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The geochemical characterization of two long distance chert tracers by ED-XRF and LA-ICP-MS. Implications for Magdalenian human mobility in the Pyrenees (SW Europe)

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ABSTRACT
We geochemically characterize two chert formations outcropping in the Pyrenees and presenting similar characteristics at the visual and microscopic scale: The Montgaillard flysch cherts and the Montsaunès cherts. Cherts presenting identical textural and micropalaeontological features as both types have been found in several Magdalenian Pyrenean sites. We are face to a long distance chert type whose geochemical characterization is essential for knowing where the tracer comes from. Analyses have been done using Energy-dispersive X-ray fluorescence (ED-XRF) and laser ablation inductively-coupled plasma mass spectrometry (LA-ICP-MS). Results show that despite obtaining similar data concerning major and minor elements, differences have been observed regarding trace elements. The establishment of differences between both formations at the geochemical level has allowed specifying the origin of this chert type recovered at the Magdalenian levels of Parco Cave (Alòs de Balaguer, Spain). Results demonstrate long lithic raw material circulation and thus, human mobility in the Pyrenees during the Upper Palaeolithic.

KEYWORDS
Chert; Pyrenees; Magdalenian; raw material characterization; geochemistry; lithic procurement

Introduction

Lithic raw material characterization is essential in Palaeolithic studies for knowing the relationship that hunter-gatherer groups had with their environment. Concerning the SW Europe and more specifically the Pyrenees mountain range, studies have mostly focused on the analyses of textural and petrographic characteristics (Terradas 2001; Grégoire 2000; Normand 2002; Ortega 2002; Foucher 2004; Briois 2005; Mangado 2005) and only a few attempts to geochemically characterize chert artefacts have been done until now. Most of them have been dedicated on the mineralogical determination (Roy-Sunyer et al. 2013) and none has attempted to determine the elemental composition. Nevertheless, in other areas several geochemical characterizations of chert artefacts were done in the last two decades, focusing on the use of one or more combined techniques, as studies concerning North America (Hawkins et al. 2008; Milne, Hamilton, and Fayek 2009; Gauthier and Burke 2011; Parish 2011; Gauthier, Burke, and Leclerc 2012; Hassler et al. 2013; Parish, Swihart, and Li 2013; Speer 2014b, 2014a; Bruggencate et al. 2016; Speer 2016; Parish 2016), the Middle East (Ekshtain et al. 2014), Northern Europe (Owen, Armstrong, and Floyd 1999; Evans et al. 2007; Olofsson and Rodushkin 2011; Hogberg, Olausson, and Hughes 2012; Hughes, Hogberg, and Olausson 2012), Eastern Europe (Hughes, Baltrunas, and Kulbickas 2011; Gurova et al. 2016) and Western Europe (Bressy et al. 2008; Navazo et al. 2008; Olivares et al. 2009; Roldan et al. 2015; Vallejo Rodríguez, Urtiaga Greaves, and Navazo Ruiz 2015; Moreau et al. 2016; Blet, Binder, and Gratuze 2000).

The Pyrenean mountain range is a mountain chain located in South-Western Europe and naturally dividing in the S-N axis the Iberian Peninsula from the rest of the continental Europe. It extends for almost 500 km from the Bay of Biscay to the Mediterranean Sea and today is the natural border dividing France and Spain (figure 1). Archaeological works in the Pyrenean region, developed since last century (Mangado et al. 2010; Utrilla et al. 2010), have confirmed that this natural barrier was occupied, at least in the eastern margins, since the Lower Palaeolithic (Falguères et al. 2015; de Lumley et al. 2004). Studies concerning the homogeneity between Cantabrian and Pyrenean rock art (Garate et al. 2015), lithic techno-typological analyses (Langlais 2011; Langlais et al. 2016) and lithic raw material procurement (Sánchez de la Torre 2015) have also demonstrated that contacts between both

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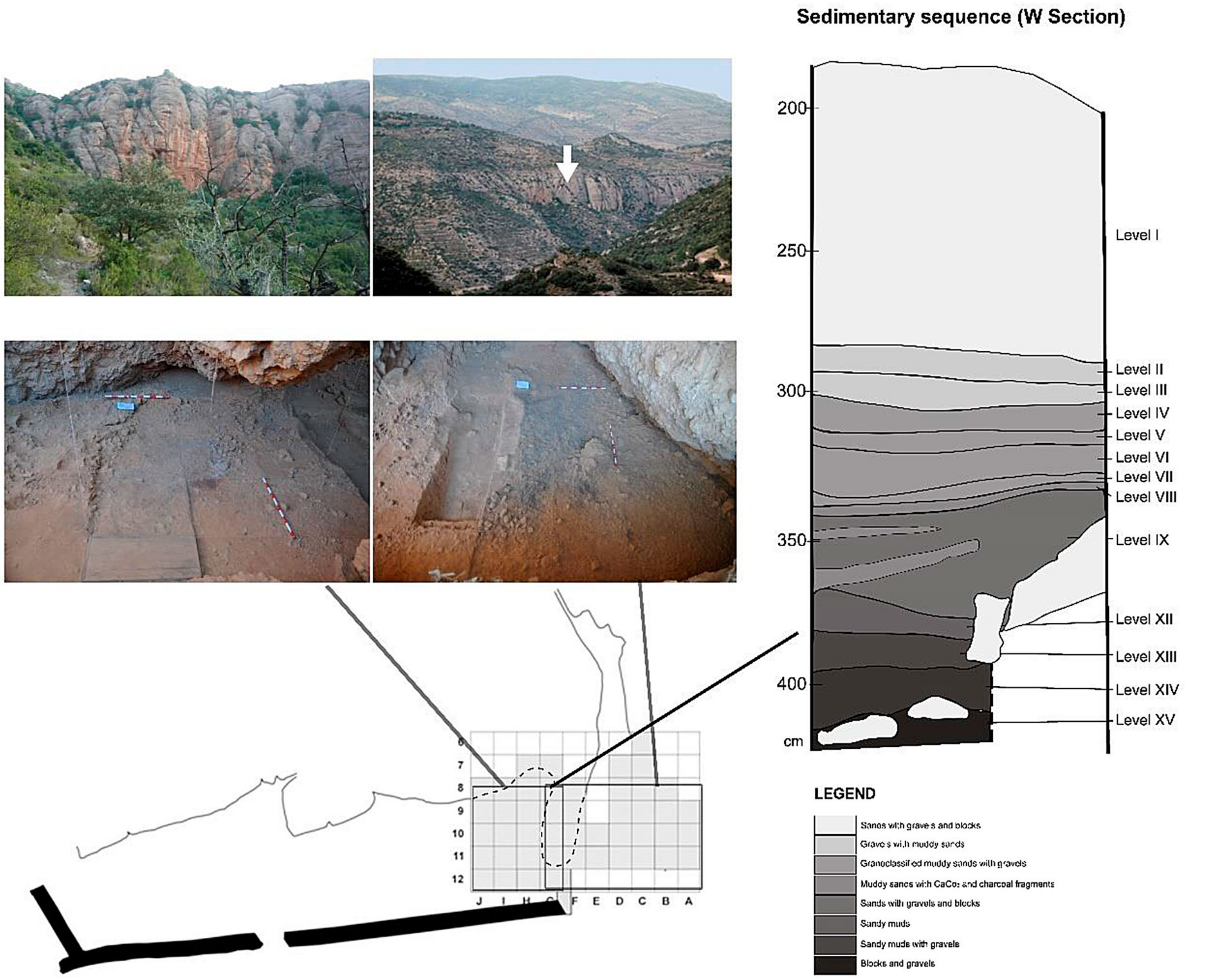

Figure 1. Parco Cave site. Location, excavation state in 2016 and plant (left, from the top to the bottom). Sedimentary sequence from the W Maluquer's trial excavation established by Bergadà (1998) (Modified).

Pyrenean slopes existed, as least, since the Upper Palaeolithic.

Parco Cave (Alòs de Balaguer, Spain) is an archaeological site located in southern Pyrenees, in the Segre river valley at 420 m asl, in a sheltered area, with a human occupation from the Palaeolithic to the Bronze Age (Mangado et al. 2010; Fullola et al. 2012; Mangado et al. 2015; Mangado et al. 2014). Discovered in the seventies and first dug by Maluquer de Motes (Maluquer de Motes 1983–1984, 1985), the site is under excavation by the Seminar of Studies and Research into Prehistory (SERP) from the University of Barcelona since 1987. The Magdalenian period is well represented, with several levels going from the Middle to the Late Upper Magdalenian (Fullola et al. 2012; Mangado et al. 2014; Mangado et al. 2015). The Late Upper Magdalenian period is dated in 14,662–15,260 cal BP, 14,426–15,055 cal BP and 14,535–15,234 cal BP. The Upper Magdalenian level has three radiometric dates: 15,447–16,245 cal BP, 15,503–16,293 cal BP and 15,616–16,387 cal BP and is characterized by the appearance of elongated scalene triangles (Langlais 2011). The Middle Magdalenian, still in excavation, has two radiocarbon dates: 15,778–16,592 cal BP and 16,022–16,839 cal BP (table 1).

From the excavation's initial stratigraphic sequence, it was possible to determine the existence of up to eleven sedimentary levels with cultural remains (Bergadà

Table 1. Radiocarbon dates from the Magdalenian occupation of Parco Cave. Calendar age calculated by Online Calpal (quickcal2007 ver.1.5). CalCurve: CalPal_2007_HULU. Lab.Ref.: Laboratory reference; Met.: Method; C: Charcoal; S.: Sample nature.

Period	Data	Lab. Ref.	Met.	S.	Calendar 68% range Cal BP	Calendric Age Cal BC	Reference
Late Up. Mag.	12605±60 BP	OxA-10796	AMS	C	14662–15260	13011±299	Fullola *et al.* 2012
Late Up. Mag.	12460±60 BP	OxA-10797	AMS	C	14426–15055	12791±314	Mangado *et al.* 2006
Late Up. Mag.	12560±130 BP	OxA-10835	AMS	C	14535–15234	12935±349	Mangado et al. 2006
Up. Mag.	12995±50 BP	OxA-13597	AMS	C	15447–16245	13896±399	Mangado et al. 2006
Up. Mag.	13025±50 BP	OxA-13596	AMS	C	15503–16293	13948±395	Mangado et al. 2006
Up. Mag.	13095±55 BP	OxA-17730	AMS	C	15616–16387	14052±385	Mangado *et al.* 2010
Middle Mag.	13255±50 BP	OxA-29336	AMS	C	15778–16592	14235±407	Mangado *et al.* 2014
Middle Mag.	13475±50 BP	OxA-23650	AMS	C	16022–16839	14481±408	Mangado *et al.* 2014

1998) (figure 1). Magdalenian occupations are characterized by a great complexity of anthropic elements structuring the space in the form of hearths or debris deposits. A variety of activities in the site has been documented thanks to use-wear and typological analysis of lithic artefacts. There are signs of hide-working (Calvo 2004) and even the smoking of these skins (Bergadà 1998).

Concerning lithic artefacts, chert was the most used rock type to make lithic tools. The archaeopetrological study of the Magdalenian levels (Mangado 2005; Sánchez de la Torre 2015) have shown the existence of several chert types. Local and regional siliceous varieties are the best represented chert types in all the Magdalenian sequence. Nevertheless, other chert types whose origin is exogenous have been detected, among which a marine chert type representing parallels with two chert types outcropping in the northern Pyrenean slopes: the Montgaillard flysch cherts and the Montsaunès cherts (figure 2). This siliceous raw material appears in a few average but repeatedly and is regularly well represented in all the Magdalenian sequence as it will be exposed later on.

The Montgaillard flysch cherts are located in the flysch limestone from the Turonian to the Santonian outcropping in primary position near Montgaillard (Hautes-Pyrénées, France) and in secondary position near Hibarette, Bénac, Saint Martin and Visker (Hautes-Pyrénées, France), where lithic remains of ancient knapping were found (Barragué et al. 2001).

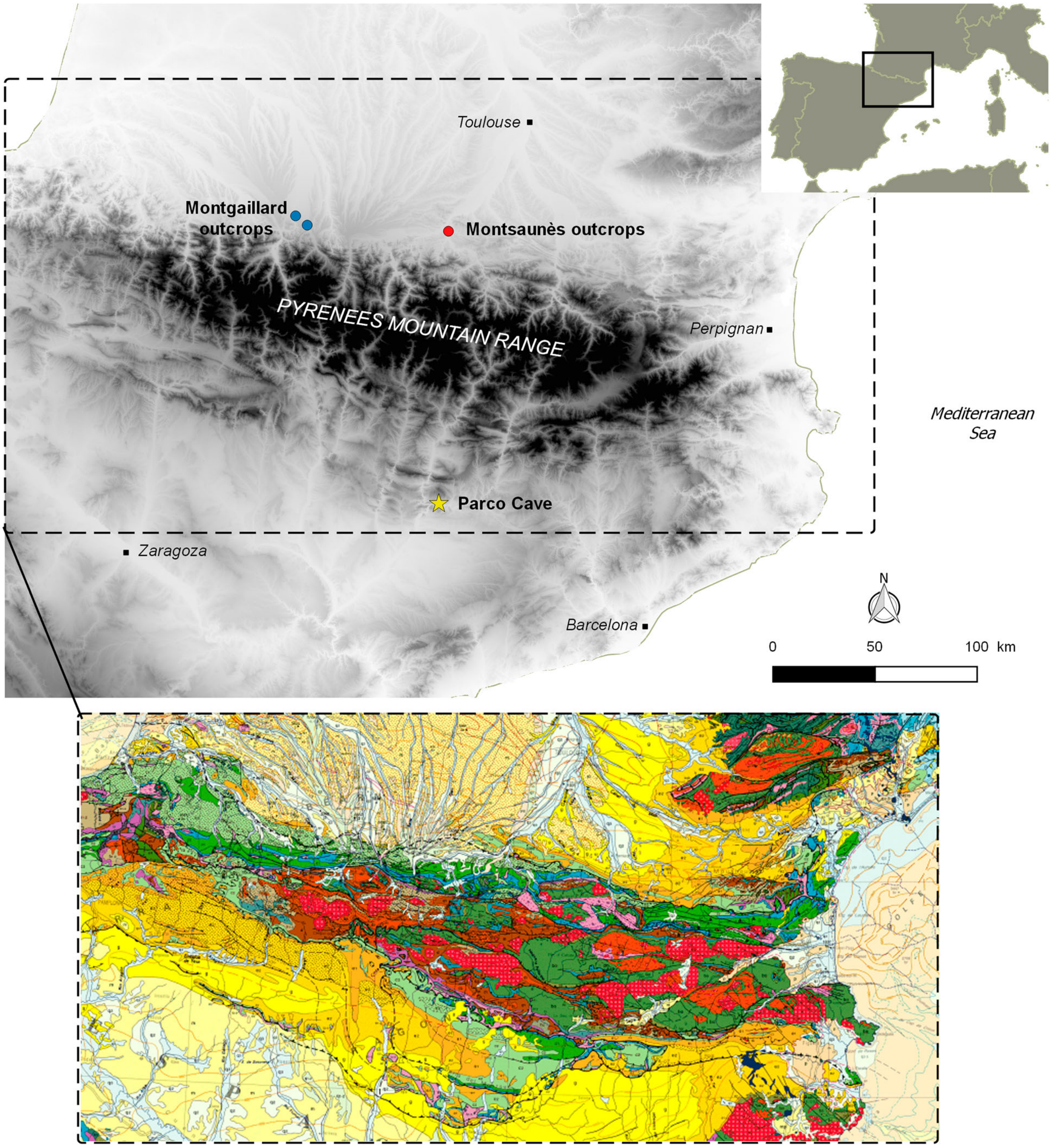

Figure 2. Location of the archaeological site of Parco Cave and the outcrops from Montgaillard and Montsaunès formations where sampling was done.

Cherts possess identical features in primary and secondary outcrops. Cortex are regulars, with variable thicknesses and colours from greys to browns with a high variability intrabloc. The micropalaeontological content is composed by sponge spicules and some small benthic foraminifera (particularly globotruncanids). In thin sections, a criptoquartz mosaic as main texture is shown. In few average length-fast chalcedony is identified. Siliceous sponge spicules are also observed. Carbonated elements are constituted by micrite and some skeletal bioclastic elements being in process to be silicified. Metal oxides are abundant and detrital components in the shape of detrital quartz are observed (Sánchez de la Torre 2015).

The Montsaunès-Ausseing cherts are inserted in the Nankin limestones dating from the Middle Maastrichtian and outcropping in the Ausseing Mountain and the ancient quarry of Montsaunès (Haute-Garonne, France) (Séronie-Vivien, Séronie-Vivien, and Foucher 2006). The micropaleontological content of these limestones is rich and an association of classical benthic Maastrichtian foraminifera as *Orbitoides apiculate*, *Lepidorbitoides socialis*, *Omphalocyclus macroporus*, *Siderolites calcitrapoides* and *Siderolites denticulatus* (Bilotte and Andreu 2006) has been identified. Cherts possess beige to brown colours with a micropaleontological content represented by sponge spicules and small foraminifera (globotruncanids and rotalids). Only in a few samples Maastrichtian benthic foraminifera as *Siderolites* have been detected. In thin sections, a criptoquartz mosaic is the main observed texture, and in a few average length-fast chalcedony is identified. Siliceous sponge spicules are also recognised. Carbonate components are represented by micrite and bioclastic elements in process to be completely silicified. In some cases, Maastrichtian benthic foraminifera that could be classified as

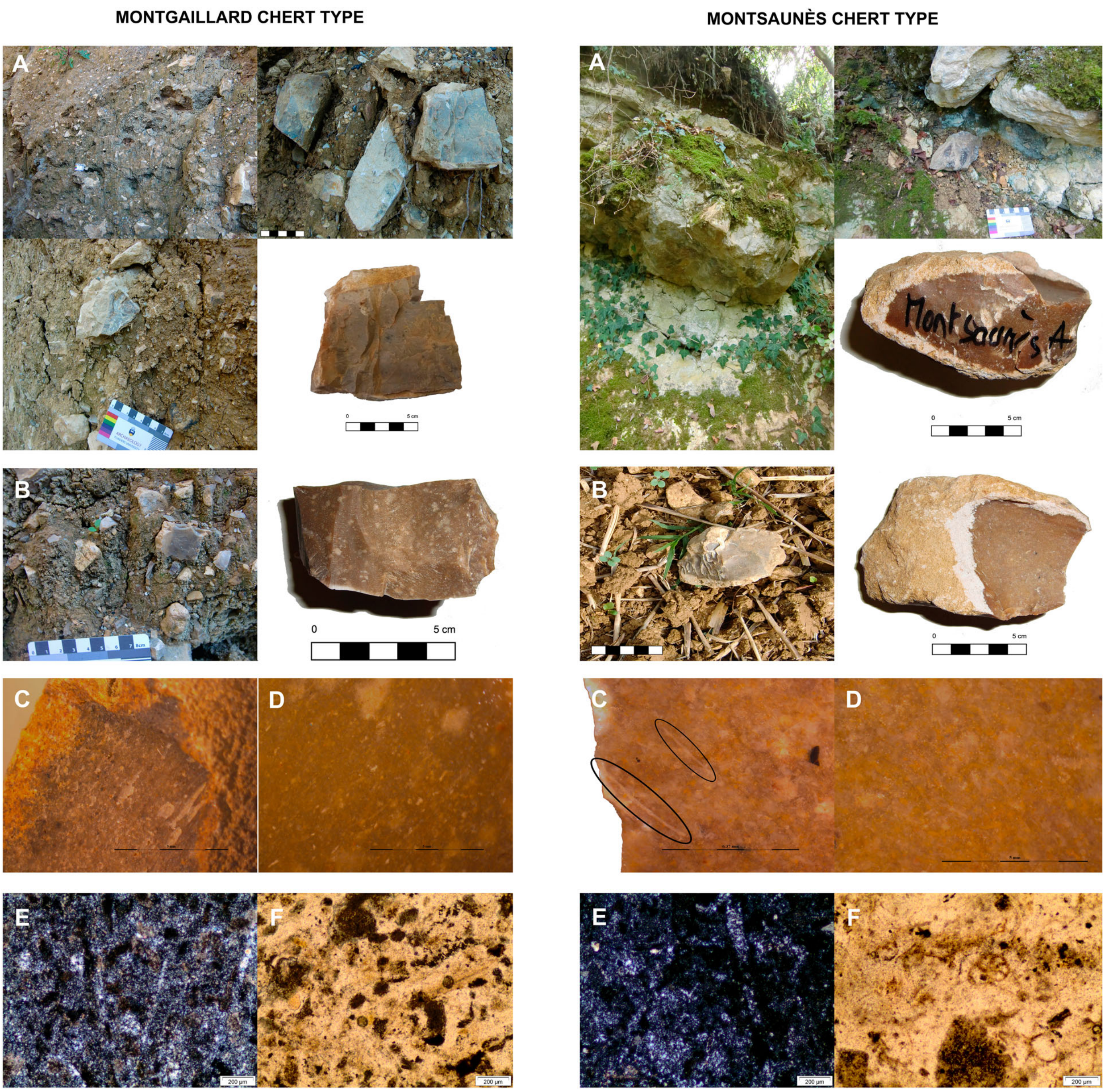

Figure 3. Montgaillard and Montsaunès cherts. Primary outcrop detail (A), secondary outcrop detail (B), macroscopic texture (C & D) and petrographic texture with crossed polarised light (E) and plane polarised light (F).

Lepidorbitoides are identified at the petrographic microscope. Metal oxides are frequent as well as detrital quartz components (Sánchez de la Torre 2015).

In short, both formations possess chert with similar characteristics (figure 3), being the presence of Maastrichtian benthic foraminifera only detected in some Montsaunès cherts. Nevertheless, as these micropaleontological elements are not regularly present, given the absence of these foraminifera in cherts, the differentiation between formations becomes impossible.

The scientific interest of this study is to geochemically characterise both Montgaillard flysch cherts and Montsaunès cherts with the aim to establish differences between them at the elemental chemical level. It is the aim of this work to prove the hypothesis of distal provenance of some lithic artefacts of Parco Cave from the north slope of the Pyrenees and to discriminate the two possible similar sources of Montgaillard and Montsaunès by geochemical methods.

Material and methods

In order to collect new chert samples and to characterize chert outcrops, some field surveys were systematically done. Samples were collected trying to obtain a major representation of the outcrop internal variability and after macroscopic analysis, 20 samples were selected for geochemical analysis after determining the existent macroscopic variability.

For the geochemical analysis, 40 geological cherts (20 from Montgaillard type and 20 from Montsaunès type) were analysed. With the aim to improve analysis time and avoid surface alterations, geological samples were prepared in squares of 5×5 mm without cortex surfaces. Concerning archaeological samples, after the macroscopic analysis of all chert tools from the Magdalenian levels of Parco Cave recovered until 2012, a selection of 51 chert artefacts was done. These samples were chosen because they had not developed post-depositional alteration processes as patines or CaCo concretions. 35 artefacts were analysed by ED-XRF and 16 by LA-ICP-MS. For LA-ICP-MS some chamber restrictions existed, so the smallest pieces were reserved for these analyses. For ED-XRF, as a collimator of 3×3 mm was selected and no chamber restrictions existed, we could analyse a larger number of samples.

To analyse major and minor elements, ED-XRF (energy-dispersive X-ray Fluorescence) was applied. Analyses were developed at the Research Centre for Applied Physics in Archaeology, IRAMAT, Bordeaux, France. 9 elements were quantified (Na, Mg, Al, Si, P, K, Ca, Ti, Fe) using an X-ray fluorescence spectrometer SEIKO SEA 6000VX (Orange et al. 2016). Fundamental parameters corrected by the granodiorite GSP2 from the U.S. Geological Survey (USGS) international standard (Wilson, 1998) were used. A 3×3 mm collimator was used and analysis time was set to 400 seconds for each measurement condition (3 conditions with air or He environment and Cr or Pb filter were established). To check machine calibration and accuracy JCh-1 chert standard from the Geological Survey of Japan (GSJ) international standard was used (Imai et al. 1996). To prove and validate the receipt and to check machine accuracy a measurement with the JCh-1 chert standard was established. Two powder tablets were analysed in several points in routine mode. Results show that the average obtained for the 17 analysed points were close to the desired value, being the standard deviation always lower than 0,08w% and validating the accuracy of the receipt (table 2).

To analyse trace elements, LA-ICP-MS (Laser ablation inductively coupled plasma mass spectrometry) at the Ernest-Babelon laboratory, IRAMAT, Orleans, France was used. Elements were quantified using a Thermo Fisher Scientific Element XR mass spectrometer associated with a Resonetics RESOlution M50e ablation device. This spectrometer offers the advantage of being equipped with a dual mode

Table 2. ED-XRF analytical data (in %w) for the JCh-1 test analysis to check machine accuracy.

		Na_2O	MgO	Al_2O3	SiO_2	P_2O_5	K_2O	CaO	TiO_2	Fe_2O_3
1	JCh-1 (A1)	<LD	0.05	0.92	98.61	<LD	0.14	0.03	0.03	0.23
2	JCh-1 (A1)	<LD	0.07	0.97	98.53	<LD	0.15	0.03	0.02	0.24
3	JCh-1 (A1)	<LD	0.04	0.56	99.14	<LD	0.09	0.02	0.01	0.14
4	JCh-1 (A2)	<LD	0.04	0.56	99.14	<LD	0.09	0.01	0.02	0.14
5	JCh-1 (A2)	<LD	0.04	0.57	99.13	<LD	0.09	0.02	0.02	0.14
6	JCh-1 (A2)	<LD	0.03	0.56	99.15	<LD	0.09	0.02	0.01	0.14
7	JCh-1 (A3)	<LD	0.09	1.37	97.84	<LD	0.23	0.05	0.03	0.39
8	JCh-1 (A3)	<LD	0.05	0.55	99.13	<LD	0.09	0.01	0.02	0.14
9	JCh-1 (A3)	<LD	0.04	0.57	99.11	<LD	0.09	0.01	0.02	0.15
10	JCh-1 (B1)	<LD	0.06	0.97	98.49	<LD	0.16	0.03	0.03	0.26
11	JCh-1 (B1)	<LD	0.04	0.55	99.14	<LD	0.09	0.01	0.02	0.15
12	JCh-1 (B1)	<LD	0.59	0.99	97.97	<LD	0.16	0.02	0.01	0.26
13	JCh-1 (B2)	<LD	0.08	1.01	98.44	<LD	0.15	0.03	0.03	0.26
14	JCh-1 (B2)	<LD	0.05	0.56	99.14	<LD	0.08	0.02	0.01	0.14
15	JCh-1 (B2)	<LD	0.05	0.57	99.11	<LD	0.09	0.01	0.02	0.14
16	JCh-1 (B2)	<LD	0.06	0.56	99.13	<LD	0.09	0.02	0.01	0.14
17	JCh-1 (A1)	<LD	0.08	1.02	98.42	<LD	0.16	0.03	0.04	0.25
	Average	<LD	0.05	0.75	98.86	<LD	0.12	0.02	0.03	0.19
	Std. Dev.	<LD	0.02	0.01	0.74	<LD	0.07	0.01	0.00	0.12
	Exp. value	0.03	0.08	0.73	97.81	0.02	0.22	0.04	0.03	0.36

(counting and analog modes) secondary electron multiplier (SEM) with a linear dynamic range of over nine orders of magnitude, associated with a single Faraday collector which allows an increase of the linear dynamic range by an additional three orders of magnitude. This feature is particularly important for laser ablation analysis of lithic samples, as it is possible to analyse major, minor, and trace elements in a single run regardless of their concentrations and their isotopic abundance. The ablation device is an excimer laser (ArF, 193 nm), which was operated at 6mJ and 10hz. A dual gas system with helium (0.6 l/min) released at the base of the chamber, and argon at the head of the chamber (1.1 l/min) carried the ablated material to the plasma torch. Ablation time was set to 40 seconds: 10s pre-ablation to let the ablated material reach the spectrometer and 30s collection time. Laser spot size was set to 100 µm and line mode acquisition was chosen to enhance sensitivity. Background measurements were run every 10 to 20 samples. Fresh fractures were analysed on geological samples to reduce potential contamination. Priority was given to characterizing the largest number of samples for each site, thus, only one ablation line was carried out per sample. However, if during analysis element spikes due to the presence of inclusions or heterogeneities were observed, results were discarded and a new ablation site selected.

Calibration was performed using standards reference glass NIST610 which was run periodically (every 10 to 20 samples) to correct for drift. NIST 610 was used to calculate the response coefficient (k) of each element (Gratuze 1999, 2014) and the measured values of each element were normalised against 29Si, the internal standard, to produce a final percentage. Glass Standard NIST612 was analysed independently of calibration to provide comparative data. After doing some tests with 56 elements, a total of 23 were measured (Mg, Al, Si, K, Ti, V, Cr, As, Rb, Sr, Y, Zr, Nb, Cs, Ba, La, Ce, Pr, Nd, Sm, W, Bi, Th).

Results

Field survey results: chert outcrops characterization

Several field survey works took place with the aim to better redefine the characteristics of the primary and secondary outcrops and to observe if differences where observed between them at the textural, micropalaeontologic and petrographic scale.

During these works several secondary outcrops of Montgaillard flysch type cherts were detected nearby Montgaillard town, where primary outcrops where already noticed. Secondary deposits were placed in Miocene sand clays and ancient knapping evidences were detected. The observation of new secondary samples and the comparison with samples from primary outcrops showed that not noticeable changes were observed between samples concerning textural, micropalaeontological and petrographic characterizations. Thus, the micropalaeontological content was composed by sponge spicules and small benthic foraminifera and in thin sections a criptoquartz mosaic was the main observed texture (table 7).

The Montsaunès cherts were collected nearby the ancient quarry of Montsaunès, first published by Barragué and colleagues (2001), where a primary deposit was detected. An important tectonism was noticed in chert nodules outcropping in the primary deposit. More homogeneous and high-quality chert nodules were found in the field located some meters below the ancient quarry. Remains of ancient flint knapping were also observed in the field. The macroscopic and petrographic analyses of cherts collected in primary and these secondary sources showed similar characteristics concerning micropalaeontological and petrographic aspects. Therefore, sponge spicules and small foraminifera was the main micropalaeontological content and a criptoquartz mosaic the main silica texture (table 7).

Geochemical characterisation of geological sources

Results obtained by Energy-Dispersive X-ray Fluorescence show that no clear differences appear between Montgaillard flysch cherts and Montsaunès cherts concerning major and minor elements (table 3). SiO_2 rate represents at least 98w% of the samples elemental chemical composition. Minor elements are represented by Al_2O_3, CaO, Fe_2O_3 and K_2O. MgO, Na_2O, P_2O and TiO_2, despite being analysed, have not been contemplated, as results are often below limits of detection and, when present, they do not exceed the 0.03w%. Thus, K_2O, Al_2O_3 and Fe_2O_3, which are the minor components represented in the analysed cherts, possess values that are always below 1w% of the total elemental composition rate, and only in a sample CaO amount is beyond 1w% (MONTG-15 CaO value: 1.43). Concentrations of K_2O, Al_2O and Fe_2O_3 could be explained by some mineral inclusions. CaO rates, which show in both chert types variations between the sample, could be explained by the presence of carbonate inclusions, which are relatively common as observed by petrographic observations. Higher Fe_2O_3 concentration rates are usually associated to Montgaillard cherts. As presented in Table 3, the average of both chert types concerning the elements analysed by ED-XRF is quite similar, presenting only CaO values some differences (Table 3).

LA-ICP-MS analyses have shown several differences between Montgaillard flysch cherts and Montsaunès cherts considering some trace elements (table 4). This

Table 3. ED-XRF analytical data (in %w) for the Montgaillard flysch cherts and the Montsaunès cherts.

	Al_2O_3	SiO_2	K_2O	CaO	Fe_2O_3		Al_2O_3	SiO_2	K_2O	CaO	Fe_2O_3
MONTG-01	0.33	99.49	0.02	0.13	0.04	**MONTS-01**	0.36	99.35	0.02	0.21	0.04
MONTG-02	0.34	99.48	0.02	0.12	0.04	**MONTS-02**	0.36	98.96	0.03	0.6	0.05
MONTG-03	0.37	99.26	0.02	0.12	0.07	**MONTS-03**	0.34	99.56	0.02	0.04	0.03
MONTG-04	0.33	99.2	0.01	0.44	0.02	**MONTS-04**	0.35	99.47	0.01	0.15	0.02
MONTG-05	0.36	99.47	0.01	0.12	0.03	**MONTS-05**	0.36	99.5	0.02	0.09	0.03
MONTG-06	0.35	99.53	0.02	0.07	0.03	**MONTS-06**	0.41	99.47	0.04	0.06	0.01
MONTG-07	0.34	99.5	0.01	0.12	0.03	**MONTS-07**	0.36	99.5	0.03	0.06	0.05
MONTG-08	0.36	99.49	0.02	0.08	0.04	**MONTS-08**	0.33	99.58	0.02	0.05	0.01
MONTG-09	0.35	99.57	0.01	0.03	0.04	**MONTS-09**	0.34	99.43	0.02	0.13	0.06
MONTG-10	0.37	99.55	0.01	0.04	0.02	**MONTS-10**	0.34	99.46	0.02	0.17	0.01
MONTG-11	0.34	99.59	0.02	0.03	0.03	**MONTS-11**	0.33	99.56	0.01	0.09	0.01
MONTG-12	0.36	99.43	0.02	0.15	0.03	**MONTS-12**	0.32	99.58	0.02	0.07	0.01
MONTG-13	0.36	99.47	0.01	0.13	0.02	**MONTS-13**	0.34	99.36	0.02	0.26	0.01
MONTG-14	0.38	98.96	0.02	0.58	0.06	**MONTS-14**	0.34	99.58	0.01	0.05	0.01
MONTG-14	0.37	99.47	0.02	0.08	0.06	**MONTS-15**	0.32	99.6	0.01	0.06	0.01
MONTG-14	0.36	99.14	0.02	0.43	0.05	**MONTS-16**	0.34	99.52	0.02	0.1	0.02
MONTG-14	0.35	99.14	0.01	0.43	0.06	**MONTS-16**	0.35	99.45	0.02	0.14	0.03
MONTG-15	0.35	98.16	0.02	1.43	0.04	**MONTS-16**	0.34	99.53	0.02	0.09	0.02
MONTG-16	0.36	98.6	0.02	0.96	0.05	**MONTS-16**	0.34	99.44	0.02	0.17	0.02
MONTG-17	0.36	99.51	0.02	0.08	0.03	**MONTS-17**	0.31	99.61	0.01	0.06	0.01
MONTG-18	0.34	98.83	0.01	0.79	0.02	**MONTS-18**	0.37	99.38	0.02	0.16	0.05
MONTG-19	0.38	99.49	0.01	0.08	0.04	**MONTS-19**	0.38	99.5	0.02	0.03	0.06
MONTG-20	0.34	99.43	0.02	0.18	0.03	**MONTS-20**	0.34	99.48	0.02	0.1	0.05
AVERAGE	0.35	99.29	0.02	0.29	0.04	*Average*	0.35	99.47	0.02	0.13	0.03
STD. DEV.	0.01	0.36	0.00	0.35	0.01	*Std. dev.*	0.02	0.13	0.00	0.12	0.02

discrimination is essentially based on Sr, Th, Cr and V contents. The non-parametric density plot concerning Log (Sr/Th) and Log (Cr/Th) shows the existence of two main discrete geochemical types, that highly coincide with the two analysed formations (figure 4). However, a potential outlier is detected (MONTS-16), not fitting with the Montsaunès maximum density. This sample, which is different at the elemental level, could also be classified as an outlier regarding the micropalaeontological content. Thus, the previous textural and micropaleontological analysis showed a specific bioclastic content composed by possible foraminifera (Siderolites) in MONTS-16 sample (figure 6). This kind of foraminifera, which can be present in Montsaunès cherts regarding the geological description of the Nankin formation content (BRGM 1971) is not commonly represented in Montsaunès recovered chert samples, and has only been detected in two analysed samples, one of these being MONTS-16 (figure 7).

Geochemical characterisation of Parco Cave artefacts

The analysis of 51 artefacts from Parco Cave by ED-XRF shows that major and minor elements are represented with similar w% than in Montgaillard and Montsaunès chert samples. Results presented in Table 5 show that most of the samples are closer to the Montgaillard and Montsaunès averages concerning the elements analysed. Nevertheless, differences appear while observing Al_2O_3 and CaO results, highly varying the values obtained depending on the sample. This huge variation of Al_2O_3 and CaO rates could be

Table 4. LA-ICP-MS analytical data (in ppm) for the Montgaillard flysch cherts and the Montsaunès cherts.

	V	*Cr*	*Sr*	*Th*		*V*	*Cr*	*Sr*	*Th*
MONTG-01	2.85	6.79	3.33	0.19	**MONTS-01**	8.88	20.1	10.4	0.91
MONTG-02	4.03	17.7	10.6	0.47	**MONTS-02**	17.3	21.7	5.53	1.40
MONTG-03	3.20	10.4	9.62	0.51	**MONTS-03**	28.5	31.4	6.96	1.54
MONTG-04	0.93	8.68	12.2	0.19	**MONTS-04**	16.0	28.8	11.7	2.15
MONTG-05	1.70	11.9	18.5	0.61	**MONTS-05**	24.2	23.0	9.40	0.79
MONTG-06	4.83	18.1	15.2	0.35	**MONTS-06**	21.7	29.0	12.4	0.99
MONTG-07	37.0	24.6	54.1	1.49	**MONTS-07**	4.09	10.9	5.11	0.36
MONTG-08	14.9	16.3	30.9	1.04	**MONTS-08**	0.25	5.15	1.99	0.29
MONTG-09	2.30	11.6	8.88	0.19	**MONTS-09**	7.50	13.3	4.67	0.95
MONTG-10	1.43	12.6	13.2	0.12	**MONTS-10**	4.71	10.6	4.22	0.48
MONTG-11	1.27	9.28	5.85	0.28	**MONTS-11**	4.37	6.89	5.92	0.38
MONTG-12	2.38	11.5	13.3	0.28	**MONTS-12**	9.01	7.02	7.03	0.36
MONTG-13	8.66	8.80	20.2	0.87	**MONTS-13**	6.35	12.8	6.65	0.64
MONTG-14	1.19	5.39	79.7	0.23	**MONTS-14**	4.30	9.64	6.29	0.42
MONTG-15	11.3	21.7	285	0.84	**MONTS-15**	12.2	16.2	4.58	1.20
MONTG-16	1.76	8.11	15.4	0.22	**MONTS-16**	9.50	25.7	45.0	1.11
MONTG-17	2.90	8.19	10.6	0.28	**MONTS-17**	14.7	22.8	5.86	0.57
MONTG-18	1.91	11.0	16.4	0.24	**MONTS-18**	28.3	41.8	11.0	1.73
MONTG-19	12.9	50.0	20.7	0.98	**MONTS-19**	8.07	26.1	7.24	0.78
MONTG-20	8.15	21.2	10.2	0.50	**MONTS-20**	7.63	14.2	3.01	0.94
Average	6.28	14.7	32.7	0.49	*Average*	11.9	18.9	8.75	0.90
Std. dev.	8.37	9.9	62.0	0.37	*Std. dev.*	8.3	9.8	9.00	0.51

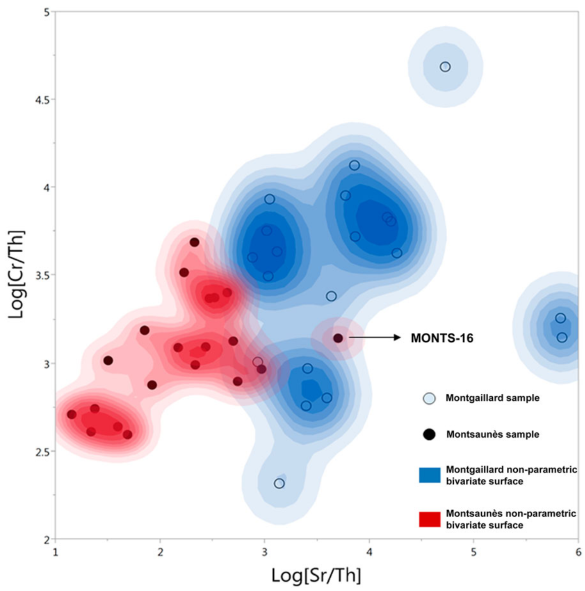

Figure 4. Non-parametric density plot concerning Log (Sr/Th) and (Cr/Th) for Montgaillard and Montsaunès samples.

Table 6. LA-ICP-MS analytical data (in ppm) for Parco Cave samples (Montgaillard and Montsaunès averages of each element are presented).

	V	*CR*	*SR*	*TH*
MONTG (AV)	*6,28*	*14,7*	*32,7*	*0,49*
MONTG (STD)	*8,37*	*9,88*	*62,0*	*0,37*
MONTS (AV)	*11,9*	*18,9*	*8,75*	*0,90*
MONTS (STD)	8,32	9,76	9,00	0,51
PARCO-30	13,0	16,9	21,8	0,52
PARCO-31	9,17	13,0	15,0	0,33
PARCO-32	28,2	23,8	16,8	0,54
PARCO-33	6,59	6,97	10,9	0,22
PARCO-34	18,6	14,3	14,8	0,42
PARCO-35	5,61	7,21	15,4	0,34
PARCO-36	15,6	14,5	19,0	1,26
PARCO-37	26,5	24,6	16,9	0,77
PARCO-38	38,7	16,8	45,5	0,86
PARCO-39	15,9	12,1	14,5	0,45
PARCO-40	16,4	23,2	31,8	0,77
PARCO-41	22,1	27,6	19,7	0,60
PARCO-42	23,1	24,8	18,5	0,84
PARCO-43	4,88	8,79	12,4	0,20
PARCO-44	20,6	18,8	30,2	0,65
PARCO-45	2,69	7,43	9,55	0,28
NIST612 (AV)	*39,5*	*37,8*	*77,3*	*36,2*
NIST612 (STD)	*0,5*	*1,1*	*0,9*	*0,7*

explained by post-depositional features that may have alterated these elements rates.

LA-ICP-MS results (table 6), however, associate all analysed Parco Cave samples to the Montgaillard dispersion area. Thus, the plot concerning Parco Cave artefacts regarding Log (Sr/Th) and Log (Cr/Th) values indicates that all samples seem to fit with the Montgaillard non-parametric surface (figure 5). During LA-ICP-MS analyses only a selection of the archaeological samples was analysed, due to the micro-destruction of the sample regarding the laser ablation. So, between both samples previously ascribed to Montsaunès cherts at the ED-XRF analyses (PARCO-1 and PARCO-37), only one of them was also analysed by LA-ICP-MS (PARCO-37) and results showed that this sample fits in the Montgaillard dispersion area regarding Cr, Sr and Th values.

Nevertheless, previous micropalaeontological analyses showed that four Parco Cave artefacts presented some foraminifera at the moment only detected in some Montsaunès chert samples (Siderolites). However, the non-parametric plot obtained after LA-ICP-MS analyses directly associate the fourth concerned archaeological samples to the Montgaillard dispersion area. These artefacts are expressed with a * symbol in figure 5. Two of them (PARCO-44 and PARCO-45)

Table 5. ED-XRF analytical data (in %w) for Parco Cave samples (Montgaillard and Montsaunès averages of each element are presented).

	Al_2O_3	SiO_2	K_2O	CaO	Fe_2O_3		Al_2O_3	SiO_2	K_2O	CaO	Fe_2O_3
MONTG (AV)	***0.35***	***99.29***	***0.02***	***0.29***	***0.04***	**PARCO-21**	0.46	99.26	0.04	0.21	0.03
MONTS (AV)	***0.35***	***99.47***	***0.02***	***0.13***	***0.03***	**PARCO-22**	0.47	99.24	0.04	0.21	0.04
PARCO-1	0.5	99.31	0.05	0.12	0.01	**PARCO-23**	0.88	98.27	0.1	0.55	0.09
PARCO-2	0.84	98.84	0.07	0.11	0.04	**PARCO-24**	<LD	99.78	0.05	0.07	0.02
PARCO-3	0.77	98.42	0.06	0.12	0.08	**PARCO-25**	0.44	99.31	0.03	0.13	0.02
PARCO-4	0.51	99.33	0.04	0.09	0.02	**PARCO-27**	0.69	98.97	0.06	0.14	0.04
PARCO-4	0.55	99.31	0.05	0.07	0.02	**PARCO-28**	0.93	98.61	0.08	0.13	0.13
PARCO-4	<LD	99.39	0.05	0.09	0.02	**PARCO-29**	0.88	97.55	0.1	0.7	0.09
PARCO-4	<LD	99.39	0.05	0.09	0.02	**PARCO-31**	1.13	97.89	0.09	0.24	0.07
PARCO-5	0.65	98.68	0.05	0.46	0.06	**PARCO-35**	0.8	98.34	0.07	0.27	0.06
PARCO-6	0.52	99.25	0.04	0.11	0.06	**PARCO-36**	0.77	98.27	0.1	0.67	0.1
PARCO-7	0.82	98.81	0.07	0.12	0.07	**PARCO-37**	0.63	99.01	0.06	0.27	0.01
PARCO-8	0.46	99.43	0.03	0.04	0.03	**PARCO-38**	0.54	99.21	0.04	0.13	0.08
PARCO-9	0.52	99.24	0.04	0.15	0.02	**PARCO-39**	0.83	98.71	0.08	0.21	0.06
PARCO-10	0.62	96.75	0.04	2.03	0.05	**PARCO-41**	0.71	98.97	0.05	0.13	0.05
PARCO-11	0.44	99.41	0.03	0.05	0.06	**PARCO-42**	1.19	97.87	0.08	0.21	0.06
PARCO-12	0.68	98.01	0.05	1.1	0.05	**PARCO-43**	0.57	99.08	0.06	0.21	0.05
PARCO-13	0.89	98.29	0.07	0.08	0.04	**PARCO-44**	<LD	99.38	0.05	0.46	0.05
PARCO-14	0.53	99.19	0.05	0.17	0.05	**PARCO-45**	0.52	99.16	0.04	0.22	0.05
PARCO-15	0.58	99.11	0.03	0.13	0.04	**PARCO-46**	0.64	98.79	0.06	0.38	0.04
PARCO-16	0.53	99.11	0.04	0.26	0.05	**PARCO-47**	1.08	97.52	0.13	0.64	0.08
PARCO-17	1.54	96.44	0.17	1.02	0.08	**PARCO-48**	0.57	99.12	0.04	0.17	0.08
PARCO-18	0.5	99.37	0.04	0.05	0.03	**PARCO-49**	1.45	96.92	0.14	0.84	0.09
PARCO-19	0.54	99.22	0.04	0.06	0.04	**PARCO-50**	0.68	99.18	0.04	0.08	0.02
PARCO-20	0.57	99.09	0.03	0.17	0.03	**PARCO-51**	0.67	99.14	0.06	0.1	0.02

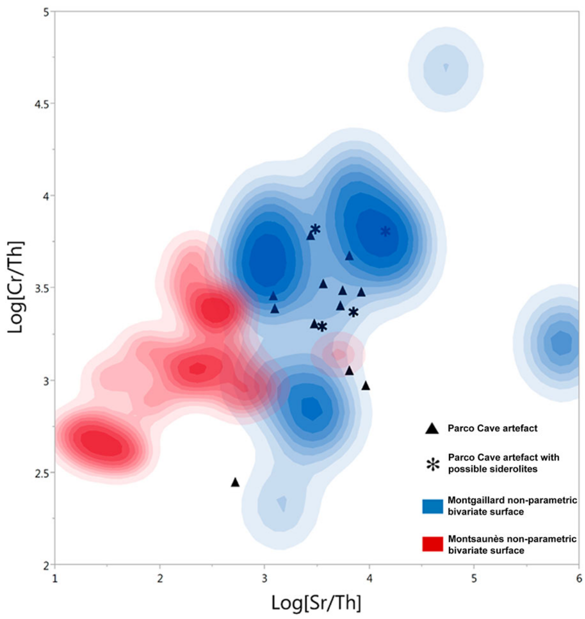

Figure 5. Non-parametric density plot concerning Log (Sr/Th) and (Cr/Th) for Montgaillard and Montsaunès samples.

are closely to the Montsaunès outlier sample (MONTS-16) that also presented a specific micropalaeontological content.

Discussion

ED-XRF and LA-ICP-MS analyses have given valuable data for better defining the Montgaillard flysch cherts and the Montsaunès cherts, which constitute an interesting lithological tracer when studying lithic raw material procurement in the Pyrenees region (table 7). Despite not having obtained encouraging results concerning major and minor elements obtained by ED-XRF analyses, LA-ICP-MS results have shown that some differences exist between both chert types regarding the trace elements content. The non-parametric plots presented above concerning Cr, Sr and Th data demonstrate the existence of two discrete geochemical groups.

However, when analysing archaeological samples from Parco Cave, some divergences are observed. Thus, the previous micropalaeontological characterisation does not always fit well with the geochemical results, as some Parco Cave samples which had been previously ascribed to Montsaunès chert type concerning the presence of probable Siderolites are ascribed to Montgaillard group within the geochemical characterisation. This discrepancy could be explained by several reasons: post-depositional alterations may have affected archaeological remains, modifying the chemical signal of surfaces analysed and more specifically affecting major and minor w% rates; or other Montgaillard flysch chert or Montsaunès outcrop could exist, possessing the found micropalaeontological content (Siderolites) and having a geo-chemical signal similar to Montgaillard analysed samples.

Nevertheless, geo-chemical data obtained within this study has allowed us to ascribe, if not all, at least the majority of samples from Parco Cave, to the Montgaillard flysch chert group. Concerning the archaeological aspect of the data obtained, results are surprising and promising as it is for the first time geo-chemically supported that lithic raw materials exchanges existed between both Pyrenean slopes at the end of the Upper Palaeolithic. Montgaillard flysch chert outcrops are located at more than 130 km NW of Parco Cave, being located between the outcrops and the site the central part of the Pyrenan mountain range, with the highest attitudes (Aneto peak 3404 m asl).

Concerning the presence of the Montgaillard flysch cherts type in the Magdalenian record of Parco Cave, a total of 84 chert tools have been ascribed to the Montgaillard flysch chert type. This group represents the 5.07% of the totality of chert artefacts recovered in all the Magdalenian unit until 2013. Concerning the typological analysis, abrupt-retouches are preferred (43.53%) over simple-retouched tools (22.35%). Cores morphologies (mostly pyramidal and prismatic) indicate blade and bladelet preference within supports

Table 7. Main characteristics of the analysed cherts (Montgaillard, Montsaunès and the archaeological samples from Parco Cave).

Characteristics	MONTGAILLARD	MONTSAUNÈS	ARCHAEOLOGICAL
Textural	Regular cortex	Regular cortex	Regular cortex
	Grey to brown colours	Beige to brown colours	Orange to brownish colours
	Metal oxides	Metal oxides	Metal oxides
	Detrital quartz	Detrital quartz	Detrital quartz
Micropalaeontological	Sponge spicules	Sponge spicules	Sponge spicules
	Small benthic foraminifera (globotruncanids)	Small foraminifera (globotruncanids and rotalids)	Small foraminifera
		Siderolites	Siderolites?
Petrographic	Criptoquartz mosaic (MT)	Criptoquartz mosaic (MT)	Criptoquartz mosaic (MT)
	Length-fast chalcedony	Length-fast chalcedony	Length-fast chalcedony
Geochemical (Major & minor)	SiO_2 (99.29%)	SiO_2 (99.47%)	SiO_2 (98.77%)
	Al_2O_3 (0.35%w)	Al_2O_3 (0.35%w)	Al_2O_3 (0.67%w)
	CaO (0.29%w)	CaO (0.13%w)	CaO (0.19%w)
	Fe_2O_3 (0.04%w)	Fe_2O_3 (0.03%w)	Fe_2O_3 (0.04%w)
	K_2O (0.02%w)	K_2O (0.02%w)	K_2O (0.05%w)
Geochemical (Trace)	V (6.28 ppm)	V (11.9 ppm)	V (13.6 ppm)
	Cr (14.7 ppm)	Cr (18.9 ppm)	Cr (14.8 ppm)
	Sr (32.7 ppm)	Sr (8.75 ppm)	Sr (18.0 ppm)
	Th (0.49 ppm)	Th (0.9 ppm)	Th (0.5 ppm)

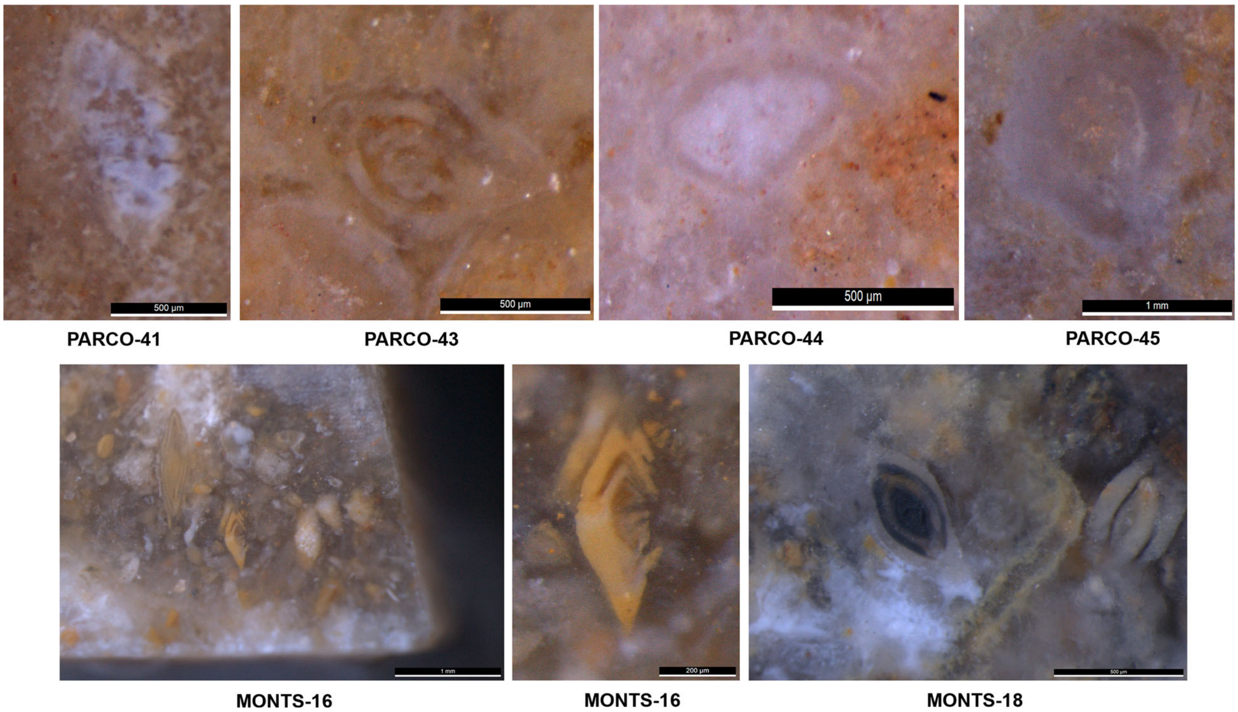

Figure 6. Non-common micropaleontological content detected in some Montsaunès and Parco Cave samples.

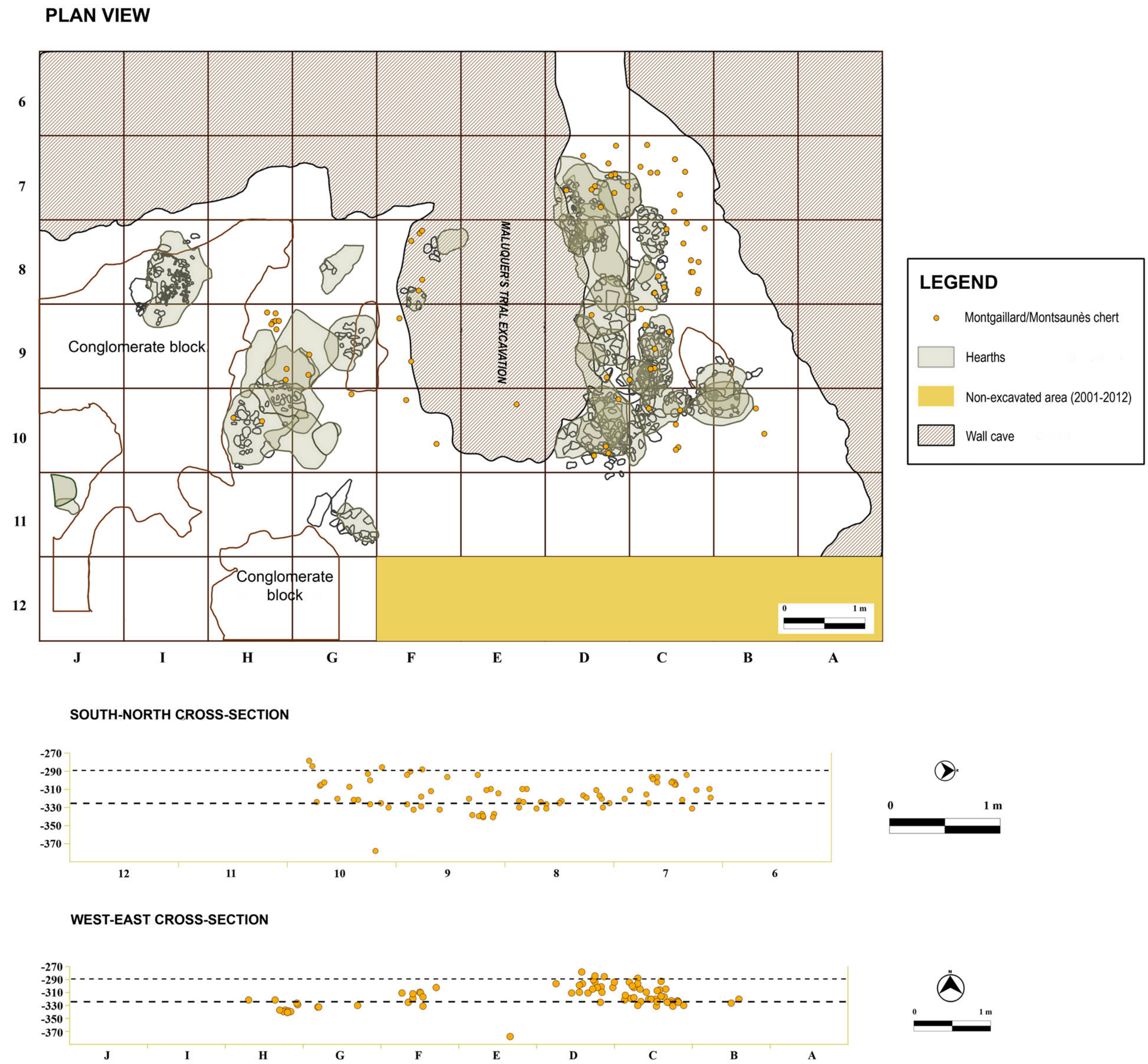

Figure 7. Parco Cave plan and cross-sections with the Montgaillard/Montsaunès chert type distribution.

confection. Non-retouched artefacts from this chert type represent the 2.2% of the set, demonstrating that despite being done the knapping process outside the cave, some works (*e.g.* repairing cutting edges) were completed at the site.

The dispersion analysis of the Montgaillard flysch cherts in the Magdalenian sequence of Parco Cave indicates that this chert type is represented in all the analysed sequence and in all the excavation area, not showing differences within the dispersion of the local and regional chert types (figure 7) (Sánchez de la Torre 2015). In summary, the reiterated presence of Montgaillard chert type in Parco Cave site are indicating frequent contacts between both Pyrenean slopes during the LGM period, showing that despite the still strict climatic conditions, the Pyrenees represented a homogeneous territory where human contacts were continuous.

Conclusions

Based on first geochemical analyses of Montgaillard flysch cherts and Montsaunès cherts, ED-XRF seems to be a limited technique for this archaeological question due to acquisition limitations. In this way, the high Si rate, always up than 98w% limits the detection of trace elements, which are always represented in a few average. However, when the archaeometric study is restricted to a small geographic area and a limited number of geological sources, LA-ICP-MS could be a useful technique to solve archaeological provenance questions thanks to the trace elements detection.

The promising results obtained concerning the archaeological value of the data have to be validated by redefining the existent variability of each analysed formation and delimit the non-parametric bivariate surface of Montgaillard flysch cherts and Montsaunès cherts. More LA-ICP-MS analyses will be considered for applying systematic analyses into geological samples and archaeological artefacts to solve this question.

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