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Phosphorus, stable isotopes and fatty acids of soils as tools for recognizing a Bronze Age droveway in the *Valli Grandi Veronesi* (North Italy)

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Abstract

This study focused on the surroundings of the *terramara* of Castello del Tartaro in the lowland of Valli Grandi Veronesi (North Italy) outside a Bronze Age village enclosed by channels and embankments, where two groups of double parallel side drainage ditches running from northwest to southeast were found. This evidence has been interpreted as a "Big Road" and more precisely a droveway, possibly used for the passage of cattle. The road was sampled along a section, at 4 different levels of depth, and the content in inorganic and organic phosphorous was assessed. In the sampled section the content of phosphorus shows the presence of discontinuities and strong phosphorus accumulation in the layers under the arable land. The analysis of the stable isotopes of carbon and nitrogen attests to traces of herbivores, while the comprehensive two-dimensional gas chromatography analysis identified a high number of significant fatty acids suggesting the presence of fats from ruminant animals. The results, as a whole, corroborate the hypothesis of a droveway. This hypothesis agrees with both paleobotanical and zooarcheological data from the territory around the embanked settlements and underlines the importance of cattle husbandry within the complex relationships of the Bronze Age world.

Keywords: Ancient road, Animal fats, FAME, Gas chromatography, Inorganic and organic phosphorus, Terramare

Introduction

1.1 Geomorphological and archaeological framework of the area under study

The Valli Grandi Veronesi occupy a morphologically depressed area between the Adige River to the northeast and the Po River to the south. Before the reclamation period, the area was marshy and vulnerable to easy dumping, due to the presence of numerous resurgences and spring rivers. The Tartar River, which flows through them and gave them their name in ancient times (*paludes Tartari fluminis*), is precisely a resurgent river.
During the Middle Bronze Age (*Bronzo Medio* or BM, from the seventeenth to the fourteenth century BC) and the Recent Bronze Age (*Bronzo Recente* or BR, from the thirteenth to the first half of the twelfth century) in the Po Plain in general, and also in the Valli Grandi Veronesi, a new type of settlement, known as *terramara*, emerges. The *terramare* are central places located on fluvial terraces, surrounded by canals and embankments that combine defensive functions with the control of water drainage (Bernabò Brea et al., 1997; Pearce 1998). Several *terramare* (also called embankment villages) developed in the Valli Grandi.
In the economy of the embankment villages, agriculture was practised, but also animal rearing was important. Regarding the former, *Avena sativa* (oats; Carra, 2013; De Grossi Mazzorin and Solinas, 2013) and millet (Tafuri et al., 2009) were probably consumed by both humans and livestock, as suggested by stable isotope analyses at Olmo di Nogara, an important cemetery in the Valli Grandi Veronesi (Tafuri et al., 2009). Zooarchaeological studies (De Grossi Mazzorin, 2015; De Grossi Mazzorin and Riedel, 1997; Riedel, 1994) have pointed to the prevalence of sheep and goats, a dearth of cattle, a moderate amount of pig and a significant presence of dogs (relevant for the possible connection with pastoral activities) in the *terramare* south of the Po, and the prevalence of sheep and scarcity of goats in the *terramare* north of the Po. In lowland sites, rich in ponds and winter grass, such as Tombola, Le Vallette (Cerea) Larda I and II, Campestrin and Amolara (Polesine), sheep and goats outnumber pigs and cattle during the BR (Bertolini et al., 2015). The precocious presence of donkeys (during the late Bronze Age at Bovolone, Fondo Paviani e Frattesina) and horses (since the mid-seventeenth century BC in the whole Veneto) might suggest their possible role in shepherds' mobility, as they proved to be widely used as pack animals by traditional pastoral groups in the modern and pre-modern period (De Grossi Mazzorin, 2015; Bertolini et al., 2015; De Grossi Mazzorin and Riedel, 1997; Riedel, 1994; Sauro et al., 2013).

1.2 The "Big Road"

The *terramara* of Castello del Tartaro, (Fig. 1), built on a natural mound at an altitude of 8.5 meters above sea level along the bed of the River Tregnone, was the core feature of a prehistoric rural landscape where concentric drainage systems, connected by transverse ditches, were probably used to water the fields and the meadows around the settlement (Cima, 2012).

Figure 1

In this carefully organized landscape, archaeological surveys and aerial-photo interpretation directed by Armando De Guio (University of Padua) (De Guio et al., 2015) in the surroundings of the *terramara* of Castello del Tartaro identified in the Costantini *Fondo*, Comune of Cerea (VR), two groups of double parallel drainage ditches running from northwest to southeast (Fig. 2).

Figure 2

The two groups of drainage ditches begin from the surroundings of the Terramara of Castello del Tartaro and finish in the road on the Southern Embankment known as *Strada su Argine Meridionale* (SAM) north of the course of the Tregnone Paleocanale. They enclose a space 30-40 m wide, and 1.2 km long. This evidence has been interpreted as a road (the "Big Road") and more precisely as a droveway, possibly used for the passage of cattle (Betto, 2013; De Guio et al., 2015). Similar evidences can be found in the prehistory of southern England (Yates, 2007), the canonical example being that of Fengate / Flag Fen (Pryor, 2006).

Figure 3

The interpretation as a droveway fits well with the reconstructed landscape surrounding the Bronze -Age embankment villages, used at a significant percentage as herbaceous pasture with scattered trees and shrubs, as suggested by ethno-archaeological (Migliavacca, 1989), geoarchaeological (Balista et al., 2016) and palaeobotanical (Mercuri et al., 2006; Mercuri, 2009) studies, and by the evidence of agricultural and rearing practices described above. A quadrangular corral, which can be interpreted as an animal enclosure, was identified on the eastern side of the embankment: highlighted in fig.2. This could have been used both for housing cattle and especially oxen, valuable working animals that were the protagonists of the agrarian revolution of the Middle-Recent Bronze Age (Halstead 1995; 2014); and, in the winter, for housing sheep, part of which were driven on their way to the summer pastures, responding to the equally important revolution in livestock by-products (Sherrat 1981, 1983; Greenfield 2005, 2010; Sabatini 2022).

1.3 Residue analysis

Physico-chemical analyses of soil samples constitute a very promising area for detecting inorganic and organic residues also in archaeological sites. By analysing different classes of compounds, useful bio-markers for explaining the origin of materials discharged into soils and sediments can be identified. Notably, manure and disused waterways/landfills produce a variety of compounds (5β-stanols and secondary bile acids) which could be considered markers. (Pepe et al.1989; Pepe, Dizabo 1990; Bull et al., 2003). Lipids present in the excreta of most animals (and humans) are particularly persistent to decay and accordingly, such archaeological studies have also addressed the recovery of lipids as shown by their identification in different archaeological periods (Pepe et al., 1989; Pepe, Dizabo 1990; Evershed, 1993, Bull et al., 2003). Moreover, lipid profiles of different livestock species were used to identify various fecal inputs to archaeological sites where waste was disposed of. Among lipids, fatty acids exist in various forms: saturated (SFAs), monounsaturated (MUFAs) and polyunsaturated (PUFAs). MUFAs are generally abundant in fish and plants, whilst mammals have a high SFA content (Barnarb et al., 2007). More specifically, the analysis of fatty acid methyl esters by gas chromatography has proved very useful when applied to very small quantities of archaeological samples (Kedrowski et al., 2009). A number of studies have identified ancient fatty acids in archaeological materials such as organic adhesives (Regert, 2004), soil residues in hearths (Kedrowski et al., 2009, cemented organic residues (Heron et al., 2010), groundstones (Buonasera, 2007), and floor sediments (Kanthilatha et al., 2014).

In the context of inorganic bio-markers archaeologists have long studied phosphorus (P) accumulation as evidence of human and/or related activities (Leonardi et al., 1999; Holliday and Gartner, 2007). Specifically, at human or animal aggregation sites, the dropping of P-

containing organic wastes into the soil was common.
The degradation of organic P from residues produces different forms of orthophosphate depending on the soil pH. Phosphorus in alkaline and calcareous soils is generally found in the form of calcium phosphate or hydroxyapatite. The latter is the most stable P form and accordingly, it is resistant to redox processes and leaching. In addition, mineral P is accumulated at the site of build-up. P accumulation becomes relevant if compared to the P content of a soil supposedly not subjected to long-term anthropogenic land use (Leonardi et al., 1999). Inorganic phosphate and total phosphorus analyses were extensively used to predict the horizontal limits of inhabited areas (Conway, 1983; Pogue, 1988). Organic P analysis, on the other hand, has not been frequently applied, even though it may be an important marker. Another useful indicator of habitation and within-site variation in the scale of land use is the C enrichment, as it correlates significantly with P and lithic debris density (Schelezinger and Howes, 2000).
Stable isotope analysis is an established technique for the study of human and other ancient remains. Applications have grown considerably in recent years, with many studies focusing on the reconstruction of complex food webs (Bonnie et al., 2022).
Since stable isotopes are generally conserved in an ecosystem or transformed in a recognized way, they can be used to identify sources of archaeological material. A change in isotope ratio values accounts for a biochemical conversion of the corresponding source molecules, while constant values denote a "conservation" of the origin molecules (Lichtfouse et al., 1998).
The most useful isotopes for archaeology are those of carbon and nitrogen. The ratios of 13C to 12C can be used to describe the types of plants present in an ecosystem and consumed by its animal and human inhabitants (Nord and Billström, 2018). It is known that fractionation of 15N content in plants and soil occurs as a consequence of biological and physico-chemical processes (Hogberg, 1997). For instance, the nitrogen isotopic signatures of dietary ingested proteins are different from those of the organism because isotopic fractionation occurs in the organism itself. The isotopic value can be used as an indicator of an organism's position in the food chain. (Papathanasiou, 2015). The ratio of 15N to 14N increases in the food chain, such that carnivores have very different isotopic signals.

On the basis of the excavations, it was hypothesized that the "Big Road" was a major road access in the economy of the embankment villages, used for the passage of cattle.
The principal goal of the present study was to identify and quantify bio-markers, such as phosphorus content, carbon and nitrogen stable isotope analysis, and fatty acids profile in the soils of the two groups of trenches, in order to demonstrate anthropogenic activities related to the use of the “Big Road” as a cattle road or transit road for flocks.

2. Materials and Methods

2.1. Sampling

The natural “background” phosphate level was assessed by sampling both the arable land and the underlying layers of soil in two of the few areas where, as far as we know, there is no evidence of archaeological settlement. The samples related to the “Big Road” were collected from a stratigraphic profile 1 m deep, along an East-West oriented drain (Fig. 4).

Figure 4

Along the profile not only the present arable land, but also two drainage ditches with a concave shape, dug in the ground at the point corresponding to the traces of the "Big Road", were evident. The two trenches were dated to the Bronze Age, on account of the absence of fragments of bricks and cooked clay and their digging from the horizons of relict late holocene paleosoil. The hydromorphous characteristics of their fillings, mixed with the sandy substrate soil matrix, are different from the Iron Age alluvial layers which covered the Valli Grandi after the 9th century BC and have silty matrices (Betto 2013). Soil samples were collected at different depths from the drainage trenches that delimit the Big Road; soil samples were also collected from the central zone of the road (samples 8, 9 and 10). A trowel was used in order to collect the samples, avoiding obvious sources of contamination (such as roots and large stones). The soil samples were collected in sealed and initialled plastic bags.

2.2 Chemical analysis

Organic carbon (OC) and total nitrogen (TN) were determined by dry combustion on a elemental analyzer (CNS Vario Macro, Elementar, Hanau, Germany) with C-correction for inorganic carbon. Phosphorus (P) was determined by ignition and extraction with HCl (Kuo, 1996). The organic P (OP) was calculated as the difference between the HCl-extractable P of a sample ignited at 513 °K (total P) and the P extracted from the untreated sample (inorganic P, IP) (Legg and Black, 1955). For this, soil (0.5 g) was placed in a 50 mL Pyrex beaker, ignited (550 °C) for 1 h and cooled. After treatment with concentrated HCl and heating at 70 °C for 10 min, an additional amount of HCl (5 mL) was added. The sample was left at room temperature for 1 h before the suspension was filtered through filter paper (Whatman No. 42). P in the extract was analysed by inductively coupled plasma-optical emission spectroscopy (ICP-OES, Spectro Analytical Instruments, Kleve, Germany). ^{13}C and ^{15}N isotope ratio analysis was performed as previously described (Francioso et al., 2005).

Briefly, stable isotope ratios C and N were analysed on the same sample by using an Isotope Ratio Mass Spectrometer (IRMS, Delta V Advantage, Thermo Fisher Scientific). The isotopic composition of the sample was expressed as units of δ13C and δ15N using Peedee Belemite (PDB) and air atmosphere as standard for C and N respectively, and calculated using the following formula:

$$\delta^{13}X‰=[Rsample\text{-}Rstandard-1]\times 1000$$

where X = 13C or 15N and R = 13C/12C or 15N/14N

All analyses were performed in triplicate.

Lipids were extracted using accelerated solvent equipment (ASE; Thermo Fisher Scientific Inc., Waltham, MA, USA). Soil samples (10.0 g) were homogenized using Hydromatrix (Phenomenex, Castel Maggiore, Bologna, Italy) and petroleum/acetone (90/10 v/v) as solvents. The extraction conditions were: temperature 125 °C; pressure 10 MPa; static time 4 min; number of static cycles 3; rinse 100%; purge 60 s. The solvent was evaporated under N2 flux. The resulting extract was weighed and transferred into tubes for transesterification and methylation (Jenkins, 2010). Each extraction was performed in triplicate. The fatty acid methyl ester derivatives (FAME) spectrum of the obtained sample was analysed using a GC x GC instrument (Agilent 7890A, Agilent Technologies, Santa Clara, CA) with 2 columns in series with a modulator (Agilent G3486A CFT). Automatic (Agilent 7693) and flame-ionization detector (FID) were interfaced with chromatography data system software (Agilent Chem Station) (Cesaro et al., 2013). Between the two columns, a modulator unit collects the analyte bands of the first column in a fixed-volume channel, which are then coupled in a narrow band to a shorter second column. The operating conditions of the GC unit were: 1) 80 m x 180 µm x 0.14 µm film thickness first column (SP-2560, Supelco, Bellefonte, PA), H2 carrier gas flow rate of 0.25 ml/min; 2) 3.8 m x 250 µm x 0.25 µm film thickness second column (J&W, Agilent Technologies), H_2 carrier gas flow of 0.25 ml/min; 3) planned oven temperature variation: ramp from 40 °C (2 min hold) to 170 °C (hold for 25 min), then ramp to 250 °C at a rate of 2 °C/min (hold for 14 min); 4) valve: modulation delay 1.00 min; modulation period 3.0 s; sampling time 2.87 s; (5) flame-ionization detector: heater, 270 °C, H_2 carrier flow 20 mL/min, air flow 43.25 mL/min; (6) splitless inlet: temperature 270 °C, pressure 20.698 MPa, septum purge 3 mL/min, split ratio 160:1 mL/min. The injection volume was 1 µml, and the run time was 83.6 min. The 2-dimensional chromatograms obtained were analysed with the comprehensive GC × GC software (Zoex Corp., Houston, TX). Identification of FAME was obtained by comparing the cone position within the recording with the cone position of FAME contained in the GC reference standards [#674, (Nu-Chek Prep Inc.) and 47080-U bacterial acid methyl esters (Sigma-Aldrich, St. Louis, MO)]. The FAME identification procedure was completed by considering the elution order and position of each cone within the 2-dimensional chromatogram based on comprehensive GC × GC software (GC Imagine Software, Zoex Corp). Quantification of every FAME was obtained considering the cone volume of every peak with respect to the volume of total FAME and was expressed in terms of grams of FAME per grams of total FAME × 100, based on the concentration in weight of the methyl 12-tridecenoate contained in the solution used as internal standard. FAMEs were summed within the following categories: saturated fatty acids (SFAs), unsaturated fatty acids (UFAs), monounsaturated fatty acids (MUFAs), polyunsaturated fatty acids (PUFAs), and branched. The fatty acid ratios were additionally thought-about and calculated (Malainey, 1997; Eerkens, 2005).

2.3. Statistical analyses

The data are the mean of 3 replications and the standard deviation was always < 5%. The Kruskal Wallis test was used to match differences between group means. Correlations analysis was determined using Spearman's coefficient. To identify the structure of the relationship between the chemical parameters, a joint Principal Components Analysis (PCA) was performed. Prior to PCA, selected FAMEs were obtained by discriminant analysis (data not shown). Standardized variables were submitted to PCA; rotated orthogonal components (varimax rotation method) were extracted and the relative scores were determined. Solely PCs with eigenvalue >1 were thought. Automatic Linear Modelling (ALM) was used to assess the parameters that best influenced organic and inorganic P. ALM was performed at the 95% confidence level. Statistical analyses were performed using SPSS for Windows, version 27.0 (IBM SPSS, Chicago, IL).

3. Results

3.1. Chemical characteristics

The main chemical characteristics along the section of the ancient road and the reference profile are reported in Table 1. The ancient road showed phosphorus values in the range of 173-488 mg kg^{-1} for inorganic P (IP) (sample East Pit 7 and EP4) and 19-203 mg kg^{-1} for organic P (OP) (sample West Pit 11 and EP4, and Central 10), while the reference profile showed values in the range of 198-552 mg kg^{-1} for IP (sample 4CK and Ap) and between 14 and 49 mg kg^{-1} for OP (sample 2B/C and 2a/c). These values appear interesting from an archaeological point of view as they are sufficiently high to point to past traces of human activity.

In particular, at the superficial land level (25-30 cm depth) (Table 1) the samples taken from the droveway had lower values in IP and higher values in OP than that found in the reference profile. Indeed, the P value was between 209-374 mg kg^{-1} for IP in samples C10 and EP3, between 68-203 mg kg^{-1} for OP in samples WP13 and C10. Instead, the sample Ap from the reference profile had high values in IP (552 mg kg^{-1}) ($p \leq 0.05$), and a rather low quantity of OP (34 mg kg^{-1}) ($p \leq 0.05$). However, the samples from the superficial land level are not to be considered useful for this study, as they may have been disturbed by the fertilization carried out in the modern age.

Below the arable land level, the droveway showed higher P values than in the reference profile. Indeed, at the depth of 40-40/50 cm (Table 1), sample 12 from the west pit (WP12) had significantly higher values in IP (290 mg kg^{-1}) than the control 2a/c (204 mg kg^{-1}) ($p \leq 0.05$). Sample 9 from the central part of the road (C9) had a very high OP value (i.e., 3.9-fold the reference 2a/c) ($p \leq 0.05$) and sample 7 from the east pit (EP7) also had a high level of OP (80 mg kg^{-1}) ($p \leq 0.05$). At the depth of 50/70 cm (Table 1), significantly high values of IP and OP were found. Sample EP6 had a high IP ($p \leq 0.05$), while the external EX14 had a high OP ($p \leq 0.05$). Moving down indepth (Table 1), high P values were again found. Sample 2 from the east pit (EP2) and the external EX16 had values in OP pair of 5.9 and 5.2-fold the reference profile 3B and 4CK, respectively ($p \leq 0.05$). However, at the depth of 120 cm sample 4 from the east pit showed a value for IP 2.4-fold that of the reference profile (sample 4CK) ($p \leq 0.05$). With regards carbon and nitrogen, the ancient road and reference profile showed values that ranged from 0.26% to 2.40% for the OC and from 0.03% to 0.25% for TN (Table 1); overall OC and TN highly and positively correlated to each other ($r = 0.98$, $p \leq 0.01$). Comparisons made at a comparable depth revealed that below the arable land level, at 40-50 cm depth, OC content was 0.91% in the east pit (sample EP7) and 1.30% in the central road (sample C9) (i.e., 2.8 and 9 times the reference 2a/c) ($p \leq 0.05$) (Table 1). At a depth of 50-70 cm, significantly high levels of OC were found in WP11 and EP6. At 80-100 cm deep, the east pit EP2 had an OC content 2.2 higher than the reference profile ($p \leq 0.05$), whereas at 120 cm of depth in the external sample EX16 the OC increased by up to 4 times compared to the control (sample 4CK) ($p \leq 0.05$). Similarly, TN (Table 1) showed a high content in the central part of the road (sample C9) (i.e., 3.5 higher than the 2a/c control) ($p \leq 0.05$), and in the east pit (sample EP2) (3.6 times the control 3B1) ($p \leq 0.05$). In the external part, at the depth of 120 cm, in the sample EX16, TN r rose to the maximum value (6.5-fold the control 4CK) ($p \leq 0.05$).

The $\delta^{13}C$ fell into a narrow range, with values between -25.94‰ and -19.95‰ (Table 1). Comparison at the same depth displayed significantly higher (more positive) values in the samples from the ancient road than from the reference profile. An exception to this rule were the samples EP4 and EX16 in which the $\delta^{13}C$ was more negative (-24.26‰ and 24.05‰) than in the reference sample (-19.95‰). Regarding the $\delta^{15}N$, the values had a wide range (i.e., from -1.09‰ to +3.71‰) (Table 1). Down to a depth of 70 cm (Table 1), the samples taken from the road showed values close to or slightly lower than those of the reference profile, while from 80 cm to 120 cm the samples from the road had markedly higher values than the control. Particularly interesting is the result shown by the scatter plot of $\delta^{13}C$ against $\delta^{15}N$ values (Fig. 4). Here it is clear that the samples coming from the reference profile were distributed along a line evidencing a gradient with depth. Along this gradient, the samples showed significant variations in $\delta^{15}N$ with lower values at the increase in depth, and small variations in $\delta^{13}C$ values. An exception was the sample 4CK likely due to the high value in $\delta^{13}C$. The samples coming from the droveway, on the other hand, formed a fair cluster characterised by small differences in $\delta^{13}C$ and $\delta^{15}N$. Of this cluster, the samples WP11 and EX14 (70 cm depth) and sample EP1 (80-100 cm depth) had relatively low values in $\delta^{15}N$, while the sample EX15 (100 cm depth) and the samples EP6 and EX14 (70 cm depth) had relatively high values in $\delta^{13}C$ values.

Figure 5

3.2. Fatty acids

FAME profiles consisted of 154 fatty acids identified in the ancient road section and the reference profile (data shown in Table 5 and 6). These fatty acids ranged in carbon chain length from C6 to C25 and consisted of saturated (SFAs), monounsaturated (MUFAs), polyunsaturated (PUFAs) and branched fatty acids. Overall, SFAs ranging 40.2%-73.7% were more abundant than unsaturated (UFAs), which ranged 26.2%-59.7%, and the sample from the droveway had in general UFA to SFA ratios lower than the reference samples. Of these, 14 fatty acids were consistently present in the samples and were used for the final data set (Table 2). Palmitic (C16:0), stearic (C18:0), arachidic (C20:0), and lignoceric (C24:0) were the dominant fatty acids. Likewise, behenic (C22:0), pentadecanoic (C15:0), myristic (C14:0) and lauric (C12:0) acids were also found even though in lower abundance. These fatty acids are typical markers of degraded animal fats and have a strong biological signature. In detail (Table 2), at the superficial land level (25/30 cm), the sample from the reference profile (sample Ap) was distinguished by the presence of lauric (C12:0), palmitic (C16:0) and C18:1n9c acids and a somewhat lower quantity of myristic (C14:0), lignoceric (C24:0) and stearic (C18:0) acids. At the same depth, the sample taken from the central part of the ancient road (sample C10) was distinguished by a significantly ($p \leq 0.05$) high presence of palmitic (C16:0), stearic (C18:0), C22:1cis9 and pentadecanoic (C15:0) acids, whereas the samples taken in the east pit and west pit (samples EP3 and WP13) were distinguished by a high presence of lignoceric (C24:0), behenic (C22:0) and arachidic (C20:0) acids ($p \leq 0.05$). Below the arable layer, at 40-40/50 cm depth, the sample taken from the reference profile (sample 2a/c) had palmitic (C16:0), C18:2c9c12n6 and C18:1n9c acids, whereas the sample from the central part of the road (sample C9) exhibited a high amount of lignoceric (C24:0), arachidic (C20:0) and C22:1cis9 acids ($p \leq 0.05$). The west pit (WP12) had a high amount of stearic (C18:0) and myristic (C14:0) acids ($p \leq 0.05$), while the east pit (EP7) had high lignoceric (C24:0) and C22:1cis9 acids ($p \leq 0.05$). At the depth of 50/70 cm, the reference profile (sample 2BC) was distinguished by palmitic (C16:0), C18:1n9c and C18:2c9c12n6 acids, whereas the central part of the road (C8) had high C22:1cisn9, pentadecanoic (C15:0) and stearic (C18:0) acids ($p \leq 0.05$). The pits by the side of the road (samples WP11 and EP6) had lignoceric (C24:0), behenic (C22:0), and arachidic (C20:0) acids ($p \leq 0.05$). C22:1cisn9, palmitic (C16:0) and stearic (C18:0) acids were abundant in the external sample (EX14). Moving down in depth (80-110 cm), the sample from reference profile 3B1 showed the presence of palmitic (C16:0), C22:1cisn9, stearic (C18:0), C18:1n9c and C18:2c9c12n6 acids, while in the east pit (EP2) and the external EX15 sample there were high amounts of lignoceric (C24:0), C18:1n9c and behenic (C22:0) acids ($p \leq 0.05$). At the depth of 120 cm, the control 4CK had palmitic (C16:0), C18:1n9c and C22:1cisn9 acids, whereas the sample from the east pit (EP4) showed a high amount of lignoceric (C24:0) acid ($p \leq 0.05$).

Another useful tool for classifying degraded archaeological lipids involves analysing key fatty acid ratios. Concerning the (C15:0+C17:0)/(C12:0+C14:0+C16:0+C18:0) ratio, the ancient road always had higher values than the reference profile (Table 3).

3.3 PCA and ALM analyses

PCA revealed that three factors accounted for 82.2% of total variance. Principal component 1 (PC1) explained 37% of the variance and was (Table 4) positively correlated with tricosanoic (C23:0), lignoceric (C24:0), behenic (C22:0), arachidic (C20:0), margaric (C17:0) acid, and negatively with palmitic (C16:0), stearic (C18:0) and linoleic (C18:2) acid. PC2 explained 25.8% and was (Table 4) positively correlated with lauric (C12:0), $\delta^{13}C$, myristic (C14:0), erucic (C22:1) and pentadecanoic (C15:0) acid, and negatively with oleic (C18:1) acid. The remaining 19.2% of variance was explained by PC3 (Table 4), which was mostly correlated with TN, OP, OC, IC, IP and $\delta^{15}N$. Plotting data according to PC1 and PC2 (data not shown), behenic (C22:0), arachidic (C20:0), tricosanoic (C23:0), margaric (C17:0) and lignoceric (C24:0) acid characterized the central part of the ancient road and the east pit (samples C40/50, EP80/100 and EP25/30), while stearic (C18:0) and palmitic (C16:0) acid were mostly related to the west pit (WP40/50) and part of the central road (C20/30 and C70). Particularly interesting is the scatter plot obtained by plotting data according to PC1 and PC3 (Fig. 5, panel A and B). Here it is revealed that chemical parameters such as TN, OP and OC correlated well together and were proximal to lignoceric (C24:0), behenic (C22:0) and tricosanoic (C23:0) acids and characterised the east pit in depth (sample EP80/100). In contrast, palmitic (C16:0) and stearic (C18:0) acids characterised the west pit and external zone (WP70 and EX100).

ALM provided the result of two linear models that explain the relationship between P content and the other parameters analysed. The model for the IP was explained by 10 out of 20 assayed variables with 95% of accuracy. The relative importance (i) and significance of the obtained predictive variables was in the order OP (i = 0.34, $p \leq 0.001$), oleic (i = 0.25, $p \leq 0.001$), OC (i = 0.18, $p \leq 0.005$), IC (i = 0.12, $p \leq 0.002$), stearic (i = 0.08, $p \leq 0.018$), pentadecanoic (i = 0.07, $p \leq 0.025$), erucic (i = 0.07, $p \leq 0.045$), TN (i = 0.06, $p \leq 0.026$), myristic (i = 0.05, $p \leq 0.016$) and $\delta^{15}N$ (i = 0.05, $p \leq 0.05$). All the entered variables positively influenced the target variable (IP). The OP model had a 82% accuracy and was entered by OC (i = 0.32, $p \leq 0.010$), TN (i = 0.28, $p \leq 0.05$), IP (i = 0.25, $p \leq 0.012$), stearic (i = 0.15, $p \leq 0.012$), palmitic (i = 0.10, $p \leq 0.016$), erucic (i = 0.08, $p \leq 0.012$), linoleic (i = 0.05, $p \leq 0.05$), $\delta^{15}N$ (i = 0.03, $p \leq 0.05$), lignoceric (i = 0.03, $p \leq 0.010$) and tricosanoic (i = 0.03, $p \leq 0.05$) acid.

Figure 6

4. Discussion

The ancient road at the Valli Grandi Veronesi was found to be high in the major chemical elements of phosphorus, organic carbon and nitrogen; these are interpreted to reflect past anthropogenic activities (Leonardi et al., 1999; Schlezinger and Howes, 2000; Migliavacca et al., 2012, 2013). The elements distribution along the vertical (*i.e.*, from superficial to 120 cm depth) and longitudinal (*i.e.*, central part, east and west trenches, external part) sections was not homogeneous and revealed peak concentrations as a result of translocation and leaching phenomena. If we consider the studies conducted on the traces of P on areas that had been subjected to road traffic of people and things (Holliday and Gartner, 2007), these reveal a low level of P in the soil.

However, a high content of organic and inorganic phosphorus in the central part of the road section can be attributed to the hypothesis that the Big Road at the Costantini farm was used for the passage of herds of animals. In contrast, an uneven distribution of phosphorus content in the two side drainage ditches could be explained by a slight but noticeable soil dimpling located in the east ditch, in which higher amounts of P were detected. The phosphorus content recovered in our samples would support the anthropogenic footprint hypothesis.

With regards the analysis of the stable isotopes of carbon and nitrogen, the overall pattern that emerges (Fig. 4) suggests a terrestrial origin C_3 (Papathanasiou, 2015). The differences between $\delta^{13}C$ values showed that the reference profile was closer to the typical values of green plants, while those of the ancient road, being higher, are closer to the typical values of terrestrial animals. With respect to $\delta^{15}N$ values, we reach the expected range for plants comprising the herbivores'diet (Papathanasiou, 2015).

We found a significant number of fatty acids in the ancient road. The principal ones are palmitic (C16:0), stearic (C18:0), myristic (C14:0), pentadecanoic (C15:0), arachidic (C20:0), behenic (C22:0) and lignoceric (C24:0). Among the most abundant metabolites are palmitic and stearic acids, which are typical indicators of degraded fats that come from animal origin (Kedrowski et al., 2009; Regert, 2011). Several other odd chain fatty acids, including tridecanoic (C13:0), pentadecanoic (C15:0), margaric (C17:0) and tricosanoic (C23:0), were also present in smaller amounts. These compositions appeared to be ruminant-derived (Evershed et al., 2002). It can be seen from the present unsaturated fatty acids composition that they are cis-10-pentadecanoic (C15:1), palmitoleic (C16:1), cis-10 heptadecanoic (C17:1), linolelaidic (C18:2), linoleic (C18:2) and erucic (C22:1) acids; these are overall less abundant than saturated fatty acids. Also present is a small amount of the unsaturated species C18:1 (oleic); this is the most significant unsaturated FAME in animal fats. According to other authors (Spangenberg et al., 2010, Kanthilatha et al., 2014), the low proportion of unsaturated fatty acids may be due to the degradation of unsaturated components as a result of oxidative reactions. Although PUFAs are not plentiful, they are probably most susceptible to oxidation or degradation under burial conditions. Similar patterns were also found in archaeological sediments from prehistoric sites, which were often used as living floors or as activity areas by prehistoric people (Kanthilatha et al., 2014). High levels of odd-chained fatty acids (*e.g.*, C15:0 and C17:0) indicate that animal fat is present, and this is true when the ratio of (C15:0 + C17:0) to (C12:0 + C14:0 + C16:0 + C18:0) is greater than 0.04. All samples from the droveway had ratios greater than 0.04, indicating the presence of ruminant fat. Similar values were found in soil residues at the hearths site (Kedrowski et al., 2009).

ALM analysis indicates that the inorganic P probably comes from the mineralization processes of the organic P as a result of anthropogenic practices. This is confirmed by the fact that the organic P is closely related to the soil organic matter content. PCA, on the other hand, clearly shows that organic and inorganic P, organic carbon, and key fatty acids like palmitic, stearic, behenic and lignoceric characterize different samples. This suggests that a different accumulation pattern of the organic residues in the soil may be due to the morphology of the road and leaching process. Overall, the qualitative and quantitative differences between the samples from the ancient road and its control demonstrate that the fatty acids residues in the samples from the road do not simply represent the background present in the soil.

5. Conclusion

In conclusion, this study presents the main results obtained through a multi-parameter chemical approach to identify the "Big Road" as a herd transit site. Using a heat map to visualize complex chemical data (Fig. 6) in a single graph assists in identifying chemical parameters likely related to anthropogenic activities at archaeological sites. Among the major elements, P can be used as a means of detecting past human activity. The high organic P of the central part of the road section supports the interpretation of the "Big Road" as a road used for the passage of herds of animals. Instead, the uneven distribution of the P content in the two side drainage trenches could

be explained by a light but significant road bed in favour of the east trench where larger quantities of P are found. The $\delta^{13}C$ values found in the "Big Road" samples confirm the trace of terrestrial animals, certainly herbivores, as also indicated by the $\delta^{15}N$ values detected. In addition, the fatty acids composition is confirmed as a valid biomarker to identify the origin of animals, in particular ruminants that transited and rested in this site.
Finally, it is worth discussing the function that a herding road might have for the *terramara* of Castello del Tartaro. Its presence suggests that the irrigated land immediately surrounding the site was entirely devoted to agriculture and that people used the droveway to carefully manage the movement of animals and thus protect the agricultural land. In the future, it would be interesting to apply the chemical-physical analyses adopted in this study to verify the reliability of the use of the quadrangular enclosure on the eastern side of the embankment by animals.

Figure 7

Funding

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Figure captions

Fig. 1.
System of canals developing South-East of Castello del Tartaro; two small circles highlight the sites where the presence of the droveway (right: profilo strada) was detected and where a reference soil profile (not affected by finds: profilo di taratura) (left) was opened. Below, left: reference profile.

Fig. 2. The concentric drainage system, connected by transverse drains, developing around the embankment site of Castello del Tartaro (the embankment is outlined in yellow). The two groups of double parallel trenches interpreted as a droveway are outlined in red; the SAM is outlined in green (modified from Cima, 2012).

Fig. 3. The profile of the 34.1 m wide section, corresponding to the ancient road (droveway) in Fondo Costantini, Verona. In the figure the three profiles along which the samples were collected are highlighted: top is the sampling area corresponding to the pit west of the road, middle is the sampling area in the middle of the road, bottom is the sampling area corresponding to the pit east of the road (drawn by Claudio Balista).

Fig. 4. $\delta^{13}C$ versus $\delta^{15}N$ of the soil samples collected from the droveway (red) and reference profile (black) labelled per depth. Abbreviations are: EP, East Pit; C, Central; WP, West Pit; EX, External, and REF, reference profile.

Fig. 5. Positions of the soil samples collected at different depths from the droveway (panel A) and variables projection (panel B) in the reduced space of the principal component 1 and 3. Abbreviations for the soil samples are : East Pit, EP; Central, C; West Pit and External, EX. Abbreviations for variables: arachidic, ARA; behenic, BEH; inorganic carbon, IC; inorganic phosphorus, IP; lignoceric, LIGN; organic carbon, OC; organic phosphorus, OP; palmitic, PAL; stearic, STE; total nitrogen, TN; tridecanoic, TRC.

Fig. 6. Heat map of some chemical parameters in the sample collected at different depths from the droveway: east pit (EP), central (C), west pit (WP) and external (EX). Intensity colours correspond to a higher presence of the related chemical parameter. Abbreviations for variables: arachidic, ARA, behenic, BEH, inorganic phosphorus, IP, lignoceric, LIGN, organic phosphorus, OP, stearic, STE.

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Table 1

Chemical characteristics in the soil samples collected from the section of the droveway (East Pit, EP; Central, C; West Pit; External, EX) and control profile (reference, REF) listed and highlighted per depth.

Sample		Depth	P_{org}	P_{inorg}	Organic C	Total N	C:N	$\delta^{13}C$	$\delta^{15}N$
		cm	mg kg^{-1}	mg kg^{-1}	%	%		‰	‰
REF	Ap	25-30	34d*	552a	2.18a	0.22a	11.5	-25.01a	3.71a
EP	3	25-30	132b	374b	1.89a	0.21a	10.5	-24.54b	3.06b
C	10	25-30	203a	209d	1.81a	0.20a	10.7	-24.45b	3.54a
WP	13	25-30	68c	311c	1.85a	0.23a	9.4	-24.66b	3.18b
REF	2a/c	40	49c	204b	0.46b	0.04b	13.6	-25.88a	2.93a
EP	7	40-50	80b	173c	0.91a	0.10a	10.7	-25.35a	2.21b
C	9	40-50	192a	211b	1.30a	0.14a	10.6	-24.52b	3.16a
WP	12	40-50	56c	290a	0.54b	0.04b	14.5	-24.58b	2.25b
REF	2B/C	50-70	14d	342c	0.37b	0.04a	12.0	-25.94a	2.21ab
EP	6	70	28b	390a	0.48a	0.06a	9.8	-23.78b	2.76a
C	8	70	26b	337c	0.31b	0.03a	12.4	-24.43b	n.d.
WP	11	70	19c	321d	0.54a	0.03a	22.2	-25.21a	1.61b
EX	14	70	43a	374b	0.26b	0.03a	9.4	-23.74b	1.71b
REF	3B	80-100	17d	348a	0.37b	0.03b	16.6	-25.29a	-1.09b
EP	1	80-100	63b	352a	0.42b	0.03b	17.8	-24.91a	1.26b
EP	5	80-100	37c	206b	0.35b	0.04b	9.8	-24.28a	3.59a
EP	2	80-100	101a	179c	1.03a	0.11a	10.7	-25.07a	2.86a
EX	15	100	50b	200b	0.87a	0.09a	11.2	-22.93b	3.14a
REF	4CK	120	39b	198b	0.59b	0.04b	18.2	-19.95b	1.88b
EP	4	120	19b	488a	0.38b	0.08b	5.7	-24.26a	3.48a
EX	16	120	200a	226b	2.40a	0.25a	11.0	-24.05a	3.52a

*Different letters in the same depth section of a column indicate differences at $p \leq 0.05$ by the Kruskal Wallis test.

Table 2

Percentage on total mass of identified fatty acid peaks alongside the section of the droveway (EP = East Pit, C = Central, WP = West Pit, EX = External) and controls (REF = reference) listed per depth.

Sample		Depth	C12:0	C13:0	C14:0	C15:0	C16:0	C17:0	C18:0	C20:0	C22:0	C23:0	C24:0	C18:1n9c	C22:1cis n9	C18:2c9c12-n6
REF	Ap	25-30	33.35a*	0.11a	7.44a	0.32c	14.25b	0.27b	3.64b	1.65b	1.74b	1.28b	4.68b	9.22a	1.77c	5.93a
EP	3	25-30	1.29b	0.29a	2.56b	1.27b	13.35b	0.81a	6.03a	5.28a	5.22a	3.69a	13.77a	5.45b	4.36b	1.68b
C	10	25-30	1.43b	0.30a	3.17b	1.47a	18.62a	0.65a	7.61a	2.41b	2.73b	0.92b	3.94b	6.65b	10.64a	2.22b
WP	13	25-30	0.96b	0.25a	1.92b	1.07b	14.66b	0.74a	5.72a	4.58a	4.76a	3.50a	13.40a	8.75a	4.46b	1.64b
REF	2a/c	40	2.47a	0.13a	1.85b	0.63b	18.26a	0.32a	5.72b	1.77b	1.49b	0.70b	2.78b	16.42a	4.61b	21.02a
EP	7	40-50	1.14b	0.28a	2.50ab	1.22a	14.50b	0.65a	6.76b	3.81a	4.42a	2.67a	9.53a	7.02c	7.11a	1.63b
C	9	40-50	1.06b	0.28a	2.31ab	1.29a	15.21b	0.79a	6.33b	4.08a	4.22a	2.68a	9.85a	8.94c	6.15a	1.50b
WP	12	40-50	1.40b	0.19a	3.70a	0.91b	20.05a	0.58a	8.20a	3.78a	2.06b	0.73b	3.01b	11.34b	6.07a	2.48b
REF	2BC	50-70	3.85a	0.10b	1.93b	0.48b	16.93a	0.26b	5.25b	1.96b	0.92b	0.33b	1.30d	16.84a	4.72c	27.09a
EP	6	70	1.28b	0.37a	2.98a	1.54a	16.42a	0.83b	6.98b	4.29a	3.99a	1.81b	7.54b	5.88c	8.53b	1.82b
C	8	70	1.32b	0.29a	3.03a	1.33a	18.86a	0.54b	9.83a	3.94a	2.60b	0.92b	3.28c	6.21c	10.46a	2.62b
WP	11	70	1.08b	0.28a	2.24b	1.19a	13.52b	1.99a	6.49b	5.12a	5.11a	3.80a	13.90a	9.91b	4.01c	2.32b
EX	14	70	1.50b	0.29a	3.58a	1.35a	19.60a	0.70b	8.60a	3.86a	2.25b	0.48b	1.88d	8.43b	12.33a	3.22b
REF	3B1	80-100	1.02b*	0.18b	2.32b	1.06b	20.59a	0.42b	6.89a	3.32a	1.40c	0.41b	2.00b	5.34c	12.66a	2.76b
EP	1	80-100	1.24a	0.28a	2.99a	1.48a	15.87b	0.66a	7.36a	4.29a	2.25b	0.69b	3.05b	5.63c	10.65a	2.86b
	5	80-100	1.24a	0.29a	3.24a	1.52a	18.33a	0.69a	7.56a	2.75b	2.90b	1.15b	4.71b	5.25c	9.35a	2.14b
	2	80-100	1.14a	0.30a	2.67ab	1.34a	14.81b	0.83a	6.51b	4.64a	4.75a	2.92a	10.71a	8.00b	5.15b	1.84b
EX	15	100	0.83c	0.25a	1.47b	0.96b	15.62b	0.73a	6.11b	4.45a	3.79a	2.73a	10.29a	19.60a	2.14b	2.66b
REF	4CK	120	1.55a	0.23a	2.76a	1.25a	18.37a	0.54a	7.55a	4.55a	3.03a	0.35b	3.30b	11.35a	11.50a	3.97a
EP	4	120	1.06a	0.28a	2.71a	1.53a	18.48a	0.75a	8.07a	3.24a	3.96a	2.15a	8.09a	5.25a	8.69a	2.16a
EX	16	120	1.28a	0.27a	2.27a	1.28a	16.28a	0.84a	6.73a	4.18a	3.16a	1.59a	6.02a	11.49a	5.66a	2.75a

C12:0, Lauric; C13:0, Tridecanoic; C14:0, Myristic; C15:0, Pentadecanoic; C16:0 Palmitic; C17:0, Margaric; C18:0, Stearic; C20:0 Arachidic; C22:0, Behenic; C23:0, Tricosanoic; C24:0, Lignoceric; C18:1n9c, Oleic; C22:1cisn9, Erucic; C18:2c9c12n6, Linoleic.

*Different letters in the same depth section of a column indicate differences at $p \leq 0.05$ by the Kruskal Wallis test.

Table 3

FAME ratio in the sample from the droveway (East Pit, EP; External, EX) and controls (REF, reference) listed per depth.

Sample		Depth	(C15:0+C17:0)/(C12:0+C14:0+C16:0+C18:0)
REF	Ap	25-30	0.01
EP	3	25-30	0.09
C	10	25-30	0.07
WP	13	25-30	0.08
REF	2a/c	40	0.03
EP	7	40-50	0.08
C	9	40-50	0.08
WP	12	40-50	0.04
REF	2BC	50-70	0.03
EP	6	70	0.09
C	8	70	0.06
WP	11	70	0.14
EX	14	70	0.06
REF	3B	80-100	0.05
EP	1	80-100	0.08
EP	5	80-100	0.07
EP	2	80-100	0.09
EX	15	100	0.07
REF	4CK	120	0.06
EP	4	120	0.08
EX	16	120	0.08

Table 4

Loading values of chemical variables on the axes identified by principal components analysis.

Variable	PC1	PC2	PC3
Tricosanoic (TRC)	0.957	-0.127	0.118
Lignoceric (LIGN)	0.957	-0.274	0.218
Palmitic (PAL)	-0.924	0.048	-0.061
Behenic (BEH)	0.908	0.180	0.076
Stearic (STE)	-0.828	0.388	-0.334
Arachidic (ARA)	0.821	-0.131	-0.359
Margaric (MAR)	0.733	-0.127	-0.206
Linoleic (LIN)	-0.662	-0.330	-0.485
Oleic (OLE)	0.068	-0.978	0.051
Lauric (LAU)	-0.010	0.964	-0.059
δ13C	-0.006	0.956	-0.102
Myristic (MYR)	-0.166	0.956	-0.089
Erucic (ERU)	-0.422	0.894	-0.082
Pentadecanoic (PEN)	0.195	0.816	0.004
Tridecanoic (TRD)	-0.036	0.672	-0.119
Total nitrogen (TN)	0.296	-0.119	0.917
Organic phosphorus (OP)	0.287	0.178	0.853
Organic carbon (OC)	0.467	-0.090	0.768
Inorganic carbon (IC)	0.215	0.175	-0.765
Inorganic phosphorus (IP)	0.112	0.252	-0.756
δ15N	-0.419	-0.001	0.705

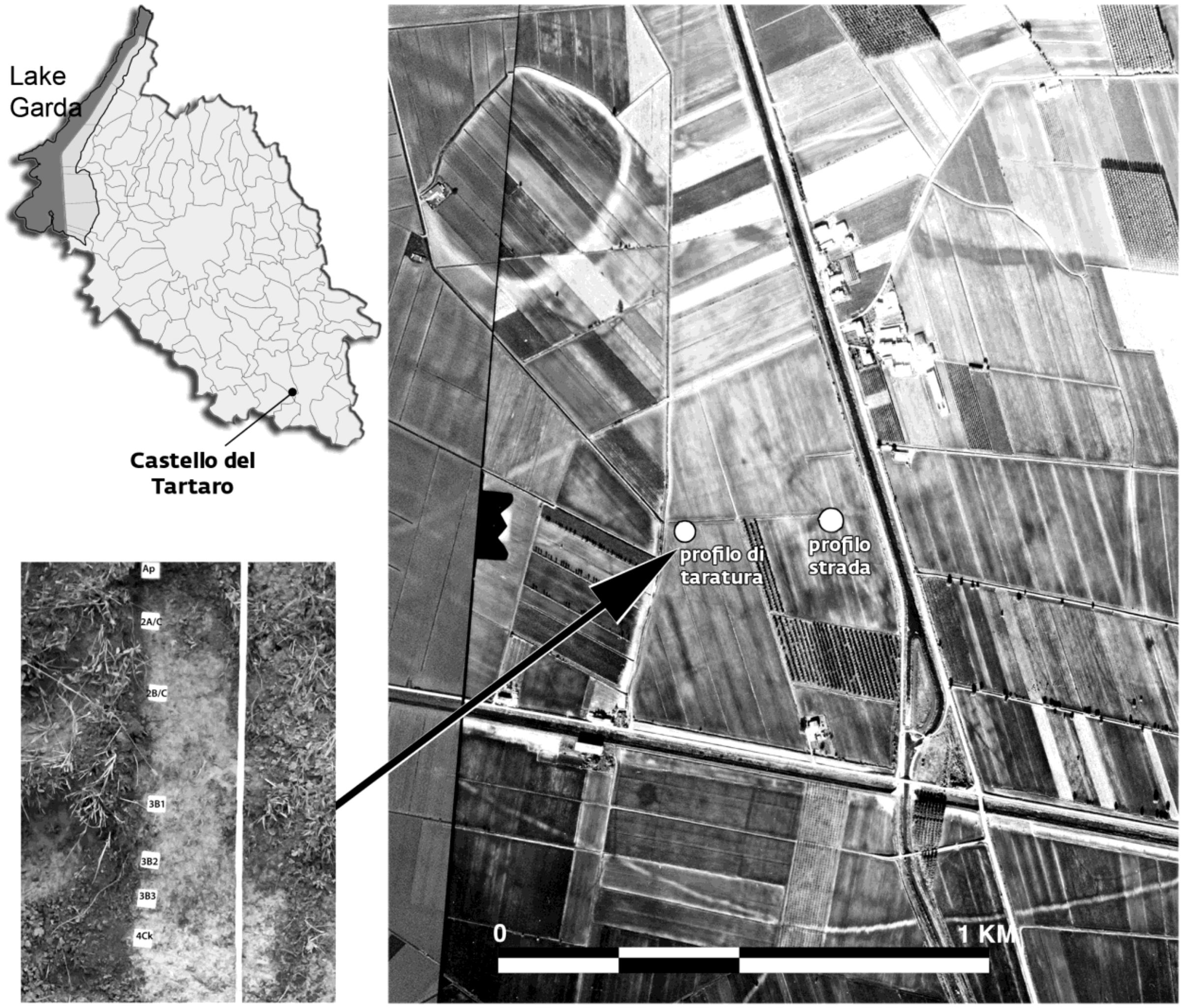

Fig. 1. System of canals developing South-East of Castello del Tartaro; two small circles highlight the sites where the presence of the droveway (right: profilo strada) was detected and where a reference soil profile (not affected by finds: profilo di taratura) (left) was opened. Below, left: reference profile.

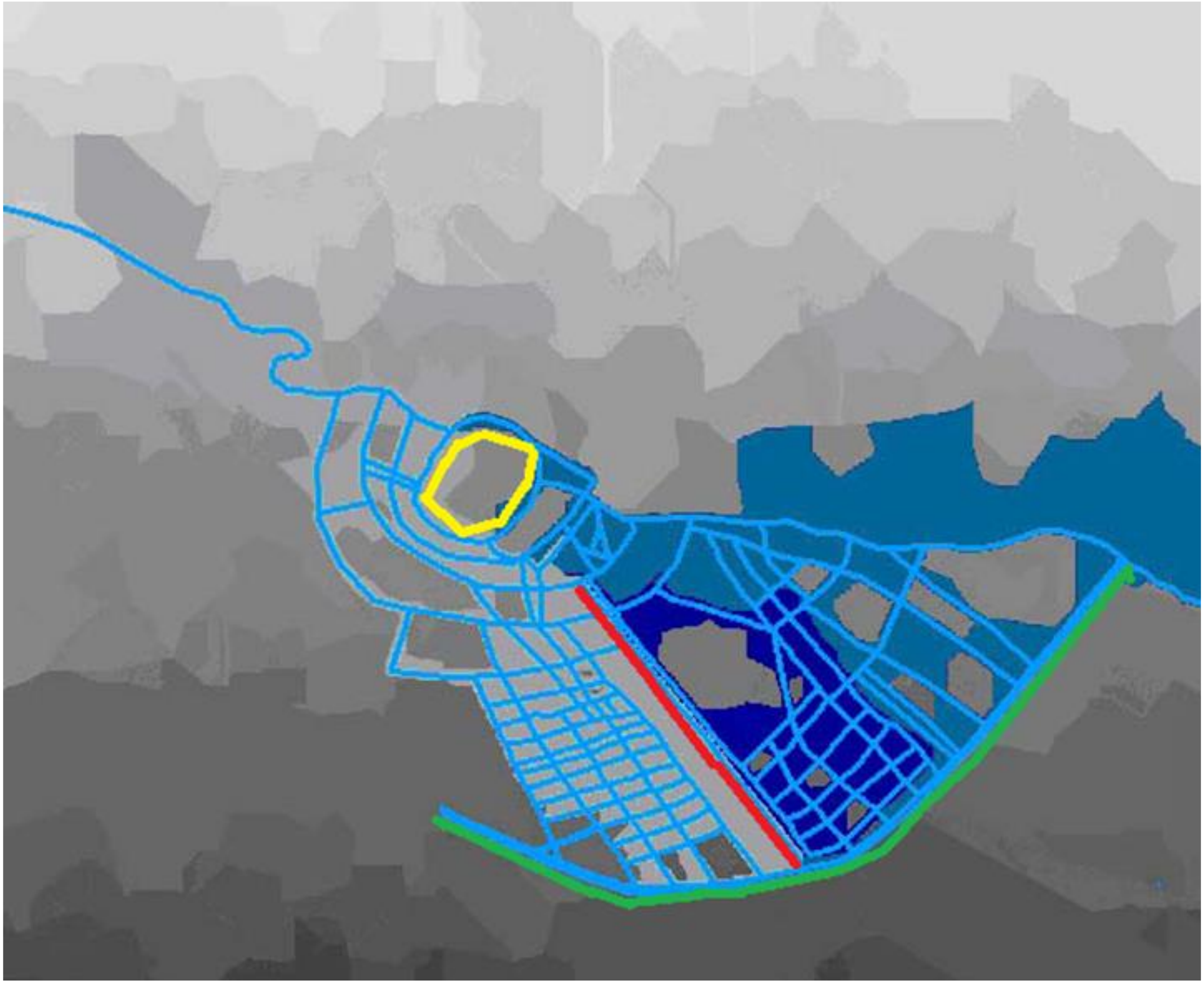

Fig. 2. The concentric drainage system, connected by transverse drains, developing around the embankment site of Castello del Tartaro (the embankment is outlined in yellow). The two groups of double parallel trenches interpreted as a droveway are outlined in red; the SAM is outlined in green (modified from Cima, 2012).

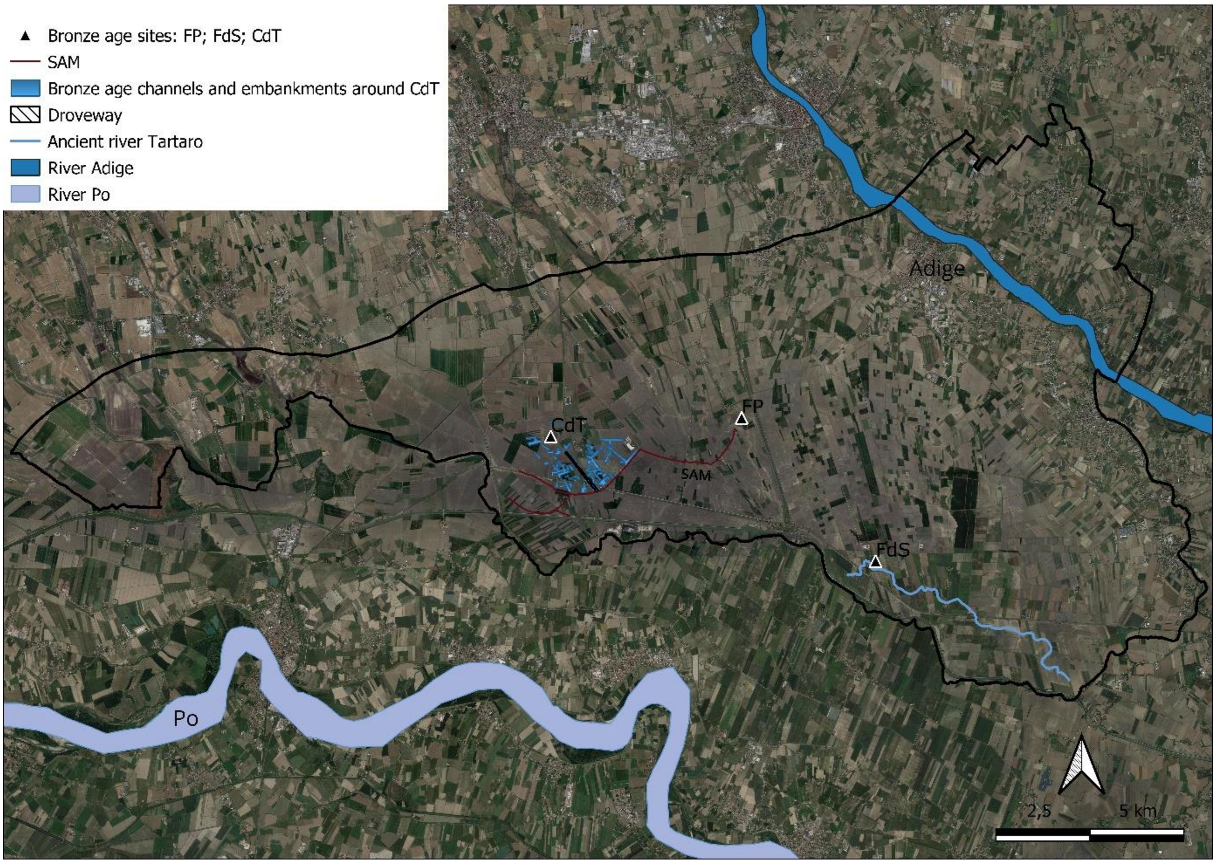

Fig.3. The area under study. The black line identifies the Valli Grandi Veronesi; the three terramare of Castello del Tartaro (CdT), Fondo Paviani (FP) and Fabbrica dei Soci (FS) are indicated therein (graphic elaboration of Vito Filadelfia).

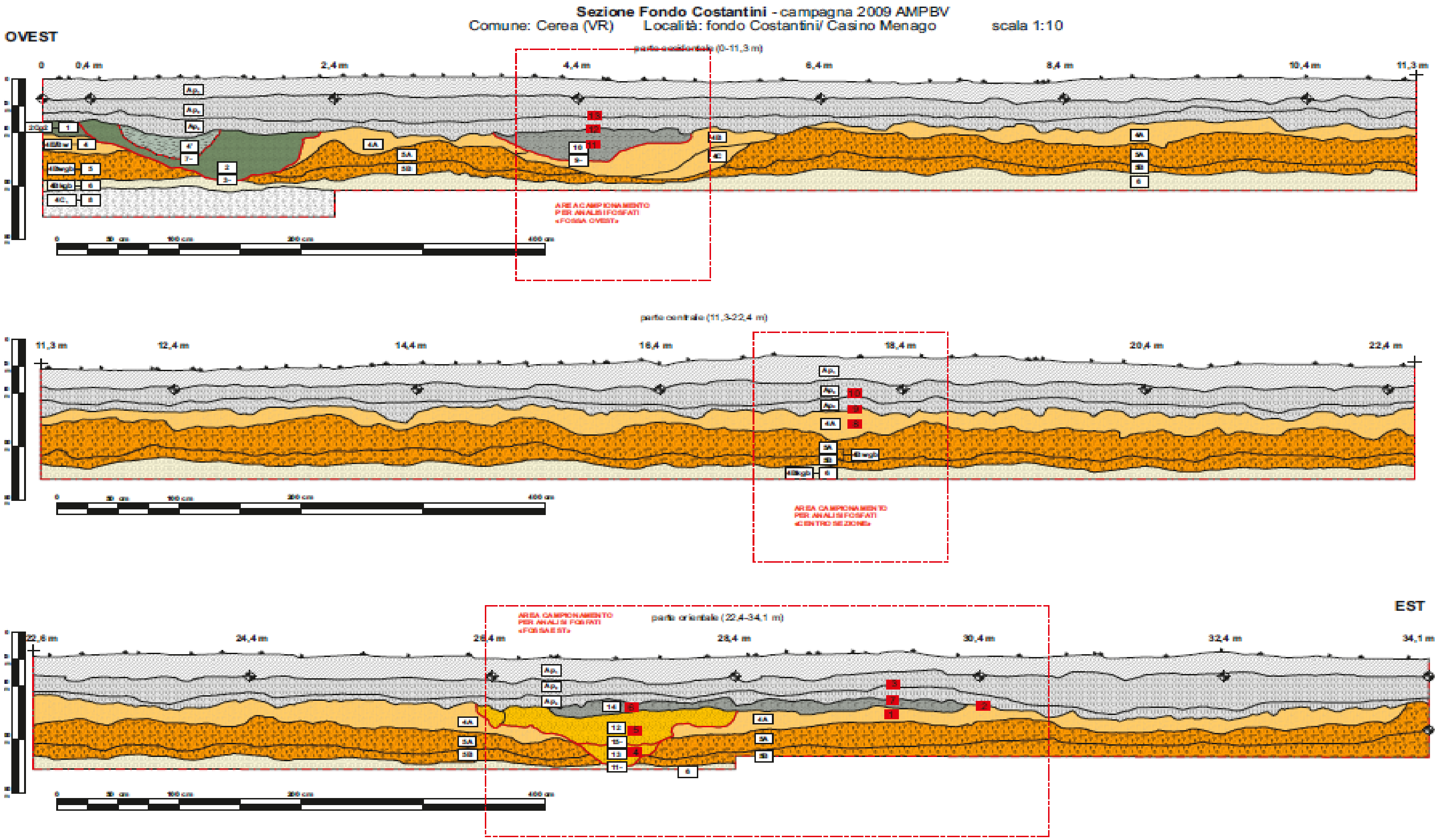

Fig. 4. The profile of the 34.1 m wide section, corresponding to the ancient road (droveway) in Fondo Costantini, Verona. In the figure the three profiles along which the samples were collected are highlighted: top is the sampling area corresponding to the pit west of the road, middle is the sampling area in the middle of the road, bottom is the sampling area corresponding to the pit east of the road (drawn by Claudio Balista).

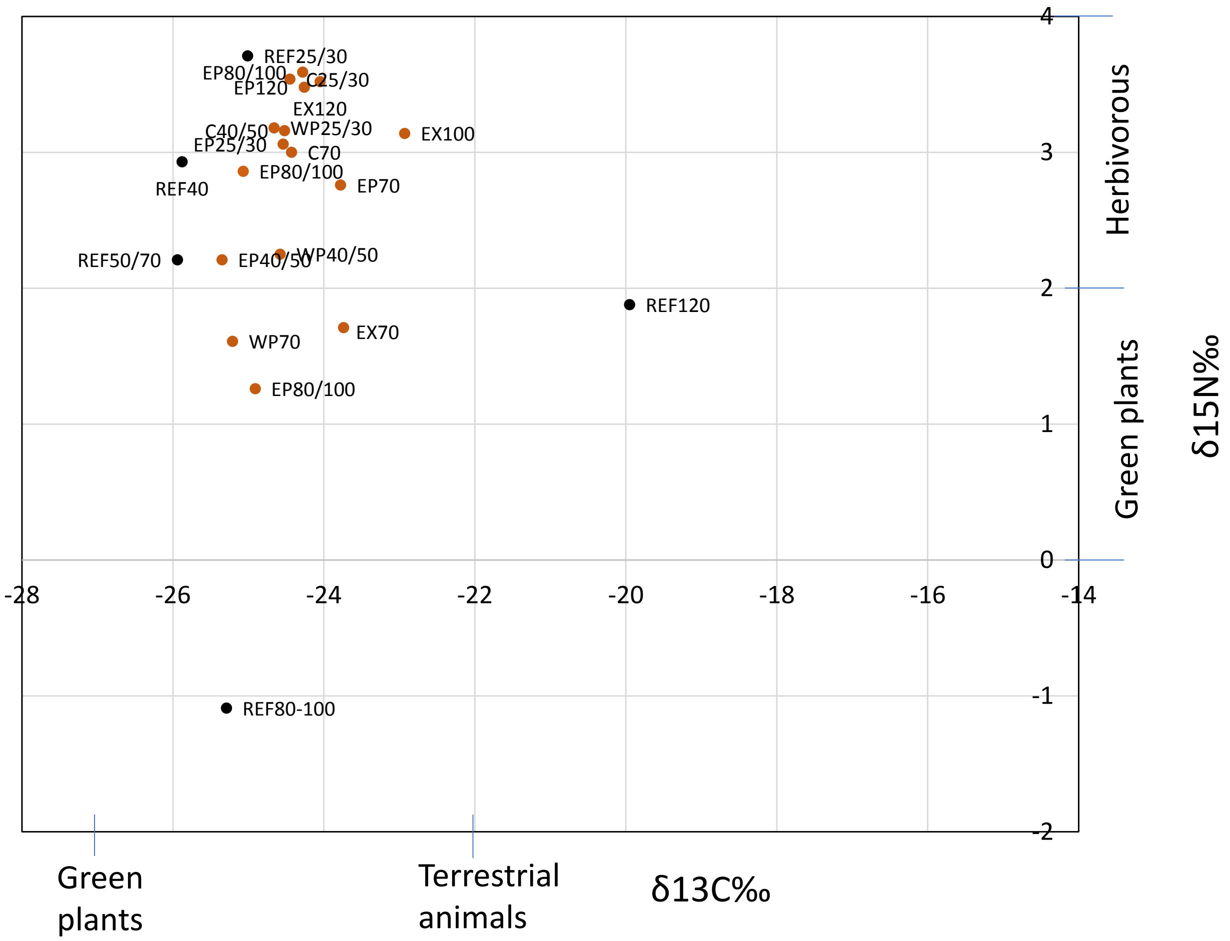

Fig. 5. $\delta^{13}C$ versus $\delta^{15}N$ of the soil samples collected from the droveway (red) and reference profile (black) labelled per depth. Abbreviations are : EP, East Pit; C, Central; WP, West Pit; EX, External, and REF, reference profile.

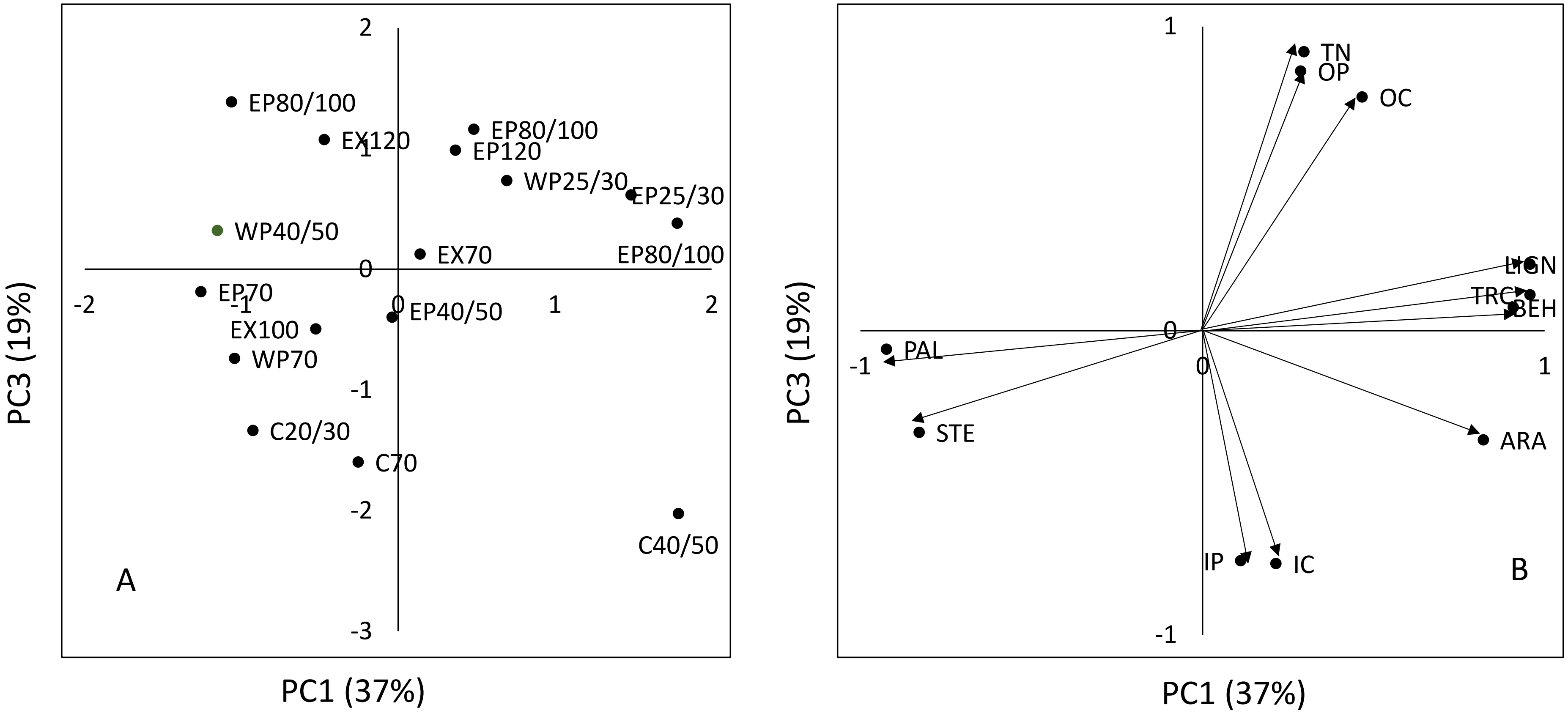

Fig. 6. Positions of the soil samples collected at different depths from the droveway (panel A) and variables projection (panel B) in the reduced space of the principal component 1 and 3. Abbreviations for the soil samples are : East Pit, EP; Central, C; West Pit and External, EX. Abbreviations for variables: arachidic, ARA; behenic, BEH; inorganic carbon, IC; inorganic phosphorus, IP; lignoceric, LIGN; organic carbon, OC; organic phosphorus, OP; palmitic, PAL; stearic, STE; total nitrogen, TN; tridecanoic, TRC.

	REF 40	EP 40/50	C 40/50	WP 40/50	REF 50/70	EP 70	C 70	WP 70	EX 70	REF 80	EP 80/100	EP 80/100	EP 80/100	EX 100	REF 120	EP 120	EX 120
IP	204	173	211	290	342	390	337	321	374	348	352	206	179	200	198	488	226
OP	49	80	192	56	14	28	26	19	43	17	63	37	101	50	39	19	200
MYR	2	3	2	4	2	3	3	2	4	2	3	3	3	1	3	3	2
STE	6	7	6	8	5	7	10	6	9	7	7	8	7	6	8	8	7
ARA	2	4	4	4	2	4	4	5	4	3	4	3	5	4	5	3	4
BEH	1	4	4	2	1	4	3	5	2	1	2	3	5	4	3	4	3
LIGN	3	10	10	3	1	8	3	14	2	2	3	5	11	10	3	8	6

Fig. 7. Heat map of some chemical parameters in the sample collected at different depths from the droveway's east drainage trench (EP), central (C), west drainage trench (WP), external (EX), and reference profile (REF). Intensity colours correspond to higher presence of the related chemical parameter. Abbreviations for variables: arachidic, ARA, behenic, BEH, inorganic phosphorus, IP, lignoceric, LIGN, organic phosphorus, OP, stearic, STE.

Table S1

Fames identified in the droveway and reference profile.

Description	Compound name	Group Name
hexanoic acid, caproic acid	C6:0	saturated acid 03
Heptanoic acid	C7:0	saturated acid 04
octanoic acid, caprilic acid	C8:0	saturated acid 05
7-methyloctanoic acid	C8:0 iso	saturated acid 050
nonanoic acid, pelargonic acid	C9:0	saturated acid 06
8-methylnonanoic acid	C10:0 iso?	saturated acid 060
decanoic acid, capric acid	C10:0	saturated acid 07
9-methyldecanoic acid	C11:0 iso	saturated acid 071
?-methylundecanoic acid	C11:0 branced?	saturated acid 0710
8-methyldecanoic acid	C11:0 anteiso	saturated acid 072
undecanoic acid	C11:0	saturated acid 08
10-methylundecanoic acid	C12:0 iso	saturated acid 081
dodecanoic acid, lauric acid	C12:0	saturated acid 09
?-methyldodecanoic acid	C13:0 branced?	saturated acid 090
11-methyldodecanoic acid	C13:0 iso	saturated acid 091
10-methyldodecanoic acid	C13:0 anteiso	saturated acid 092
tridecanoic acid	C13:0	saturated acid 10
12-methyltridecanoic acid	C14:0 iso	saturated acid 101
11-methyltridecanoic acid	C14:0 anteiso	saturated acid 102
tetradecanoic acid, myristic acid	C14:0	saturated acid 11
13-methyltetradecanoic acid	C15:0 iso	saturated acid 111
12-methyltetradecanoic acid	C15:0 anteiso	saturated acid 112
pentadecanoic acid	C15:0	saturated acid 12
14-methylpentadecanoic acid	C16:0 iso	saturated acid 121 branched
13-methylpentadecanoic acid	C16:0 anteiso	saturated acid 122 branched
hexadecanoic acid, palmitic acid	C16:0	saturated acid 13
C17:0 ramified?	C17:0 branced?	saturated acid 130 branched
C17:0 ramified?	C17:0 branced1?	saturated acid 1301 branched

C17:0 ramified?	C17:0 branced2?	saturated acid 1301 branched
15-methylhexadecanoic acid	C17:0 iso	saturated acid 131 branched
14-methylhexadecanoic acid	C17:0 anteiso	saturated acid 132 branched
heptadecanoic acid, margaric acid	C17:0	saturated acid 14
methyl-n?-C18:0 ramified?	C18:0 branced?	saturated acid 140 branched
16-methylheptadecanoic acid	C18:0 iso	saturated acid 141
15-methylheptadecanoic acid	C18:0 anteiso	saturated acid 142
octadecanoic acid, stearic acid	C18:0	saturated acid 15
methyl-n?-C19?:0 ramified?	C19:0 branced?	saturated acid 1501 branched
17-methyloctadecanoic acid	C19:0 iso	saturated acid 151
methyl-n?-C19:0 ramified?	C19:0 branced?	saturated acid 151 branched
nonadecanoic acid, nonadecylic acid	C19:0	saturated acid 16
eicosanoic acid, arachidic	C20:0	saturated acid 17
methyl-n?-C21?:0 ramified?	C21:0 branced?	saturated acid 171 branched
methyl-n?-C21?:0 ramified?	C21?0 branced?	saturated acid 172 branched
heneicosanoic acid	C21:0	saturated acid 18
docosanoic acid, behenic acid	C22:0	saturated acid 19
tricosanoic acid	C23:0	saturated acid 20
tetracosanoic acid, lignoceric acid	C24:0	saturated acid 31
pentacosanoic acid	C25:0	saturated acid 32
16-methyloctadecanoic acid	C19:0 anteiso	saturated acid 152
C15:1 anteiso?	C15:1 anteiso?	monounsaturated acid
C15:1iso?	C15:1 iso?	monounsaturated acid
(Z)-9-decenoic acid, caproleic acid	C10:1	monounsaturated acid 01
(Z)-undec-10-enoic acid	C11:1	monounsaturated acid 0121
(Z)-dodec-?-enoic acid	C12:1cn?	monounsaturated acid 022
(E)-dodec-?-enoic acid	C12:1tn?	monounsaturated acid 03
C13:1 iso?	C13:1 iso?	monounsaturated acid 031
(E)-tetradec-?-enoic acid	C14:1tn?	monounsaturated acid 039
(E)-tetradec-9-enoic acid	C14:1tn5	monounsaturated acid 04
(Z)-dodec-11-enoic acid	C12:1cn1	monounsaturated acid 04
(Z)-tetradec-7-enoic acid	C14:1n7	monounsaturated acid 06
(Z)-tetradec-?-enoic acid	C14:1n?cis	monounsaturated acid 061
(Z)-tetradec-9-enoic acid, myristoleic acid	C14:1cn5	monounsaturated acid 07

(Z)-tetradec-?-enoic acid	C14:1n?	monounsaturated acid 071
(Z)-tetradec-?-enoic acid	C14:1n?	monounsaturated acid 08
(E)-pentadec-?-enoic acid	C15:1t isomer 01	monounsaturated acid 10
(E)-pentadec-?-enoic acid	C15:1tn?	monounsaturated acid 101
(E)-pentadec-?-enoic acid	C15:1t isomer 02	monounsaturated acid 11
(10E)-pentadecenoic acid	C15:1n5t	monounsaturated acid 12
	C13:1?n?c	monounsaturated acid 121
(Z)-pentadec-?-enoic acid	C15:1 isomer01	monounsaturated acid 13
(Z)-pentadec-?-enoic acid	C15:1 isomer02	monounsaturated acid 14
(10Z)-pentadecenoic acid, pentadecenoic acid	C15:1n5	monounsaturated acid 15
C16:1 iso?	C16:1 iso?	monounsaturated acid 151 branched
C16:1 ramified?	C16:1 branced?	monounsaturated acid 152 branched
C16:1 ramified?	C16:1 branced?	monounsaturated acid 153 branched
(E)-hexadec-?-enoic acid	C16:1t isomer01	monounsaturated acid 17
(E)-hexadece-6-enoic acid	C16:1n10t	monounsaturated acid 18
(E)-hexadec-9-enoic acid	C16:1n7t	monounsaturated acid 19
(E)-hexadec-10?-enoic acid	C16:1n6t?	monounsaturated acid 20
(E)-hexadec-11?-enoic acid	C16:1n5t?	monounsaturated acid 201
(Z)-hexadec-7-enoic acid	C16:1n9c	monounsaturated acid 202
(Z)-hexadec-9-enoic acid, palmitoleic acid	C16:1n7c	monounsaturated acid 203
(Z)-hexadec-11?-enoic acid	C16:1n5c	monounsaturated acid 204
(Z)-hexadec-13-enoic acid	C16:1n3c?	monounsaturated acid 205
(E)-hexadec-?-enoic acid	C16:1cis isomer03	monounsaturated acid 206
(10E)-heptadec-10-enoic acid	C17:1n7t	monounsaturated acid 251
(Z)-heptadec-8-enoic acid	C17:1n9	monounsaturated acid 251
(E)-heptadec-6-enoic acid	C17:1n6t	monounsaturated acid 251
C17:1?iso?	C17:1?	monounsaturated acid 251 branched
(Z)-heptadec-6-enoic acid	C17:1n6?	monounsaturated acid 252
(Z)-heptadec-12-enoic acid	C17:1n5	monounsaturated acid 252
(Z)-heptadec-13-enoic acid	C17:1n4	monounsaturated acid 252
C17:1?iso?	C17:1? branced	monounsaturated acid 252 branched
C19:1tn?	C19:1tn?	monounsaturated acid 2521
(Z)-heptadec-9?-enoic acid	C17:1n8	monounsaturated acid 26
(Z)-heptadec-10-enoic acid	C17:1n7c	monounsaturated acid 27

	C18:1n14t?	monounsaturated acid 321
(E)-octadec-8-enoic acid	C18:1n10t?	monounsaturated acid 33
(E)-octadec-9-enoic acid, elaidic acid	C18:1n9t	monounsaturated acid 34
(E)-octadec-10-enoic acid	C18:1n8t	monounsaturated acid 341
(11E)-octadec-11-enoic acid, trans-vaccenic acid	C18:1n7t	monounsaturated acid 35
(9Z)-octadec-9-enoic acido, oleic acid	C18:1n9c	monounsaturated acid 39
(11Z)-octadec-11-enoic acid, vaccenic acid	C18:1n7c	monounsaturated acid 40
(Z)-octadec-12-enoic acid?	C18:1n6c	monounsaturated acid 41
(Z)-octadec-13-enoic acid?	C18:1n5c	monounsaturated acid 42
(E)-octadec-16-enoic acid?	C18:1n2t	monounsaturated acid 43
(Z)-octadec-14-enoic acid?	C18:1n4c	monounsaturated acid 44
(Z)-octadec-15-enoic acid?	C18:1n3c	monounsaturated acid 45
(Z)-octadec-16-enoic acid?	C18:1n2c	monounsaturated acid 46
(E)-nonadec-10-enoic acid	C19:1n9t	monounsaturated acid 48
(Z)-nonadec-7-enoic acid	C19:1n12	monounsaturated acid 49
(Z)-nonadec-10-enoic acid	C19:1n9c	monounsaturated acid 50
(E)-eicos-11-enoic acid	C20:1tn9	monounsaturated acid 53
(Z)-eicos-8-enoic acid	C20:1n12	monounsaturated acid 54
(Z)-eicos-11-enoic acid, gondoic acid	C20:1n9	monounsaturated acid 55
(Z)-eicos-13-enoic acid ?	C20:1n7	monounsaturated acid 56
(E)-docos-13-enoic acid, brassidic acid	C22:1tn9	monounsaturated acid 571
(Z)-docos-13-enoic, erucic acid	C22:1cis n9	monounsaturated acid 573
(Z)-docos-15-enoic acid	C22:1cis n7	monounsaturated acid 574
(Z)-tetracos-15-enoic acid nervonic acid	C24:1	monounsaturated acid 62
(?)methylhexadecenoic acid ramified?	?MeC16:1?	monounsaturated acid branched 134
7-methyl-6-methyl-hexadecenoic acid	7Me 6MeC16:1	monounsaturated acid branched 135
3,6-dodecadienoic acid?	C12:2	polyunsaturated acid 01
(?,?)-tetradeca-dienoic acid	C14:2n?	polyunsaturated acid 01
(?,?)-hexadeca-?,?-dienoic acid	C16:2n?	polyunsaturated acid 01
(?,?)-hexadeca-dienoic acid, non methylene interrupted	C16:2 NMI	polyunsaturated acid 011
(?Z,?Z)-tridecadienoic acid	C13:2?	polyunsaturated acid 02
(Z6,Z9)-hexadecadienoic acid	C16:2n7	polyunsaturated acid 02
(?Z,?Z)-hexadeca-dienoic acid	C16:2 isomer	polyunsaturated acid 021
(?,?)-tetradeca-dienoic acid	C14:2	polyunsaturated acid 03

(?,?)-tetradeca-dienoic acid	C14:2?	polyunsaturated acid 03
(?,?)-pentadeca-dienoic acid	C15:2?	polyunsaturated acid 04
C17:2 n? 02	C17:2n?	polyunsaturated acid 052
(E,E)-octadeca-?,?-dienoic acid	C18:2t?t? 01	polyunsaturated acid 08
(9E,12E)-octadeca-9,12-dienoic acid	C18:2t9t12	polyunsaturated acid 10
(Z,E)-octadeca-9,13-dienoic acid	C18:2c9t13	polyunsaturated acid 11
(E,Z)-octadeca-8,13-dienoic acid	C18:2t8c13	polyunsaturated acid 12
(Z,E)-octadeca-9,12-dienoic acid	C18:2c9t12	polyunsaturated acid 13
(E,Z)-octadeca-9,12-dienoic acid ?	C18:2t9c12	polyunsaturated acid 14
(E,Z)-octadeca-11,15-dienoic acid ?	C18:2t11c15	polyunsaturated acid 15
(9Z,12Z)-octadeca-9,12-dienoic acid, linoleic acid	C18:2c9c12n6	polyunsaturated acid 16
(Z,Z)-octadeca-9,15-dienoic acid ?	C18:2c9c15	polyunsaturated acid 17
(Z,Z)-octadeca-11,14-dienoic acid	C18:2n4	polyunsaturated acid 18
(Z,Z)-octadeca-?,?-dienoic acid ?	C18:2 isomer02	polyunsaturated acid 19
all-cis-6,9,12-octadecatrienoic acid, gamma-linolenic, pinolenic acid	C18:3n6	polyunsaturated acid 22
all-cis-9,12,15-octadecatrienoic acid, alpha-linolenic acid	C18:3n3	polyunsaturated acid 23
(?,?)-eicosadienoic acid	C20:2 isomer01	polyunsaturated acid 34
(?,?)-eicosadienoic acid	C20:2 isomer02	polyunsaturated acid 35
(Z11,Z14)-eicosadienoic acid	C20:2n6	polyunsaturated acid 37
all-cis-5,8,11,14-eicosatetraenoic acid, arachidonic acid	C20:4n6	polyunsaturated acid 41
all-cis-13,16-docosadienoic acid, cetoleic acid	C22:2n6	polyunsaturated acid 51
3,7,11,15-tetramethyl exadecanoic acid	Phytanic	branched chain
cis-9,10-methyleneoctadecanoic acid	C19:0Δ	cyclic 02
2,6,10,14-tetramethylpentadecanoic acid	Pristanic	terpenoid acid

Table S2

Percentage on total mass (mean value, n = 3) of identified fatty acid peaks along the section of the droveway (West Pit, Central, East Pit and Extern) and controls (reference profile).

		Reference					West Pit			Central								
		Ap	2a/c	2BC	3B	4CK	13	12	11	10	9	8	3	7	1	6	5	4
Fatty acid		25-30	40	50/70	80-110	120	25/30	40/50	70	25/30	40/50	70	25/30	40/50	80/100	70	80/100	120
C6:0	Saturated	0.21	0.56	0.48	1.12	1.06	0.40	1.20	0.17	0.69	0.91	1.46	0.72	0.70	1.16	0.99	1.19	1.12
C7:0		0.21	0.40	0.37	0.94	0.55	0.43	0.73	0.12	0.76	0.78	1.30	0.58	0.58	0.82	0.55	0.88	1.16
C8:0		0.27	0.61	0.49	0.93	0.94	0.67	1.24	0.32	1.10	0.88	1.72	1.06	0.97	1.43	1.00	1.21	1.25
C8:0 iso		0.03	0.06	0.03	0.07	0.06	0.09	0.07	0.00	0.10	0.11	0.11	0.11	0.10	0.08	0.09	0.09	0.10
C9:0		0.39	0.78	0.56	1.23	0.94	1.25	1.28	0.44	2.37	1.54	2.40	1.86	1.78	1.59	1.55	1.58	1.46
C10:0 iso?		0.02	0.04	0.02	0.00	0.05	0.08	0.07	0.00	0.08	0.08	0.05	0.07	0.08	0.07	0.10	0.06	0.07
C10:0		0.98	0.69	0.68	0.94	0.93	0.72	1.34	0.44	1.16	0.84	1.51	1.03	0.94	1.18	0.94	0.98	0.94
C11:0 iso		0.03	0.05	0.02	0.08	0.07	0.15	0.14	0.10	0.20	0.16	0.00	0.15	0.15	0.00	0.12	0.10	0.12
C11:0 branced?		0.03	0.03	0.00	0.05	0.00	0.04	0.06	0.00	0.10	0.05	0.07	0.04	0.07	0.06	0.05	0.00	0.00
C11:0 anteiso		0.00	0.03	0.00	0.00	0.00	0.05	0.04	0.00	0.01	0.05	0.00	0.05	0.05	0.00	0.07	0.00	0.04
C11:0		0.09	0.09	0.06	0.14	0.12	0.22	0.16	0.14	0.22	0.21	0.19	0.21	0.24	0.15	0.22	0.16	0.17
C12:0 iso		0.02	0.02	0.01	0.00	0.00	0.11	0.05	0.08	0.06	0.09	0.00	0.10	0.08	0.05	0.09	0.07	0.04
C12:0		33.35	2.47	3.85	1.02	1.55	0.96	1.40	1.08	1.44	1.06	1.32	1.29	1.14	1.24	1.28	1.24	1.06
C13:0 branced?		0.03	0.02	0.00	0.07	0.09	0.03	0.00	0.00	0.14	0.00	0.06	0.08	0.07	0.11	0.00	0.11	0.00
C13:0 iso		0.03	0.07	0.00	0.12	0.11	0.20	0.13	0.15	0.16	0.20	0.12	0.24	0.17	0.15	0.23	0.15	0.18
C13:0 anteiso		0.08	0.07	0.05	0.13	0.08	0.10	0.09	0.07	0.18	0.11	0.07	0.13	0.12	0.16	0.18	0.17	0.15
C13:0		0.11	0.14	0.10	0.18	0.23	0.25	0.19	0.28	0.30	0.28	0.29	0.29	0.28	0.28	0.37	0.29	0.28
C14:0 iso		0.19	0.36	0.22	0.53	0.94	0.70	0.45	0.75	0.66	0.72	0.58	0.80	0.69	0.70	1.04	0.82	0.81
C14:0 anteiso		0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.00	0.00	0.02	0.00	0.04	0.03	0.00	0.00	0.00	0.00
C14:0		7.44	1.85	1.93	2.32	2.76	1.92	3.70	2.24	3.18	2.31	3.03	2.56	2.50	2.99	2.98	3.24	2.71
C15:0 iso		0.47	0.70	0.38	0.79	0.72	2.54	1.27	2.49	1.25	2.12	1.37	2.57	1.92	1.54	2.64	1.81	2.04
C15:0 anteiso		0.51	0.96	0.55	1.22	1.45	1.84	1.32	1.86	1.65	1.91	1.62	2.02	1.96	1.96	2.82	2.24	2.26
C15:0		0.32	0.63	0.48	1.06	1.25	1.07	0.91	1.19	1.47	1.29	1.33	1.27	1.22	1.48	1.54	1.52	1.53
C16:0 iso		0.26	0.41	0.26	0.64	0.75	1.62	0.85	1.91	0.97	1.65	0.79	1.72	1.32	1.34	1.88	1.46	1.68
C16:0 anteiso		0.00	0.00	0.00	0.00	0.00	0.12	0.03	0.11	0.03	0.10	0.00	0.13	0.09	0.05	0.14	0.00	0.083

C16:0		14.25	18.26	16.93	20.59	18.37	14.66	20.05	13.52	18.62	15.21	18.86	13.35	14.50	15.87	16.42	18.33	18.48
C17:0 branced?		0.18	0.39	0.16	0.25	0.15	1.27	0.89	1.39	0.74	1.31	1.47	1.26	1.44	1.65	1.42	1.33	1.31
C17:0 branced1?		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.24	0.00	0.27	0.00	0.14	0.30	0.07	0.24	0.04
C17:0 branced2?		0.01	0.07	0.10	0.05	0.10	0.18	0.03	0.08	0.21	0.18	0.14	0.15	0.14	0.15	0.18	0.19	0.05
C17:0 iso		0.22	0.37	0.25	0.23	0.27	0.93	1.56	1.09	0.50	0.77	1.29	0.97	1.77	1.76	0.97	0.64	0.72
C17:0 anteiso		0.00	0.27	0.17	0.34	0.42	0.85	1.44	1.14	0.50	0.75	0.48	0.87	0.92	0.66	1.01	0.73	1.13
C17:0		0.27	0.32	0.26	0.42	0.54	0.74	0.58	1.99	0.65	0.79	0.54	0.81	0.65	0.66	0.83	0.69	0.75
C18:0 branced?		0.00	0.00	0.00	0.00	0.00	0.15	0.00	0.09	0.04	0.10	0.00	0.17	0.03	0.00	0.10	0.03	0.04
C18:0 iso		0.00	0.00	0.03	0.05	0.07	0.15	0.11	0.05	0.04	0.15	0.06	0.02	0.08	0.06	0.04	0.04	0.11
C18:0 anteiso		0.01	0.06	0.04	0.05	0.00	0.00	0.03	0.00	0.00	0.00	0.00	0.02	0.07	0.08	0.04	0.08	0.00
C18:0		3.64	5.72	5.25	6.89	7.55	5.72	8.20	6.49	7.61	6.33	9.83	6.03	6.76	7.36	6.98	7.56	8.07
C19:0 branced?		0.01	2.15	1.56	2.89	2.85	0.05	0.28	0.00	0.49	0.17	0.41	0.17	0.42	0.70	0.18	0.62	0.00
C19:0 iso		0.00	0.00	0.00	0.00	0.00	0.03	0.00	0.00	0.00	0.00	0.00	0.03	0.00	0.00	0.00	0.00	0.00
C19:0 branced?		0.00	0.23	0.29	0.29	0.35	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C19:0		0.11	0.14	0.10	0.12	0.12	0.36	0.23	0.36	0.21	0.35	0.27	0.39	0.29	0.12	0.31	0.19	0.29
C20:0		1.65	1.77	1.96	3.32	4.55	4.58	3.78	5.12	2.41	4.08	3.94	5.28	3.81	4.29	4.29	2.75	3.24
C21:0 branced?		0.00	0.04	0.17	0.07	0.13	0.30	1.48	0.00	2.86	0.91	2.34	1.00	2.99	4.28	1.22	4.10	0.00
C21?0 branced?		0.00	0.00	0.00	0.00	0.00	0.00	0.21	0.00	0.00	0.10	0.06	0.08	0.25	0.41	0.04	0.61	0.00
C21:0		0.31	0.28	0.18	0.26	0.20	0.82	0.31	1.03	0.39	0.70	0.26	0.88	0.72	0.25	0.50	0.42	0.52
C22:0		1.74	1.49	0.92	1.40	3.03	4.76	2.06	5.11	2.73	4.22	2.60	5.22	4.42	2.25	3.99	2.90	3.96
C23:0		1.28	0.70	0.33	0.41	0.35	3.50	0.73	3.80	0.92	2.68	0.92	3.69	2.67	0.69	1.81	1.15	2.15
C24:0		4.68	2.78	1.30	2.00	3.30	13.40	3.01	13.90	3.94	9.85	3.28	13.77	9.53	3.05	7.54	4.71	8.09
C25:0		0.11	0.00	0.00	0.00	0.00	0.00	0.00	0.45	0.10	0.20	0.00	0.00	0.05	0.00	0.00	0.00	0.00
C19:0 anteiso		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.02	0.00	0.00	0.00	0.00
C15:1 anteiso?	Monounsaturated	0.00	0.00	0.00	0.09	0.00	0.01	0.02	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.05	0.00	0.04
C15:1 iso?		0.00	0.05	0.00	0.08	0.21	0.04	0.02	0.00	0.21	0.07	0.09	0.10	0.07	0.20	0.07	0.18	0.07
C10:1		0.00	0.00	0.00	0.00	0.00	0.00	0.08	0.00	0.02	0.00	0.00	0.00	0.02	0.00	0.00	0.00	0.00
C11:1		0.04	0.15	0.14	0.48	0.21	0.14	0.26	0.05	0.76	0.03	0.30	0.23	0.22	0.26	0.45	0.19	0.28
C12:1cn?		0.06	0.00	0.00	0.13	0.00	0.00	0.00	0.00	0.02	0.00	0.00	0.00	0.00	0.04	0.00	0.00	0.00
C12:1tn?		0.06	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C13:1 iso?		0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C14:1tn?		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C14:1tn5		0.02	0.00	0.00	0.00	0.00	0.03	0.00	0.00	0.01	0.02	0.00	0.02	0.01	0.00	0.00	0.00	0.00
C12:1cn1		0.01	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.00	0.00	0.00

C14:1n7	0.04	0.09	0.10	0.34	0.52	0.09	0.19	0.20	0.52	0.18	0.38	0.20	0.17	0.21	0.15	0.46	0.29
C14:1n?cis	0.00	0.00	0.03	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.21	0.15	0.00	0.00
C14:1cn5	0.21	0.08	0.05	0.32	0.20	0.05	0.25	0.00	0.10	0.02	0.00	0.03	0.03	0.00	0.05	0.04	0.00
C14:1n?	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.02	0.00	0.03	0.02	0.00	0.07	0.00	0.00
C14:1n?	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.04	0.00
C15:1t isomer 01	0.07	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.02	0.00	0.00	0.00	0.00	0.04	0.00	0.04	0.00
C15:1tn?	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.02	0.00	0.00	0.00	0.00
C15:1t isomer 02	0.00	0.09	0.00	0.05	0.00	0.09	0.04	0.04	0.04	0.10	0.09	0.10	0.10	0.00	0.09	0.07	0.07
C15:1n5t	0.01	0.00	0.00	0.00	0.00	0.00	0.02	0.00	0.00	0.00	0.00	0.00	0.02	0.00	0.00	0.00	0.00
C13:1?n?c	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C15:1 isomer01	0.00	0.06	0.08	0.22	0.31	0.02	0.10	0.12	0.34	0.12	0.21	0.15	0.12	0.32	0.21	0.28	0.20
C15:1 isomer02	0.00	0.00	0.00	0.00	0.00	0.09	0.00	0.00	0.00	0.02	0.00	0.03	0.02	0.00	0.00	0.00	0.00
C15:1n5	0.01	0.00	0.00	0.00	0.00	0.04	0.02	0.00	0.02	0.08	0.00	0.02	0.04	0.00	0.00	0.00	0.00
C16:1 iso?	0.01	0.00	0.00	0.00	0.00	0.10	0.00	0.00	0.00	0.05	0.00	0.11	0.05	0.00	0.00	0.00	0.04
C16:1 branced?	0.01	0.10	0.13	0.40	0.53	0.05	0.13	0.00	0.59	0.07	0.15	0.13	0.14	0.42	0.29	0.46	0.13
C16:1 branced?	0.12	0.15	0.12	0.31	0.45	0.30	0.30	0.00	0.26	0.32	0.21	0.37	0.36	0.34	0.34	0.42	0.32
C16:1t isomer01	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.06	0.00	0.02	0.07	0.00	0.00	0.00
C16:1n10t	0.01	0.28	0.15	0.15	0.19	0.29	0.16	0.12	0.41	0.27	0.46	0.17	1.40	1.31	0.08	0.00	0.00
C16:1n7t	0.02	0.00	0.06	0.00	0.00	0.01	0.05	0.00	0.00	0.02	0.12	0.00	0.02	0.08	0.11	0.34	0.00
C16:1n6t?	0.04	0.00	0.05	0.08	0.11	0.00	0.11	0.00	0.00	0.02	0.00	0.10	0.02	0.00	0.00	0.00	0.00
C16:1n5t?	0.00	0.03	0.00	0.00	0.00	0.03	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C16:1n9c	0.27	0.67	0.78	2.40	2.95	0.44	2.04	1.15	2.97	0.89	1.98	1.09	1.30	2.70	1.60	2.13	1.26
C16:1n7c	2.64	0.50	0.47	0.57	0.67	1.42	2.51	1.75	0.81	2.47	0.60	1.47	1.84	0.74	1.15	0.85	1.37
C16:1n5c	1.49	2.40	1.44	6.93	0.81	3.78	1.23	2.29	0.69	2.22	0.52	1.97	1.38	0.55	1.73	2.11	2.97
C16:1n3c?	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.10	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C16:1cis isomer03	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.05	0.04	0.00	0.00	0.01	0.00	0.00	0.00	0.00	0.00
C17:1n7t	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.00	0.02	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C17:1n9	0.00	0.66	0.21	0.42	0.43	0.04	0.65	0.00	0.00	0.00	0.86	0.91	0.00	1.37	0.00	1.64	0.00
C17:1n6t	0.00	0.00	0.03	0.05	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C17:1?	0.32	0.08	0.05	0.00	0.00	0.50	0.25	0.00	0.27	0.42	0.00	0.55	0.32	0.14	0.49	0.19	0.27
C17:1n6?	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.06	0.00	0.00	0.00	0.00	0.00
C17:1n5	0.00	0.00	0.00	0.00	0.00	0.11	0.02	0.00	0.03	0.11	0.00	0.05	0.07	0.00	0.00	0.00	0.00
C17:1n4	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C17:1? branced	0.21	0.03	0.10	0.33	0.44	0.14	0.11	0.00	0.42	0.20	0.15	0.22	0.09	0.33	0.15	0.16	0.14

C19:1tn?		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.07	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C17:1n8		0.00	0.10	0.08	0.07	0.00	0.00	0.00	0.00	1.31	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C17:1n7c		0.12	0.00	0.00	0.00	0.00	0.57	0.43	0.46	0.31	1.06	0.17	0.41	0.31	0.40	0.47	0.32	0.43
C18:1n14t?		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.10	0.00	0.00	0.00	0.00	0.00	0.00
C18:1n10t?		0.00	0.00	0.00	0.00	0.13	0.00	0.00	0.00	0.00	0.00	0.20	0.32	0.00	0.08	0.00	0.00	0.00
C18:1n9t		0.31	0.23	0.19	0.20	0.11	0.31	0.46	0.38	0.37	0.33	0.10	0.00	0.37	0.17	0.41	0.37	0.18
C18:1n8t		0.04	0.00	0.00	0.00	0.00	0.00	0.41	0.08	0.08	0.00	0.08	0.00	0.00	0.05	0.06	0.00	0.12
C18:1n7t		0.00	0.13	0.13	0.50	0.10	0.14	0.00	0.00	0.07	0.11	0.05	0.09	0.12	0.00	0.00	0.08	0.00
C18:1n9c		9.22	16.42	16.84	5.34	11.35	8.75	11.34	9.91	6.65	8.94	6.21	5.45	7.02	5.63	5.88	5.25	5.25
C18:1n7c		0.93	1.86	1.58	6.05	1.02	3.26	1.31	1.74	1.45	2.09	0.94	1.55	1.29	1.11	1.36	1.31	1.99
C18:1n6c		0.18	0.15	0.03	0.00	0.00	0.01	0.12	0.00	0.04	0.00	0.05	0.02	0.04	0.00	0.06	0.05	0.04
C18:1n5c		0.04	0.10	0.05	0.26	0.07	0.27	0.27	0.45	0.03	0.25	0.06	0.24	0.16	0.05	0.10	0.06	0.17
C18:1n2t		0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.04	0.00	0.00	0.00	0.03	0.00	0.00	0.00	0.05	0.04
C18:1n4c		0.03	0.10	0.02	0.00	0.00	0.01	0.06	0.05	0.05	0.02	0.10	0.03	0.01	0.11	0.05	0.04	0.04
C18:1n3c		0.04	0.08	0.03	0.10	0.06	0.01	0.02	0.06	0.01	0.02	0.11	0.03	0.02	0.06	0.04	0.05	0.06
C18:1n2c		0.00	0.03	0.03	0.05	0.17	0.01	0.04	0.12	0.02	0.02	0.20	0.02	0.02	0.24	0.06	0.04	0.05
C19:1n9t		0.00	0.00	0.00	0.00	0.00	0.03	0.02	0.00	0.00	0.00	0.11	0.00	0.00	0.00	0.00	0.00	0.00
C19:1n12		0.20	0.06	0.03	0.06	0.00	1.08	0.31	1.04	0.18	1.24	0.06	0.81	0.40	0.05	0.00	0.19	0.30
C19:1n9c		0.00	0.00	0.03	0.00	0.00	0.07	0.00	0.00	0.02	0.03	0.08	0.01	0.00	0.00	0.00	0.00	0.00
C20:1tn9		0.00	0.00	0.00	0.00	0.06	0.08	0.05	0.00	0.19	0.16	0.10	0.33	0.07	0.09	0.06	0.00	0.05
C20:1n12		0.00	0.00	0.00	0.00	0.00	0.03	0.00	0.00	0.00	0.63	0.76	0.43	0.00	0.73	0.00	0.77	0.67
C20:1n9		0.13	0.35	0.34	0.56	0.88	0.48	0.55	0.43	0.84	0.07	0.00	0.02	0.58	0.00	0.80	0.00	0.00
C20:1n7		0.01	0.02	0.00	0.12	0.06	0.15	0.06	0.04	0.18	0.17	0.19	0.10	0.13	0.15	0.19	0.23	0.19
C22:1tn9		0.00	0.07	0.19	0.61	1.41	0.74	3.25	1.23	2.37	1.27	2.23	0.34	1.79	2.02	1.34	1.13	0.91
C22:1cis n9		1.77	4.61	4.72	12.66	11.50	4.46	6.07	4.01	10.64	6.15	10.46	4.36	7.11	10.65	8.53	9.35	8.69
C22:1cis n7		0.02	0.12	0.09	0.14	0.30	0.02	0.16	0.00	0.23	0.00	0.09	0.00	0.14	0.19	0.07	0.19	0.16
C24:1		0.01	0.05	0.02	0.19	0.00	0.34	0.04	0.00	0.15	0.03	0.00	0.11	0.05	0.04	0.05	0.09	0.12
?MeC16:1?		0.02	0.00	0.00	0.00	0.00	0.15	0.03	0.79	0.06	0.14	0.00	0.17	0.09	0.00	0.08	0.00	0.04
7Me 6MeC16:1		0.00	0.00	0.00	0.06	0.00	0.01	0.00	0.04	0.03	0.00	0.08	0.02	0.00	0.06	0.00	0.00	0.16
C12:2	Polyunsaturated	0.00	0.22	0.21	0.58	0.22	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C14:2n?		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C16:2n?		0.00	0.03	0.07	0.25	0.18	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C16:2 NMI		0.03	0.00	0.00	0.00	0.00	0.02	0.09	0.04	0.00	0.04	0.08	0.09	0.00	0.17	0.15	0.00	0.00
C13:2?		0.00	0.05	0.04	0.11	0.06	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00

C16:2n7		0.00	0.00	0.09	0.00	0.00	0.05	0.00	0.00	0.00	0.05	0.00	0.00	0.00	0.07	0.00	0.00	0.00
C16:2 isomer		0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.09	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C14:2		0.01	0.00	0.00	0.00	0.00	0.05	0.08	0.00	0.12	0.07	0.16	0.06	0.10	0.14	0.10	0.05	0.12
C14:2?		0.00	0.06	0.05	0.17	0.15	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C15:2?		0.00	0.00	0.03	0.10	0.08	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C17:2n?		0.00	0.00	0.00	0.08	0.10	0.02	0.03	0.00	0.00	0.07	0.05	0.04	0.00	0.00	0.00	0.00	0.04
C18:2t?t? 01		0.03	0.06	0.07	0.37	0.27	0.01	0.00	0.00	0.04	0.02	0.07	0.00	0.00	0.00	0.05	0.00	0.00
C18:2t9t12		0.05	0.04	0.05	0.11	0.34	0.01	0.14	0.00	0.00	0.00	0.05	0.00	0.03	0.09	0.07	0.00	0.04
C18:2c9t13		0.01	0.03	0.00	0.10	0.00	0.05	0.16	0.05	0.28	0.02	0.05	0.11	0.09	0.19	0.14	0.18	0.10
C18:2t8c13		0.02	0.12	0.19	0.06	0.21	0.07	0.09	0.06	0.14	0.13	0.10	0.11	0.13	0.05	0.07	0.13	0.14
C18:2c9t12		0.00	0.00	0.08	0.09	0.00	0.06	0.00	0.00	0.17	0.06	0.10	0.02	0.00	0.16	0.09	0.11	0.12
C18:2t9c12		0.00	0.00	0.00	0.00	0.00	0.00	0.03	0.05	0.04	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C18:2t11c15		0.10	0.10	0.15	0.05	0.00	0.02	0.02	0.09	0.00	0.00	0.09	0.00	0.00	0.04	0.00	0.00	0.00
C18:2c9c12n6		5.93	21.02	27.09	2.76	3.97	1.64	2.48	2.32	2.23	1.50	2.62	1.68	1.63	2.86	1.82	2.14	2.16
C18:2c9c15		0.06	0.00	0.00	0.11	0.06	0.01	0.02	0.04	0.06	0.04	0.11	0.00	0.02	0.04	0.12	0.05	0.00
C18:2n4		0.03	0.03	0.07	0.00	0.06	0.00	0.11	0.05	0.05	0.09	0.23	0.05	0.00	0.06	0.12	0.14	0.04
C18:2 isomer02		0.03	0.00	0.11	0.11	0.00	0.00	0.15	0.06	0.09	0.00	0.22	0.11	0.43	0.28	0.17	0.12	0.70
C18:3n6		0.02	0.00	0.04	0.15	0.07	0.04	0.03	0.11	0.16	0.11	0.08	0.12	0.06	0.18	0.04	0.06	0.07
C18:3n3		0.88	1.66	2.25	0.55	1.10	0.31	0.53	0.21	0.37	0.11	0.05	0.58	0.16	0.54	0.21	0.21	0.06
C20:2 isomer01		0.00	0.00	0.00	0.00	0.00	0.07	0.02	0.00	0.01	0.00	0.00	0.04	0.12	0.06	0.00	0.00	0.00
C20:2 isomer02		0.00	0.03	0.00	0.08	0.06	0.00	0.05	0.05	0.10	0.10	0.07	0.03	0.00	0.06	0.08	0.14	0.15
C20:2n6		0.01	0.02	0.00	0.00	0.08	0.02	0.02	0.00	0.00	0.09	0.06	0.01	0.05	0.00	0.00	0.00	0.03
C20:4n6		0.01	0.02	0.00	0.00	0.00	0.06	0.03	0.00	0.04	0.06	0.00	0.00	0.02	0.00	0.00	0.00	0.00
C22:2n6		0.05	0.11	0.08	0.25	0.38	0.16	0.12	0.10	0.13	0.18	0.17	0.16	0.19	0.12	0.22	0.25	0.36
Phytanic	Branced	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.10	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.07	0.16
C19:0Δ	Cyclic	0.13	0.06	0.06	0.00	0.00	0.03	0.04	0.00	0.13	0.03	0.05	0.08	0.03	0.07	0.47	0.09	0.00
Pristanic	Terpenoic	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
SFA		73.7	46.2	40.7	53.4	57.2	68.2	61.9	69.7	61.7	66.5	66.6	73.4	69.1	63.4	69.0	66.9	68.4
UFA		26.2	53.7	59.2	46.6	42.8	31.8	38.1	30.2	38.2	33.5	33.3	26.5	30.9	36.6	30.5	33.0	31.5
MUFA		18.9	30.1	28.5	40.5	35.4	29.0	33.8	26.9	34.0	30.7	28.9	23.2	27.8	31.4	27.0	29.3	27.3
PUFA		7.3	23.7	30.7	6.2	7.5	2.8	4.3	3.3	4.2	2.8	4.4	3.3	3.1	5.2	3.5	3.6	4.2
Branced		1.4	4.3	3.4	5.9	6.5	6.8	7.6	6.8	8.3	7.3	7.9	8.2	10.6	12.7	8.6	11.3	6.4
P/S		0.1	0.5	0.8	0.1	0.1	0.0	0.1	0.0	0.1	0.0	0.1	0.0	0.0	0.1	0.1	0.1	0.1

C15:0+C17:0	0.60	0.96	0.76	1.49	1.80	1.81	1.49	3.19	2.13	2.09	1.88	2.08	1.88	2.15	2.39	2.22	2.29
(C15:0+C17:0)/(C12:0+C14:0+C16:0+C18:0)	0.01	0.03	0.03	0.05	0.06	0.08	0.04	0.14	0.07	0.08	0.06	0.09	0.08	0.08	0.09	0.07	0.08
UFA/SFA	0.35	1.16	1.45	0.87	0.75	0.46	0.61	0.43	0.62	0.50	0.50	0.36	0.45	0.57	0.44	0.49	0.46

Author Agreement

Conflict of Interest or Competing Interest

Declaration of Interest

All authors have seen and approved the final version of the manuscript being submitted. They warrant that the article is the authors' original work, hasn't received prior publication and isn't under consideration for publication elsewhere.

No conflict of interest or competing interest is present.

All authors declare that there's no financial/personal interest that could affect their objectivity.

2022, March the first

Legnaro, Padova

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