geophysical and geochemical responses of common archaeological features.
 The magnetic response is shown in red and the earth resistance in blue (© Carmen Cuenca-Garcia).

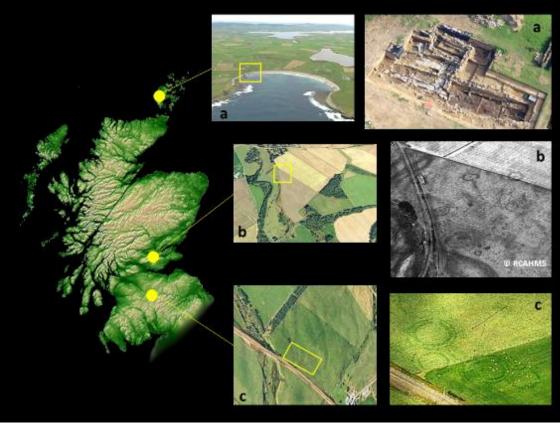
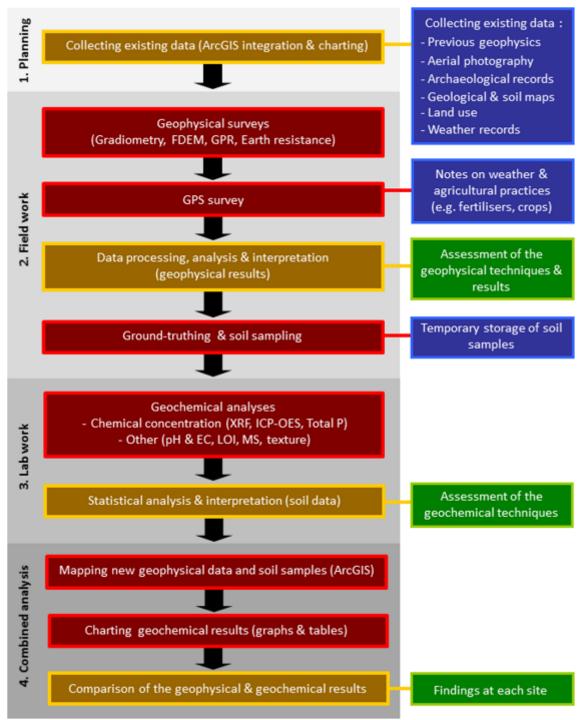


Figure 2: Location of the three study sites and general views of the survey areas (a: Bay of Skaill, Orkney, b: Forteviot, Perthshire, c: Chesterhall Parks Farm, South Lanarkshire)





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Figure 3: Diagram showing the combined research strategy in four phases. Geophysical, geochemical and other methods are in red, analytical stages are in brown, 'satellite' data collection is in blue, and the expected outcomes are in green. The acronyms are defined as follows: frequency domain electromagnetic (FDEM), ground-penetrating radar (GPR), global positioning system (GPS), X-ray fluorescence (XRF), inductively coupled plasma optical emission spectrometry (ICP-OES), total phosphate (Total P), electrical conductivity (EC), loss on ignition (LOI) and magnetic susceptibility (MS).

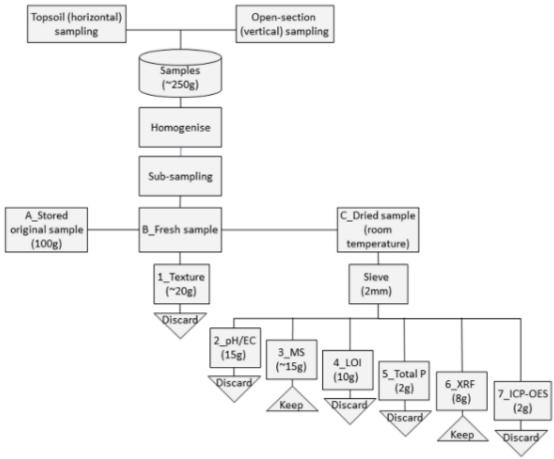


Figure 4: Flowchart showing the plan followed to carry out the soil analyses. The different analyses were performed
following the Soil Analysis Support System for Archaeology-<u>SASSA</u> (University of Stirling) standard analytical
methods and other adapted protocols from the Department of Archaeology (University of Glasgow) and the Scottish
Alliance for Geosciences, Environment and Society-SAGES (University of Edinburgh). The acronyms are defined as
follows: electrical conductivity (EC), magnetic susceptibility (MS), loss on ignition (LOI), total phosphate (Total P), Xray fluorescence (XRF) and inductively coupled plasma optical emission spectrometry (ICP-OES).

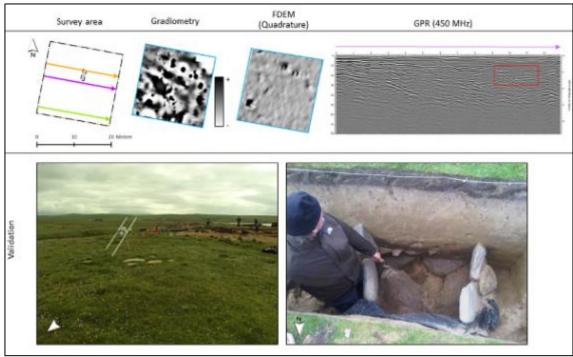
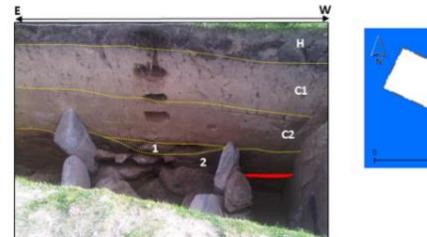
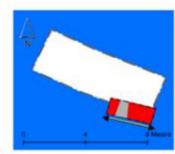


Figure 5: Results of the multi-technique geophysical survey at the Bay of Skaill, survey area and excavated target. The coloured arrows within the survey area show the location of the collected GPR profiles. The gradiometer data were plotted at 10 (black)/-10 (white) nT. The FDEM (quadrature) plot shows higher and lower values (mS/m) in black and white, respectively. High and low amplitudes in the GPR reflection profile are shaded in black and white, respectively.





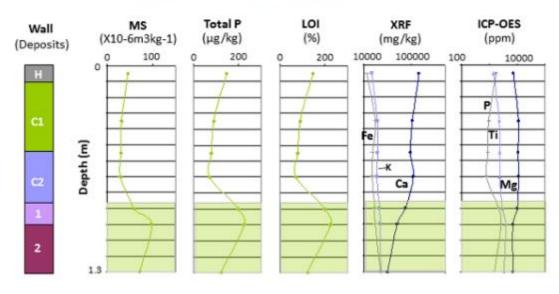


Figure 6: Selected results of the soil analyses of the samples collected from the exposed north facing section at the Bay of Skaill. The picture of the exposed section (top left) shows the location of a weakly cemented iron-pan deposit (in red).

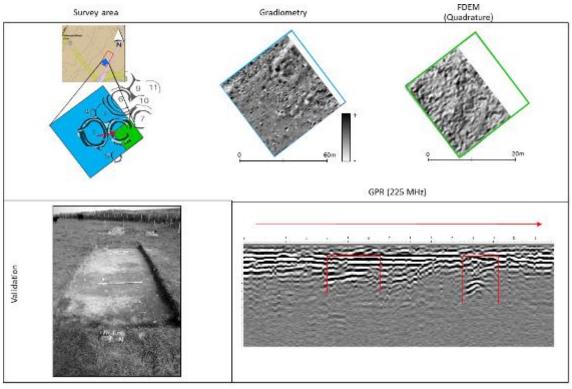


Figure 7: Results of the multi-technique geophysical survey at Chesterhall Parks Farm. The picture (bottom left) shows the ditch (enclosure 2) exposed during previous test trench excavations (L. Sharpe photographic archive). The gradiometer data were plotted at 10 (black)/-10 (white) nT. The FDEM (quadrature) plot shows higher and lower values (mS/m) in black and white, respectively. High and low amplitudes in the GPR reflection profile are shaded in black and white, respectively.

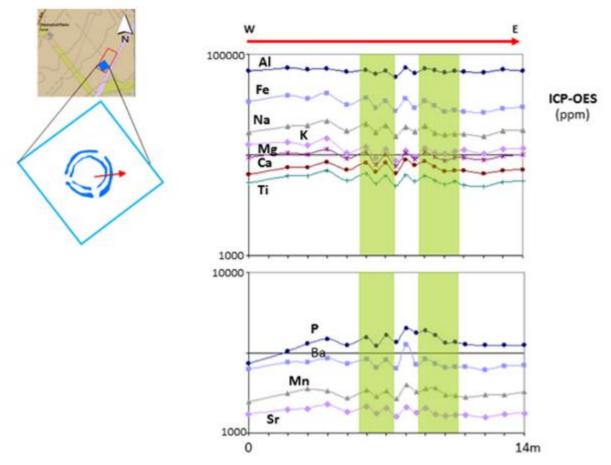
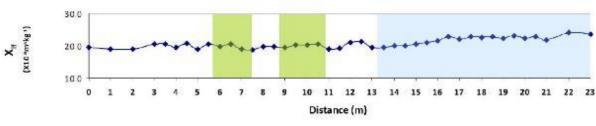


Figure 8: Topsoil sampling and ICP-OES results of a sampling line collected over the ditched enclosure 1 (Figure 7) at Chesterhall Parks Farm. The coloured bars mark the location of the ditches detected with the gradiometer survey.

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 $\begin{array}{l} \text{Distance (m)} \\ 652 \\ 652 \\ 653 \end{array}$ Figure 9: Surface distribution of magnetic susceptibility (χ_{ij}) of the soil samples collected at Chesterhall Parks Farm. $\begin{array}{l} 653 \\ 654 \end{array}$ $\begin{array}{l} \text{Distance (m)} \\ \text{Figure 9: Surface distribution of magnetic susceptibility (} \chi_{ij}) of the soil samples collected at Chesterhall Parks Farm. \\ \text{for any other of the ditch anomalies relating enclosure 1 (in green) and enclosure 2 (in blue). \\ \text{for any other of the ditch anomalies relating enclosure 1 (in green) and enclosure 2 (in blue). \\ \text{for any other of the ditch anomalies relating enclosure 1 (in green) and enclosure 2 (in blue). \\ \text{for any other of the ditch anomalies relating enclosure 1 (in green) and enclosure 2 (in blue). \\ \text{for any other of the ditch anomalies relating enclosure 1 (in green) and enclosure 2 (in blue). \\ \text{for any other of the ditch anomalies relating enclosure 1 (in green) and enclosure 2 (in blue). \\ \text{for any other of the ditch anomalies relating enclosure 1 (in green) and enclosure 2 (in blue). \\ \text{for any other of the ditch anomalies relating enclosure 1 (in green) and enclosure 2 (in blue). \\ \text{for any other of the ditch anomalies relating enclosure 1 (in green) and enclosure 2 (in blue). \\ \text{for any other of the ditch anomalies relating enclosure 1 (in green) and enclosure 2 (in blue). \\ \text{for any other of the ditch anomalies relating enclosure 1 (in green) and enclosure 2 (in blue). \\ \text{for any other of the ditch anomalies relating enclosure 1 (in green) and enclosure 2 (in blue). } \\ \text{for any other of the ditch and the ditch and the ditch anomalies relating enclosure 1 (in green) and enclosure 2 (in blue). } \\ \text{for any other of the ditch anomalies relating enclosure 1 (in green) and enclosure 2 (in blue). } \\ \text{for any other of the ditch anomalies relating enclosure 1 (in green) and enclosure 2 (in blue). } \\ \text{for any other of the ditch anomalies relating enclosure 1 (in green) and enclosure 2 (in blue). } \\ \text{for any other of the ditch anomalies relating enclosure 2 (in$

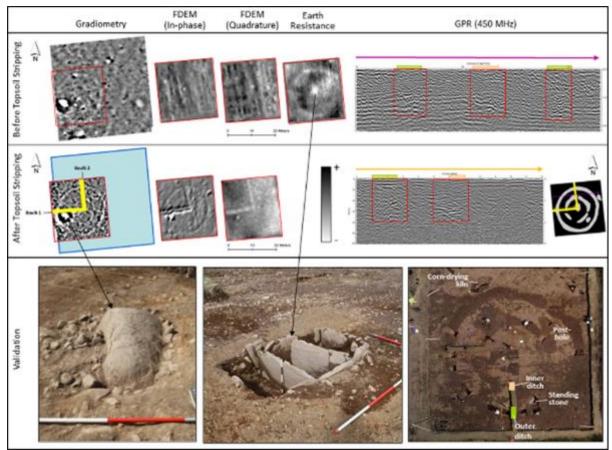
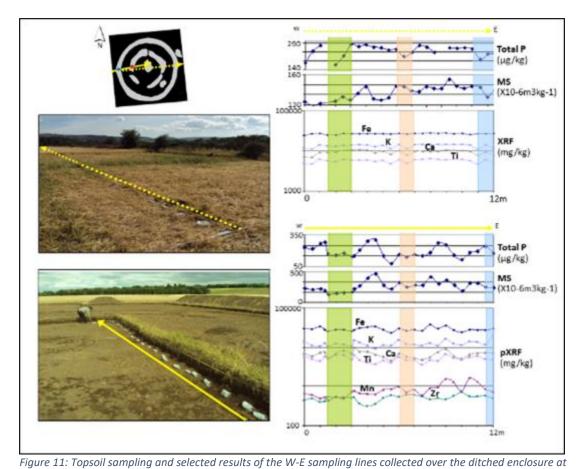


Figure 10: Results of the multi-technique geophysical survey (before and after topsoil stripping) and related archaeological features confirmed during the excavation at Forteviot. Following topsoil stripping, the exposed ditch deposits were mapped using a differential GPS, and their locations are indicated in the GPR reflection profiles and aerial photograph with the coloured bars. The gradiometer data were plotted at 10 (black)/-10 (white) nT. The FDEM (quadrature) plots show higher and lower values (mS/m) in black and white, respectively. High and low amplitudes in the GPR reflection profiles are shaded in black and white, respectively.



Forteviot before and after topsoil stripping (dotted and solid yellow arrows). The coloured bars mark the location of

the outer (left) and inner (central) ditches and the triple cist burial (right).

