Characterizations of the Siliceous Rocks at Stélida, an Early Prehistoric Lithic Quarry (Northwest Naxos, Greece) by Petrography and Geochemistry: A First step towards Chert Sourcing

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Abstract

This article presents the results of a combined petrographic and geochemical characterization study of raw materials from the early prehistoric chert source of Stélida, on Naxos (Cyclades, Greece). The project represents the first step in a larger provenience studies programme dedicated to documenting which communities exploited this source during the Lower Palaeolithic to Mesolithic ($\geq 250,000$ – 9,000 BP). Field- and lab-based studies conclude that the cherts originated by pervasive silicification of the upper part of a clastic sedimentary sequence by hydrothermal fluids moving along a detachment fault separating them from the underlying Naxos granodioritic intrusive. Quartz is the dominant mineral, while zircon, anatase, hematite and barite are accessories. Petrographic features that are considered characteristic of the Stélida raw materials (e.g. colour and lustre, massive microcrystalline texture, abundant cavities, quartz crystals projecting into cavities and thin quartz veinlets crosscutting bedding planes) are described. The cherts are strongly depleted in trace and Rare Earth Elements. The petrographic and geochemical study of any stone tool made of chert showing similar macro- and microtextures, as well as the same geochemical signature, indicates it could potentially originate from Stélida.

Keywords – Aegean, chert characterization, petrography, chemistry, siliceous toolstones, Naxos, Cyclades, Palaeolithic – Mesolithic

This paper is dedicated to the memory of John E. Dixon, a friend, mentor, and ground-breaking geo-archaeologist.

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1. Introduction: Insular lithic resources of the Mediterranean

Insular lithic resources – primarily for making chipped stone tools - played a significant role for prehistoric Mediterranean communities, particularly from the Neolithic when most of these islands were colonized (cf. Cherry, 1981; Farr, 2006). The best-known raw materials are the volcanic products of the region, particularly the obsidian sources on Sardinia, Pantellaria, Lipari, Melos, Antiparos, and Giali, for which there is an extensive literature detailing their geochemical characterisation and history of exploitation (e.g. Carter, 2009; Costa, 2007; Renfrew et al., 1965; Williams-Thorpe, 1995). Less well-known island resources whose raw materials tended to have more local spheres of circulation include flint/chert on Corsica (Chiari et al., 2000), Sardinia (Bressy et al., 2008), Sicily (Robb and Farr, 2005: 28), Crete (Brandl, 2010), and Cyprus (Manning et al., 2010).

While most of these insular sources' primary period of exploitation began with the advent of farming, there is some evidence for pre-Neolithic exploitation. The best-known example of this was the procurement of Melian obsidian by hunter-gatherers on the Greek mainland in the 11th millennium cal BC (Upper Palaeolithic), which at the time of discovery represented the earliest – indirect – evidence for seafaring in the northern hemisphere (Renfrew and Aspinall, 1990). The received wisdom at the time was that any ventures into the Mediterranean by hunter-gatherers were limited in scale and distance, representing seasonal fishing/foraging ventures, rather than island colonisation (Cherry, 1981). More recent work has produced more robust evidence for pre-Neolithic (Late Pleistocene-Early Holocene) insular settlement in the Mediterranean (Broodbank, 2013: 148-156), including Epi-Palaeolithic sites on Lemnos (Efstratiou et al., 2014), and Cyprus (Simmons, 1999), plus a few Mesolithic habitations in Corsica/Sardinia (a single island at the time [Costa, 2004]), and the Aegean islands (Sampson, 2014; Strasser et al., 2015). More radical claims have also been made for earlier Lower-Middle Palaeolithic activity in the Aegean (Runnels, 2014), though the evidential bases for these claims has until recently relied almost entirely on surface finds (though see Strasser et al., 2011).

Little attention has been paid to the particulars of the non-obsidian raw materials within the chipped stone assemblages of Palaeolithic and Mesolithic sites of the Aegean Basin, despite the fact that chert(s) and quartzes were the predominant conchoidally fracturing materials used by these hunter-gatherer populations (**Table 1**). The chert source of Stélida on Naxos (Cyclades [**Figures 1-2**]) offers direct evidence of long-term exploitation of chert, and raises the question of whether chert was a widely-available and locally-exploited resource in the pre-Neolithic Aegean Basin, or instead an exotic, valued, and traded material as obsidian would later come to be. In part this question is purely geologic: how abundant are cherts in the region? Unfortunately, given their limited appeal to economic geology and their relatively small extent, they have not been widely noted in geologic mapping efforts.

The question may also be addressed archaeologically, by examining the chert components of lithic assemblages at archaeological sites – but approaching the question from that perspective/direction necessitates the ability to discriminate between distinct geologic outcrops of chert. Here we explore the characterization of Stélida chert through petrographic and geochemical characterization, creating the basis for broader study of diversity within Stélida chert and between various Aegean cherts.

While sourcing studies are well-established in the Aegean with regard to obsidian (starting with Renfrew et al., 1965), there have been far fewer attempts to characterize other siliceous raw materials, despite the prevalent outcroppings of chert and other materials throughout the region (Bornovas and Rondoyanni, 1983; Creuzburg et al., 1977). This paper part-aims to

redress this archaeometric imbalance. Employing a data-set of 12 geo-referenced source samples, we identify both major and accessory minerals and macro- and micro-textural features. Analytically this involved macroscopic visual inspection, as well as inspection via a petrographic microscope and a Scanning Electron Microscope [SEM]. Major, trace, and Rare Earth Elements [REE] were then detailed by X-ray fluorescence spectrometry [XRF] and Inductively Coupled Plasma - Mass Spectrometry techniques [ICP-MS].

As detailed below, the exploitation of the Stélida chert source began in the Middle Pleistocene / Lower Palaeolithic, and continued – probably intermittently – until the Early Holocene / Mesolithic (Carter et al., 2014, 2016a). This paper briefly summarizes the history of archaeological investigation and the geology of the area, then presents a petrographic and geochemical characterization of the raw material. This study forms a component of the larger *Stélida Naxos Archaeological Project*, initiated by our team in 2013. The longer term aim of undertaking this characterization study is to be able to document the raw material's regional significance by tracking its distribution through space and time (the quantity of knapping debris at the site leads us to believe that the source had supra-Naxian significance).

2. Archaeology of Stélida

The chert source of Stélida comprises the majority of a hill rising 151 m above what today is the coast of north-west Naxos (**Figure 2.A**). While Stélida was the focus of a geological study in the 1960s (Roesler 1969), it was not until 1981 that the prehistoric exploitation of the raw materials was documented during an island-wide survey (Séfériadès 1983). The associated archaeology comprised a large quantity of stone tool manufacturing debris, the date of which remained far from clear due to a lack of comparable material from the region. Tentative claims were forwarded for Stélida being of Early Neolithic or Epi-Palaeolithic date (Séfériadès 1983: 72-73), though this went against the accepted models at the time, which suggested that the Cyclades were uninhabited until the Late Neolithic, i.e. the 5th millennium cal BC (Cherry, 1981). That said, the archipelago was known to have been visited by (Greek) mainland populations from the Upper Palaeolithic onwards (11th millennium cal BC), as attested indirectly by the recovery of Cycladic (Melian) obsidian in late Pleistocene cultural layers at the Franchthi Cave in the Argolid (Renfrew and Aspinall, 1990). Stélida remained something of a chronological enigma until archaeologists of the Greek Ministry of Culture proclaimed the site to have been exploited during the Mesolithic, Upper- and Middle Palaeolithic, based on the techno-typological characteristics of finds from a series of small-scale rescue excavations over the past 15 years (Legaki, 2012, 2014).

In 2013, we initiated the *Stélida Naxos Archaeological Project* to undertake a detailed geo-archaeological characterisation of the site (www.stelida.mcmaster.ca). The research was part-motivated by the fact that the archaeology is being lost at an alarming rate due to modern construction. In turn, recent claims for Middle Pleistocene – Early Holocene sites elsewhere in the Aegean islands provide Stélida with a broader evidential context for its Palaeolithic – Mesolithic activity (Runnels, 2014; Sampson, 2014). These data are reconfiguring our understanding of the Aegean Basin's early prehistory, and potentially challenging orthodoxies concerning early humans' maritime capabilities (for a counter-point see Leppard, 2014).

Over the first two seasons we undertook a pedestrian survey of the site, using a combination of transects and grids to systematically document approximately 40 hectares of the undeveloped areas of Stélida, and parts of the promontory to the south. Standardised recovery methods using transects and grids led to the collection of 17,910 surface artefacts. Artefacts were found widely distributed across Stélida (**Figure 3**), not only in those areas immediately surrounding the outcrops,

but also the flanks of the hill in widely varying densities. Aside from a handful of pottery sherds and hammerstones, the finds comprised flaked chert artefacts (**Figure 2.C-D**), of which a significant number had technological and typological traits associated with material from well-dated Lower – Upper Palaeolithic and Mesolithic sites in the region (Carter et al., 2014). We here provide a precis of the four main periods of activity documented at the chert source.

The Mesolithic period is represented by artefacts whose form and techniques of production are in keeping with excavated material from Early Holocene sites elsewhere in the southern Aegean (see Kaczanowska and Kozłowski 2014), including the sites of Maroulas (Kythnos), Kerame 1 (Ikaria), and Franchthi Cave (Argolid [**Figure 1**]). The material is microlithic (sub-2cm) and largely flake-based, percussion-knapped from multi-directional cores; there is also a minority bladelet component (**Figures 2C** and **4**). Retouched pieces include those with linear retouch, notches, denticulates, piercer/borers ('spines'), and end-scrapers; true geometrics are rare (Carter et al., 2016a).

The Upper Palaeolithic assemblage is comprised of percussion blade industries (**Figure 5**); the technical and morphological characteristics of both cores and end-products suggest that there are at least two phases represented within this period at Stélida. Distinctive carinated end-scrapers / bladelet cores whose products have a distinctive twisted profile are diagnostic features of the earlier Upper Palaeolithic on the Greek mainland, as for example at the Franchthi Cave (c. 39,000 – 36,000 BP) in the Argolid (Douka et al., 2011). There are also larger blades with faceted platforms derived from more prismatic cores, with modified pieces such as scrapers, notched pieces and burins. Such material is distinctive of the Epigravettian (Late Pleistocene) phase of the Upper Palaeolithic, as evidenced in the Klissoura Cave (Argolid), and in south-eastern Europe more generally (Kaczanowska et al., 2010; Kozłowski, 2005).

The Middle Palaeolithic component at Stélida (**Figure 6**) is dominated by products from discoidal cores, plus lesser quantities of Levallois flakes and blades (**Figure 2.D**). Tools include numerous denticulates, various scrapers, and a handful of Mousterian points (Carter et al., 2014: Fig. 5). These types of artefacts are known from elsewhere in Greece, with the blades more specifically associated with the earlier Middle Palaeolithic, as for example at the Asprochaliko Cave in Epirus where they were dated to c. 100 kya (Huxtable et al., 1992).

Finally, the survey also recovered diagnostic Lower Palaeolithic artefacts, including handaxes and other bifaces (some made of non-local emery), a cleaver, plus a range of large flake-tools such as denticulates and scrapers (**Figure 7**). Examples of the same kinds of large cutting tools, handaxes, cleavers, scrapers and unifaces have been published recently from Rodafnidia on Lesbos (Galanidou et al. 2013), while the Stélida bifaces and flake cores are similar to those from the Lower Palaeolithic survey site of Rodia in Thessaly on the Greek mainland (Runnels and van Andel, 1993), and the Preveli region of southwest Crete (Strasser et al., 2011).

In sum, SNAP has provided the first direct evidence for Palaeolithic activity in the Cyclades (Runnels 2014: 217). The quarry apparently went out of use after the Mesolithic, as Neolithic and later populations apparently preferred to exploit Melian obsidian for tool production (Carter 2009: 202-203). This long-term exploitation of the chert source by hunter-gatherers thus likely occurred from $\geq 250,000$ to 9,000 BP based on the current dating of the Lower Palaeolithic – Mesolithic periods in an Aegean context; the populations involved during this time conceivably included *Homo heidelbergensis*, Neanderthals, and *Homo sapiens* (Harvati et al., 2009; Sampson, 2014).

Exactly when - and how - these characters visited the source currently remains unclear. For the Upper Palaeolithic and Mesolithic periods it is generally accepted that Naxos was insular

(albeit part of a larger landmass), whereby an expedition to Stélida would have required maritime voyaging for anyone travelling from distance (Lambeck, 1996). For the Lower-Middle Palaeolithic the situation is more complex. Current palaeogeographic reconstructions suggest that during glacial periods sea-levels may have been sufficiently low to produce a dry-route to the quarry from continental Greece and/or Anatolia, while intervening warmer eras may have left (greater) Naxos wholly insular (Lykousis, 2009: Fig. 5; Sakellariou and Galanidou, 2015). While there have been recent claims for earlier Pleistocene seafaring in the Aegean by pre-modern human populations (Runnels, 2014; Strasser et al., 2011), neither the chronology of Aegean sea levels nor the chronology of Stélida's exploitation are sufficiently precise to determine whether Stélida was continuously visited or only exploited during those cold periods when terrestrial routes to the source existed. New excavations at Stélida, in concert with a major absolute dating programme and ongoing sea-level/land-scape reconstruction studies, will hopefully clarify many of these issues in due course.

3. The siliceous materials of Stélida: their significance and characterisation

Given the evidence for the circulation of tool-making raw materials over long distances during the Middle Pleistocene – Early Holocene within the larger region (Carter et al., 2011; Moutsiou 2014; Spinapolice, 2012), the scale of chert exploitation at Stélida, and the rarity of Palaeolithic sites within the island Aegean, it seems unlikely that there was a large and permanent population on Naxos that was entirely responsible for producing the quantities of knapped stone now found there. Rather, it seems likely that this raw material source had significance to populations beyond Naxos. Evaluating such a hypothesis, however, will involve both fleshing out the corpus of Palaeolithic sites in the region and studying their lithic assemblages with the specific goal of identifying the raw materials used and their sources.

3.1 Chert characterisation and sourcing studies in the Aegean

While there is a long history of obsidian characterisation and sourcing studies in the Aegean (e.g. Carter and Contreras, 2012; Carter et al., 2016b; Milic, 2014; Renfrew et al., 1965), there are far fewer analyses focusing on other siliceous raw materials. The reason for this is twofold. Firstly, until the recent discovery of pre-Neolithic activity in the archipelago, most of the region's chipped stone assemblages were nigh-exclusively comprised of obsidian (Torrence, 1986). For example, on Naxos itself, obsidian comprises 98% of the Zas Cave Late Neolithic – Early Bronze Age material, despite the fact that the Stélida chert source is significantly closer, and would not have required maritime activity to exploit (Zachos, 1999: 158). It thus followed that most research energy was expended on obsidian studies, with the minority component of other siliceous materials given significantly less attention. Secondly, siliceous resources such as chert, jasper, chalcedony, and radiolarite are well-known to be significantly more difficult to characterise by source than obsidian; their greater geochemical and petrographic heterogeneity makes it much more difficult to clearly discriminate between products of different sources (Luedtke, 1992: 5-16). Outcrops of these lithic resources also tend to be more small-scale and intermittent than obsidian (Luedtke, 1992: 5-16), making individual sources more difficult to locate. For these reasons, there have been far fewer detailed petrographic and geo-chemical studies of non-volcanic raw materials.

As a result there has been little geo-archaeological work on the non-obsidian lithic resources of Greece, despite the fact that there are extensive outcrops of chert in the various lithostratigraphic-tectonic units of the Hellenides (Bornovas and Rondoyanni, 1983; Creuzburg et al., 1977). These include bedded, usually radiolaria-bearing cherts of the Pindos, Ionian and

Subpelagonian units in mainland Greece, Peloponnese and Crete, as well as bedded cherts capping oceanic ophiolitic lithologies (e.g. Othrys; Koziakas, Euboea, Argolid), together with siliceous materials (silcrete) formed in the course of lateritisation of ultramafic lithologies, as for example in Boeotia, the island of Euboea, and Thrace (Aubouin, 1959; Efstratiou and Ammerman, 2004: 186-187; Ferrière 1982; Sarantea-Micha, 1996; Skarpelis, 2006).

There are three relatively detailed studies of Hellenic siliceous materials, two undertaken in the Argolid, the other in southeast Crete. These projects employed macroscopic visual, textural, and knapping property descriptions to characterise lithic resources, and then used that data to claim provenience for the stone tool raw materials from prehistoric sites in the vicinity (Brandl, 2010; Kozłowski et al., 1996; Newhard, 2007). In each case the studies focused on a relatively restricted geographic scale, as opposed to trying to track the circulation of these materials at distance, and none employed elemental means of characterization.

3.2 Stélida in context: The geological background of Naxos

Naxos, the largest of the Cycladic islands and one of many above-water peaks of submerged mountains, forms part of the Attico-Cycladic belt of the Hellenides (**Figure 1**). The dominant geological unit of Naxos is a part of the Cycladic Blueschist Unit (CBU), which is the predominant lithological unit of the Attico-Cycladic Belt. The CBU of Naxos comprises mainly marbles with lenses of metabauxites (emeries and diasporites) and metapelites; minor meta-ultramafics and amphibolites are exposed throughout the sequence (Jansen and Schuiling, 1976; Wijbrans and MacDougall, 1988). The CBU experienced a complex petrological evolution between c. 45Ma to 25Ma (Altherr et al., 1982; Andriessen et al., 1979; Avigad, 1998; Jansen, 1977). The CBU was intruded by granodiorite between 14-12 Ma in the western part of the island (Andriessen et al., 1979; Pe-Piper et al., 1997; Pe-Piper and Piper, 2002). It is this western part of the island where Stélida is located (**Figures 1-2.A**).

The metamorphic rocks of the CBU and the granodiorite are juxtaposed against a non-metamorphic sedimentary pile by a detachment fault, which is the brittle expression of the Moutsouna extensional fault system, well-exposed on the eastern part of the island (Buick, 1991; Gautier et al., 1993). This fault system, which was active between ~13 to 9 Ma (Brichau, 2004), is correlated with the ductile-to-brittle low-angle fault exposed at the top of the CBU in Paros (e.g. Gauthier and Brun, 1994). The sedimentary rocks in the hanging wall of the Naxos-Paros detachment are detrital marine deposits of early Miocene age and lacustrine to fluvial deposits of early to mid-Miocene age (Boger, 1983; Kuhleemann et al., 2004; Roesler, 1969, 1973; Sánchez-Gómez et al., 2002). Naxos and Paros non-metamorphic sediments may be stratigraphically linked, since it is inferred they share the same low-angle detachment fault (Gauthier et al., 1993), and they have similar gross petrographical features. We return to the geological links between these two islands below, specifically to the occurrence of chert at both Stélida and on the Molos peninsula on the opposing Parian coast (Sánchez-Gómez et al., 2002).

The non-metamorphic rocks of Stélida comprise argillically altered shales, sandstones, and conglomerates overlain by pervasively silicified sediments (**Figures 8-9**), with a shear-zone separating the two lithologies. Conglomerates are scarce, comprised of unsorted pebbles and granules of dolomite, quartzite and biotite-microgranite in a clayey matrix. Clay minerals (illite-montmorillonite) and detrital quartz, K-feldspar, zircon and anatase were identified in the argillically altered shales and sandstones, while veinlets of quartz and pseudomorphs of goethite after pyrite crosscut bedding planes. The upper part of the sequence, raw material for toolstone

production, consists primarily of pervasively silicified shale, together with some silicified sandstones.

3.3 Previous studies of the Stélida siliceous materials

The first major study of Stélida was undertaken by Roesler (1969), who mapped the area and carried out a lithostratigraphic study of the non-metamorphic sediments, assigning them an Upper Oligocene to Middle Miocene age. That conclusion was based on marine fossil findings in pelitic and sandstone beds such as gastropods, corals, echinoderms, and lamellibranchia. The major contact of the sediments with the underlying granodiorite was interpreted as a transgressive one. He attributed the origin of the cherts to a volcanic-exhalative activity under submarine conditions (see also Roesler 1973, 1978).

The cherts that Roesler described later drew archaeological attention as well. From an archaeological standpoint, the Stélida source and its associated stone tool manufacturing debris (**Figure 2.B**) was first reported by Séfériadès (1983) after the site was discovered in a single-season survey in 1981. The raw material was described as a form of chert close to chalcedony, occasionally veined, often fractured, and ranging in colour from white, to blue, to pink. The chert was generally considered to be of poor knapping quality, or worse.

Séfériadès employed the general French term '*silex*'; we here (and elsewhere; cf. Carter et al., 2014) use the term 'chert' to refer to the siliceous rocks found at Stélida, i.e. those composed primarily of microcrystalline quartz, either formed as a sediment or as a result of pervasive silicification of sedimentary precursors. In geological parlance *chert* is the generic name for the broad group of highly siliceous microcrystalline and cryptocrystalline materials that are not primarily igneous in origin (Luedtke, 1992). Chert is essentially a chemical precipitate formed from a solution that has become super-saturated with silica. As these solutions may arise by several different geological processes, there are several different routes to chert formation. In some cases, a specific mode of origin may give rise to diagnostic features, but in the majority of cases the resulting cherts are not visually distinct, leading to problems in their characterization and sourcing. This is further complicated by the tendency for some cherts to vary in appearance, sometimes on a sub-centimetre scale.

The two primary types of chert are bedded cherts and silcretes. Bedded cherts are formed by biogenic sedimentation under submarine conditions, and comprise coarse-grained quartz as a result of diagenesis. Well preserved skeletons of radiolaria, relics of sponge spicules as well as other microfossils are common constituents, while detritus of chert or clayey material within a quartz-dominated cement are common (e.g. Aiello et al., 2008; De Wever, 1989; Danelian, 1995). They are strongly recrystallized with large voids filled with quartz. Their colour ranges between light grey to greenish- to reddish-grey. Usually they contain manganese oxide minerals in various proportions, as well as clay minerals and very minor detrital zircon and monazite. Their major, trace and REE chemistry depends on the depositional environment (continental margin, pelagic and ridge-proximal [Murray, 1994]). In contrast, silcretes are formed under subaerial conditions at the expense of ultramafic lithologies. They lack stratification and lamination, their structure resembling that of textureless fine grained quartz aggregates, which are crosscut by thin quartz veinlets. Their diagnostic mineralogical feature is the occurrence of relictic chromite or spinel, magnetite, serpentine, talc and clays, usually nickeliferous. Their chemical composition indicates predominance of SiO₂ and relatively high Fe, Cr, Ni and Mg contents.

4 Methodology

Geo-archaeologists have employed various methods to characterize chert and other (non-obsidian) siliceous toolstones, including visual analyses, geochemistry, isotopic studies, and optical petrography (Shackley, 2008: 197-198; Boaretto et al., 2009). From a brief review one might argue that combined approaches produce some of the most successful studies, i.e. those able to link artefact raw materials to specific sources, as for example the geochemical and petrographic approach undertaken recently in central Anatolia by Nazaroﬀ et al. (2013); this is the dual-methodology applied in this study (see also Bressy 2002).

Geochemical approaches to chert sourcing have a long and often frustrating history for archaeologists. This is largely because the processes of chert formation are diverse, with the chemical precipitation of silicates occurring in marine (deep-sea, or shallow water), lacustrine, and on-land contexts, which serve to produce heterogeneous materials that vary according to both their parent material, chemical, and thermal diagenetic histories, as well as their metamorphic histories (Calvert, 1971; Jones and Murchey, 1986; Parnell, 1988). Moreover chert sources can often cover very large areas, in contrast to obsidian sources which tend to be much more discrete (Shackley, 2008: 197).

The techniques employed in this study, i.e. XRF, SEM and ICP spectrometry, have been applied with degrees of success elsewhere by archaeologists to discriminate source products of different origins (e.g. Bressy, 2002; Evans et al., 2007; Nazaroﬀ et al., 2013). No clear patterns can be discerned within these studies as to which elements might be best-suited to our analyses. A range of REE were found to be most useful source discriminants in NW France (Bressy 2002: 124), while in central Anatolia ternary graphs achieved group separation using a mix of major and trace elements (Nazaroﬀ et al., 2013), and simple bivariate plots of Mn .v. Sr produced successful results in one Northern England study (Evans et al., 2007, 2167-2168, Fig. 6). Petrographic studies have variously focused on attributes of the matrix (texture, grain type, etc.), and bioclasts (micro- and macro-fossils) as a means of characterizing source products (Bressy, 2002; Luedtke, 1992; Prothero and Lavin, 1990). Once again, successful methods are context-dependent, depending on both the genetic characteristics of the material in question and its relationship to the other materials from which it must be distinguished.

Twelve geological samples of chert were collected across Stélida (**Figure 8, Table 1**), focusing on those outcrops visually similar to the raw materials most commonly used for the stone tools and the manufacturing waste the archaeological survey had documented at Stélida. Chip sampling was applied on individual outcrops; in certain areas two samples were taken. Further work will be necessary to determine whether these samples have captured the internal diversity of Stélida cherts.

For the study of the Stélida source samples mineralogical and petrographic investigation was carried out on thin and polished thin sections of the rock by conventional Plane Polarized Microscopy. Emphasis was given to the textural features of the rock. In describing the colours of the rocks we used the “Rock Colour Chart” of the Geological Society of America (Munsell® colour chips, revision 2009). The following terminology was used for description of the micro-textural features of quartz: *Microcrystalline quartz*: Massive quartz aggregates made of crystal grains that are visible in an optical microscope. *Cryptocrystalline quartz*: Dense varieties whose texture can be resolved under the SEM.

Details on the type of fine-grained accessory minerals are provided after a study of the rocks by scanning electron microscopy, specifically a JEOL JSM-5600 SEM facility with energy dispersive spectroscopy (SEM-EDS) system of the Laboratory of Economic Geology and

Geochemistry, University of Athens. Excitation potential was 20 KV, the beam current 0.5 nA, and the standards were natural sulphides or metals. The data were reduced with the aid of the ZAF programme. Rock chips were crushed and pulverized using a tungsten carbide mill, the resultant grain size of pulps being less than 75µm.

Major elements were analysed by XRF spectrometry at ALS Labs in Ireland. Lithium borate was added to calcined sample, then the mixture was fused between 1050 to 1100°C. A glass disc was prepared and then analysed. For trace- and rare earth elements (REE) a prepared sample was digested with perchloric, nitric, hydrofluoric, and hydrochloric acids and analysed by ICP-MS. Results were corrected for spectral inter-element interferences. Loss on ignition was measured at 1000°C.

5 Results

5.1 Petrography

X-Ray Diffraction (**Figure 10**) and microscopic examination of the samples show that quartz is the dominant mineral. The Stélida siliceous rock exhibits *macro-textures*, i.e. those large enough to be visible in hand specimen, and *micro-textures*, those clearly visible using optical- and scanning electron microscope techniques. The following gives a brief summary of the textures observed.

5.1.1 Macro-Textures

The Stélida rock pile occurs as thick tabular beds. It can be petrographically characterized as a brittle, massive, chert pile. It consists predominantly of silica. Bedding structure, petrographic textures and the lack of relicts of pebbles and granules (in the samples studied) indicate that the Stélida chert resulted after pervasive hydrothermal alteration (silicification), of shales or predominantly fine-grained sandstones.

It is very hard, fractures conchoidally, and has semivitreous, vitreous, or waxy lustre. The colour of the rock is very light grey to white, locally light grey with a honey hue. It displays irregular, sub-parallel, wavy bedding. Those particular textures originated by deposition of quartz into the sedimentary protolith. It appears that silica-laden hot waters (hydrothermal solution) penetrated the brittle, porous clastic sediments, leading to a fine replacement of precursor minerals by quartz. Movement of hot water was facilitated by the detachment fault at Stélida, acting as a flow path. The underlying granodiorite acted possibly as a thermal engine for heating of deeply circulating waters, in the course of cooling of the intrusion. Brown coloured quartz veinlets crosscutting massive chert, are considered as being formed at the late stage of hydrothermal alteration. Examples of the rocks cropping out at Stélida are represented in **Figure 11**.

5.1.2 Micro-Textures

The following description derives from a detailed study of thin sections of the rocks using a conventional plane polarized microscope. Microphotographs are provided showing the fine texture of the rocks and critical diagnostic textures (**Figure 12**).

The gross original texture of the protolith (layering) is preserved. Laminations on a sub-millimetre to centimetre scale are observed in all samples studied, and consist of stacked siliceous laminae of variable thicknesses. Quartz typically forms massive micro-crystalline aggregates. Open cavities are abundant. Subhedral coarser quartz crystals projecting into cavities are abundant, as well as cavities that have not totally occluded by silica. There are also late quartz veinlets crosscutting bedding planes and cavities filled with quartz.

5.2 Accessory minerals

A study of the rocks by SEM shed light on relict resistant-to-silicification minerals (zircon and anatase), as well as on minerals formed in the course of hydrothermal alteration of the sedimentary protolith (**Figure 13**). Relictic zircon grains are rare, their size being smaller than 3µm. Subhedral anatase (TiO₂) grains occur within the siliceous matrix ranging in size between 1-5µm. Hematite occurs as euhedral platy grains, as well as pseudomorphs after pyrite. Euhedral to subhedral barite grains are dispersed within the siliceous rock.

5.3 Rock geochemistry

Data on the chemistry of rocks are presented in **Tables 2** and **3**. Silica is the principal chemical constituent of all samples analysed. All other elements participate in negligible amounts. The geochemical character of the elements of the silicified sediment indicates the SiO₂ content increasing, reaching 99.8wt% mostly, whereas all other elements decreasing clearly in comparison to the underlying argillically altered shale. The major (with the exception of SiO₂), trace and REE (Y) concentration of the pervasively silicified rocks is very low compared with the composition of Post-Achaean Australian crust (PAAS), the Average European Shale (ES), as well as with the argillically altered underlying shales. The Rare Earths are strongly depleted in the silicified sediment as a result of leaching during silicification, so that most of them are below the detection limit of the analytical method. Hence construction of REE normalized patterns relative to REE average shale is not possible. The absolute concentrations of REE in the silicified rocks are lower by a factor of 35 relative to the underlying argillically altered shale, which is interpreted as a dilution effect produced by the silicification. They are also strongly depleted in all trace and REE relative to the NASC (North American shale composite) standard (Gromet et al., 1984). Light REEs were simultaneously leached out together with heavy REEs. The presence of quartz has a diluting effect on major, trace and REE concentrations. The very low proportions of anatase and zircon, as heavy minerals of the protolith, may explain the REE geochemical signature of the samples.

6 Discussion

The combined field- and lab-work, in conjunction with prior geological reconstructions of Naxos, indicate that the clastic sediments at Stélida are separated by the Naxos-Paros detachment fault from the underlying granodiorite and relics of the Cycladic ophiolitic unit. The lower sedimentary sequence has been argillically altered, whereas the upper sequence has been pervasively silicified. Those sediments were infiltrated by hydrothermal fluids moving along the Naxos-Paros detachment fault, which is correlated to the Moutsouna ductile-to-brittle extensional fault system. The fluid movement was facilitated by the detachment faults, as evidenced by deposition of hydrothermal mineral assemblages and mineralization elsewhere in the Attico-Cycladic Belt, as for example on the Lavrion peninsular of Attica (Skarpelis, 2007). Similar silicified sediments have been mapped on the hill of Agios Antonios [Skarpelis, unpublished], part of the Molos peninsula, and neighbouring Kefalos hill on the east coast of Paros, and a related siliceous formation is found on north-east Mykonos (Menant et al., 2013; Skarpelis and Gilg, 2006).

Conventional petrographic and SEM techniques demonstrate that the predominant mineral in the siliceous materials is quartz, with a number of accessory minerals in extremely low proportions, including zircon, anatase, hematite and barite. These accessory minerals cannot be

taken as diagnostic by themselves as the single diagnostic criterion to distinguish Stélida rocks from other cherts because they can be identified as accessories in other siliceous lithologies as well.

The macro- and micro-textures (e.g. lamination, colour and lustre, massive microcrystalline quartz texture, abundant coarser quartz crystals projecting into cavities, quartz veinlets crosscutting bedding planes, and cavities filled with quartz) are however, judging from out sample, characteristic of Stélida chert, as is the depletion of trace and RE elements in the silicified rock. These characteristics distinguish the Stélida siliceous rock from silcretes and bedded cherts, and may be unique to the particular genetic characteristics of these silicified sediments (i.e., the depletion in REE likely results from leaching during silicification, a process to which genetically distinct cherts would not have been subjected. Bedded cherts are formed by biogenic sedimentation under submarine conditions. They comprise coarse grained quartz as a result of diagenesis. Well preserved skeletons of radiolaria, relics of sponge spicules as well as other microfossils are common constituents. Detritus of chert or clayey material within a quartz dominated cement are common. They are strongly recrystallized with large voids filled with quartz. Their colour ranges between light grey to greenish- to reddish-grey. Usually they contain manganese oxide minerals in various proportions, as well as clay minerals and very minor detrital zircon and monazite. Their major, trace and REE chemistry depends on the depositional environment (continental margin, pelagic and ridge-proximal [Skarpelis et al., 1992; Murray, 1994]). Silcretes are formed under subaerial conditions at the expense of ultramafic lithologies. They lack stratification and lamination, their structure resembling textureless fine grained quartz aggregates, which are crosscut by thin quartz veinlets. Their diagnostic mineralogical feature is the occurrence of relictic chromite or spinel, magnetite, serpentine, talc and clays, usually nickeliferous (Skarpelis 2006). Their chemical composition indicates predominance of SiO₂ and relatively high Fe, Cr, Ni and Mg contents. Analysis of a further range of Aegean cherts will determine whether these can be distinguished as bedded cherts or silcretes, and whether those that are silicified sediments have particular diagnostic characteristics; for the latter it is necessary to establish for the region in general whether between-source heterogeneity is greater than within-source heterogeneity.

In sum, this paper has characterized the siliceous products of Stélida using combined petrographic and geo-chemical analyses on 12 geo-referenced source samples. From field- and lab-based studies it can be concluded that the chert is the product of hydrothermal alteration of shales or fine-grained sandstones, a distinct thesis to that proposed originally by Roesler (1969). Concerning the most distinctive characteristics of the raw material, i.e. those attributes that may enable the recognition of Stélida raw materials at other archaeological sites, macro- and micro-textures are potentially diagnostic, as is the depletion of REE.

7 Conclusions and Future Directions

Stélida comprises a significant stone tool raw material source in a southern Aegean context, amongst known lithic sources second in scale only to the obsidian quarries on nearby Melos in terms of evidence of on-site exploitation and quantity of knapping debris visible on the surface (Torrence, 1982, 1986). The only other documented large / archaeologically significant chert sources in Greece are located in the north, including outcrops in the Pindus mountain range in Macedonia, and the Petrota graben, an 100km² region in Thrace that is rich in outcrops both of which were exploited from the Middle Palaeolithic onwards (Efstratiou et al., 2011; Efstratiou and Ammerman, 2004: 186-187).

With regard to characterising the chert, and more specifically finding means by which the raw materials might be viewed as *distinctive* (with implications for future discrimination from products of other sources), then petrography appears to be the most significant form of analysis. The macro- and micro-textures of the siliceous rocks, including their lamination, colour, lustre, and massive microcrystalline quartz texture - amongst other features – are apparently characteristic of Stélida chert, as is the depletion of trace and RE elements. We remain hopeful that this combined petrographic and geochemical approach will ultimately allow us to move from characterisation to sourcing. As noted above, previous sourcing work on cherts in Greece has tended to employ petrographic and colour/textural approaches (Brandl, 2010; Kozłowski et al., 1996; Newhard, 2007), an analytical strategy that has achieved a certain level of success, albeit primarily at the local-mesolocal scales (<2-30km radius from site). Arguably better results stem from multi-attribute analyses that include geo-chemical data, which is then integrated with an appreciation of how the various lithic resources were being employed by the prehistoric community, i.e. the technological, and typological specificities of the artefacts by raw material. Such an approach has recently been developed and implemented successfully in central Anatolia by Nazaroff et al. (2013), and it is this approach that we aim to replicate with regard to the Naxian and other Cycladic chert studies.

As emphasised from the outset, this paper represents the first step in developing provenancing capabilities with regard to Stélida chert, providing a method with which to reconstruct the socio-economic networks within which the raw material circulated during the Middle Pleistocene – Early Holocene. The petrographic and geochemical characterization of Stélida chert presented above, apparently particular to the area's geologic history, suggest means of distinguishing Stélida chert from other Aegean/Cycladic cherts. The second step is to repeat these analyses upon other chert, and related siliceous products, from elsewhere in the region to determine whether the Stélida products are distinctive, i.e. can be geologically, and/or chemically discriminated from other raw materials. Obvious data-sets to include in this project's second stage are the siliceous materials from the Molos peninsula and Kefalos hill on eastern Paros, plus those from north-east Mykonos. Given that these are geologically related outcrops to those from Stélida it will be important to see if their respective raw materials can be discriminated, not least because Palaeolithic artefacts and related knapping debris have been seen at the Parian outcrops (T. Carter pers. obs.).

Geo-referenced collections of siliceous raw materials from the sources on Paros and Mykonos were made in the summer of 2016, and their chemical characterization using EDXRF (<http://maxlab.mcmaster.ca/>) is underway at the time of writing (petrography to follow). Beyond these nearby Cycladic chert sources, one could consider the products of various other siliceous raw materials known throughout the region (Jansen, 1973; see also Baltuck, 1982). While there is a relatively rich geological literature on these Aegean lithic resources, they have received much less attention from an archaeological, or archaeometric point-of-view (notable exceptions being Brandl, 2010; Efstratiou et al. 2011; Kozłowski et al., 1996, 297-299; Newhard 2007). The geographical remit of such a study might need to be period-dependent in its design, with the understanding that – generally speaking - procurement ranges tend to expand over time, from relatively limited during the Lower-Middle Palaeolithic (cf. Moutsiou, 2014), to long-distance by the Mesolithic (cf. Lovis et al., 2005).

If it proves possible to characterise and discriminate the products of the major Cycladic chert sources, then we should be in a position to begin provenience studies on chert tools from other prehistoric sites to see if any of them have geo-chemical/petrographic signatures that

exclusively match those of the Stélida products. Arguably Mesolithic assemblages represent the most obvious point of departure for such an analysis, as given the similar lithic traditions of these Mesolithic communities – Stélida included - and their common access to Melian obsidian (Carter et al., 2016a, in press), one might hypothesize that Stélida chert circulated through the same exchange systems (see also Carter 2016). Thus the “white patinated flint” tools from nearby Roos (**Figure 1**) on the west coast of Naxos would be a prime analytical target (Sampson, 2015), as would be the “extralocal” white chert at Maroulas on Kythnos (Sampson et al., 2014: 42). Beyond the Cyclades, the imported “silex bleu” from the Franchthi Cave in the Argolid (Perlès, 1990: 47), represents another potential data-set for archaeometric comparisons with Stélida chert.

While our paper suggests that a combined petrographic and geochemical approach is the one best-suited to pay dividends, the former are unfortunately destructive in nature which may not be an option for artefact analyses for reasons of cultural sensitivity. Ultimately it may be necessary to examine further the potential of source discrimination by non-destructive elemental techniques. To that end, the second stage of this larger characterization study has introduced the use of XRF analyses, initially involving desktop EDXRF instrumentation in a lab well-established with regard to Aegean obsidian studies (Carter, 2016; Carter and Contreras, 2012; Carter et al., 2016b). This work involves not only the aforementioned analysis of new source materials from Paros and Mykonos, but also a new suite of 177 samples from 22 geo-referenced locations on Stélida itself. With export permits for artefact analysis difficult, if not impossible, to obtain in Greece, it would be ideal if discrimination between chert sources were possible with portable XRF [pXRF] technologies (see Milić, 2014; Nazarov et al., 2013, 2014).

If ultimately Stélida chert can be identified at any of these sites, such data can be employed to reconstruct the various procurement networks that intersected at the source, and by extent allow us to comment on Aegean Mesolithic lifeways more generally (cf. Evans et al., 2007). This process can be repeated for earlier periods once Palaeolithic sites have been documented in the Aegean basin. As to the mechanisms by which those procuring Stélida chert accessed the site from the Middle Pleistocene – Early Holocene, i.e. by foot or through maritime activity, this information shall hopefully be forthcoming via the ongoing research at the site: excavation, absolute dating, and sea-/landscape reconstructions.

Ultimately understanding human/hominin activity in the Mediterranean during the Palaeolithic-Mesolithic periods, and the role of Stélida in that activity, requires a three-stage approach. Firstly, we need to characterize the siliceous products of Stélida through mineralogical, petrographic, and chemical methods. Secondly, we need to be able to use one, or more of these techniques to discriminate Stélida chert from raw materials of other regional sources, including the nearby outcrops on Paros and Mykonos. At that point we could turn to chert tools from archaeological assemblages, and attempt to match the mineralogical, petrographic, and/or geochemical signature of an artefact’s raw material to that of Stélida chert. These are the foundations required in order for us to reconstruct the socio-economic networks that came together at Stélida, and this paper represents the first stage in this process. In outlining this tripartite research scheme we are in essence following the well-established and highly successful field of obsidian-sourcing studies in the Mediterranean (Pollard and Heron, 2008). Achieving the first part of this work is the aim of this paper; stages two and three will – hopefully - be achieved in subsequent studies.

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Figures

Figure 1: Stélida on Naxos and main locations detailed in text

Figure 2: **A.** View of the double-peaked hill of Stélida from east, Paros in the background; **B.** waste flakes of chert produced by prehistoric knapping; **C.** Upper Palaeolithic and Mesolithic artefacts; **D.** Middle Palaeolithic artefacts.

Figure 3: Distribution and counts of surface artefacts collected by the *Stélida Naxos Archaeological Project* 2013-14 (S. Doyle).

Figure 4: Examples of main Mesolithic stone tool types from Stélida (1-3 flake cores, 4-5 denticulates, 6-10 'spines', 11 notch, 12 – linear, 13 – truncation, 14 – backed flake ('pseudo-trapeze'), 15 – scraper, 16 – burin (3, 10-12 are Melian obsidian).

Figure 5: Examples of main Upper Palaeolithic stone tool types from Stélida (a – unipolar blade core with lateral preparation, b – unipolar retouched blade, c – combined tool end-scraper and denticulate on flake, d-f – multiple burins on flakes)

Figure 6: Examples of main Middle Palaeolithic stone tool types from Stélida (1 – Levallois blade core, 2 – Levallois flake core; a) Mousterian point, b) Levallois point).

Figure 7: Examples of main Lower Palaeolithic stone tool types from Stélida (1 – cleaver, 2 – Clactonian notch, 3 – Tayacian point, 4 – scraper, 5 - denticulate).

Figure 8: Geological map of Stélida hill. Coordinates of samples collected for petrographical, mineralogical and geochemical studies are shown in **Table 1** (1. Naxos granodiorite, 2. Chert (pervasively silicified sediment), 3. Argillically altered sediment, 4. Diabase, 5. Aluvium, 6. Sampling points, 7. Detachment fault, 8. Abandoned quarry for aggregates).

Figure 9: **A.** Outcrop of the argillically altered shaly layers at NW Stélida, overlain by chert; **B.** Hydrothermally altered conglomerate beds. Note the dolomite (Dol) and microgranite (Mgr) clasts. Yellow-brown staining is due to oxidation of dispersed iron sulphides.

Figure 10: X-Ray diffractogram of a representative rock sample, indicating that quartz is the dominant mineral constituent. Accessory minerals are not detected due to low modal proportion.

Figure 11: **A, B, D.** Massive, semivitreous, light grey cherts with conchoidal fracturing; locally a honey hue is observed. **C:** Hand specimen of chert.

Figure 12: Microphotographs of chert (*crossed polarized light*); note the massive microcrystalline quartz texture. **A.** The bedding plane (white arrow) of the protolith is recognized by the finer-grained quartz aggregates; subhedral coarser quartz crystals are projecting into a cavity

(black arrow); **B.** Subhedral coarser quartz crystals projecting into cavities (white arrows); abundant open cavities (black arrows); **C.** Preserved layering of the protolith; subhedral coarser quartz crystals filling cavities (white arrows). Abundant open cavities (black arrows); **D.** Layering of the protolith is preserved; subhedral coarser quartz crystals projecting into cavities (white arrows). Late quartz veinlet crosscutting bedding plane and cavities filled with quartz (black arrow); abundant open cavities.

Figure 13: Back scattered scanning electron microscope images (BSE-SEM) of accessory minerals identified within the siliceous rock (Qz). Hem: hematite, Ant: anatase, Zrn: zircon, Brt: barite.

Tables

Table 1: Relative proportions of major stone tool raw materials from excavated insular Mesolithic sites in the Aegean Basin

Table 2: Coordinates of chert samples (UTM system)

Table 3: Major and trace element composition of samples as determined by XRF and ICP-MS

Table 4: REE composition of samples (ppm) as determined by ICP-MS

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Tables

Table 1: Relative proportions of major stone tool raw materials from excavated insular Mesolithic sites in the Aegean Basin

Table 2: Coordinates of chert samples (UTM system)

Table 3: Major and trace element composition of samples as determined by XRF and ICP-MS

Table 4: REE elements composition of samples (ppm) as determined by ICP-MS

Site	Date	Obsidian	Chert/Flint	Quartz
Maroulas (Kythnos)	Mesolithic	31% (n=1911)	11 (n=635)	56% (n=3382)
Kerame 1 (Ikaria)	Mesolithic	c. 40% (n=?)	c. 47% (n=?)	c. 3% (n=?)
Cyclops Cave (Youra)	Mesolithic	8% (n=15)	82% (n=147)	2% (n=4)
Livari (SW Crete)	Mesolithic	2% (n=4)	98% (n=246)	0.4% (n=1)

Table 1: Relative proportions of major stone tool raw materials from excavated insular Mesolithic sites in the Aegean Basin (data from Carter et al., in press; Kaczanowksa and Kozlowski 2008; Sampson et al. 2010, 2012)

STL 1a, b	35S 0352920E, 4105818N
STL 2a, b	35S 0352936E, 4105801N
STL 3	35S 0352964E, 4105789N
STL 4	35S 0352964E, 4105774N
STL 5a, b	35S 0352966E, 4105767N
STL 6a, b	35S 0352960E, 4105729N
STL 7a, b	35S 0352956E, 4105720N

Table 2. Coordinates of chert samples (UTM system)

Table 3: Major and trace element composition of samples as determined by XRF (major) and ICP-MS (trace)

SAMPLE	STL1A	STL 1B	STL 2A	STL 2B	STL 3	STL 4	STL 5A	STL 5B	STL 6A	STL 6B	STL 7A	STL 7B	STL 16	STL 18	NASC
SiO ₂ *	99,1	99,3	98,6	99,2	98,9	99,2	99,8	99,3	99,2	99,1	99,1	99	81,5	85,2	64,8
TiO ₂	0,01	0,01	0,01	0,01	0,01	0,01	0,01	0,03	0,01	0,01	0,01	0,01	0,48	0,42	0,78
Al ₂ O ₃	0,12	0,18	0,11	0,1	0,21	0,11	0,12	0,14	0,09	0,08	0,12	0,07	9,9	9,44	16,90
FeO ^T	0,03	0,02	0,01	0,01	0,02	0,01	0,01	0,01	0,01	0,01	0,01	0,01	0,7	0,42	5,70
MnO	<0,01	<0,01	<0,01	<0,01	<0,01	<0,01	<0,01	<0,01	<0,01	<0,01	<0,01	<0,01	0,04	0,03	0,06
MgO	0,01	0,01	0,01	0,01	0,01	0,01	0,01	0,01	0,01	0,01	0,01	0,01	0,87	0,67	2,85
CaO	0,02	0,01	0,01	0,01	0,02	0,01	0,04	0,01	0,01	0,01	0,01	0,01	0,03	0,01	3,56
Na ₂ O	0,02	0,04	0,05	0,05	0,05	0,05	0,07	0,06	0,05	0,06	0,05	0,05	2,78	0,25	1,15
K ₂ O	0,02	0,05	0,04	0,04	0,04	0,05	0,03	0,04	0,03	0,03	0,04	0,03	2,91	2,9	3,99
P ₂ O ₅	<0,01	<0,01	<0,01	<0,01	<0,01	<0,01	<0,01	<0,01	<0,01	<0,01	<0,01	<0,01	0,08	0,07	0,11
LOI	0,07	0,04	0,19	0,14	0,07	0,09	0,09	0,09	0,08	0,08	0,05	0,09	0,02	0,01	
TOTAL	99,40	99,66	99,03	99,57	99,33	99,54	100,18	99,69	99,49	99,39	99,40	99,28	99,19	99,32	
Ba ^{**}	9,1	17,2	10	10,6	30,2	8,7	44,9	10,8	10,7	4,4	13,4	8,4	191	213	636
Cr	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10	99	101	124,5
Zr	2	<2	<2	2	3	2	2	5	5	<2	5	<2	100	83	200
Rb	2,9	3,3	2,5	4,2	3,6	4,1	2,9	3,6	2,3	2,7	2,3	2,6	117	140,5	125
Sr	6,9	8,8	5,1	5,4	12,7	18,7	14	9,1	6,8	6,4	9,2	6,7	31,5	18,6	142
Ta	1,9	2	4	3,3	2,6	2,5	2,7	3,6	2,8	2,4	2,8	2,1	n.a.	n.a.	1,12
Th	0,12	0,13	0,14	0,15	0,26	0,08	0,14	0,19	0,08	<0,05	0,05	0,05	3,57	5,53	12,3
U	<0,05	0,09	<0,05	<0,05	<0,05	<0,05	0,08	0,17	0,07	<0,05	0,1	<0,05	1,36	1,8	2,66
Ga	16,5	44,6	12	10,5	27,4	38	15,6	16,8	12,5	18,1	20	13,3	12,5	15,3	n.a

STL 1A – 7B: siliceous raw material; STL 16 and 18: representative analyses of underlying argillically altered shale; NASC: North American shale composite after Gromet et al. (1984); *Major elements in wt%, **Trace elements in ppm; Tl<0,5ppm, V<5ppm; n.a.: non available

Table 4: REE elements composition of samples as determined by ICP-MS (ppm)

SAMPLE	STL1A	STL1B	STL2A	STL2B	STL3	STL4	STL5A	STL5B	STL6A	STL6B	STL7A	STL7B	STL16	STL18	PAAS ¹	ES ²	NASC ³
La	<0.5	<0.5	<0.5	<0.5	0.6	<0.5	0.5	<0.5	<0.5	<0.5	<0.5	<0.5	13.9	15.7	38.2	41.1	31.1
Ce	<0.5	<0.5	<0.5	<0.5	1	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	28	33.2	79.2	81.3	66.7
Pr	<0.03	0.05	0.03	0.03	0.1	0.03	0.05	0.04	0.04	<0.03	0.03	<0.03	3.26	4.23	8.83	10.4	7.5
Nd	0.1	0.2	<0.1	0.2	0.5	<0.1	0.2	0.2	<0.1	<0.1	0.1	0.2	11.4	14.6	33.9	40.1	27.4
Sm	0.04	<0.03	<0.03	<0.03	<0.03	0.05	<0.03	<0.03	<0.03	0.04	<0.03	<0.03	2.75	2.57	5.55	7.3	5.59
Eu	0.12	0.04	0.08	<0.03	<0.03	<0.03	0.08	0.08	0.1	0.09	0.05	0.06	0.35	0.48	1.08	1.52	1.18
Gd	0.1	0.08	<0.05	<0.05	0.07	0.05	0.08	0.05	<0.05	<0.05	<0.05	<0.05	1.92	2.27	4.66	6.03	4.9
Tb	0.01	0.01	0.01	0.01	0.03	0.02	<0.01	0.02	0.01	0.01	0.01	<0.01	0.22	0.33	0.774	1.05	0.85
Dy	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	0.1	<0.05	0.05	0.05	<0.05	<0.05	2.16	2	4.68	-	4.17
Ho	0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.01	<0.01	<0.01	<0.01	<0.01	0.37	0.42	0.991	1.2	1.02
Er	<0.03	0.07	<0.03	0.03	<0.03	<0.03	0.03	0.04	<0.03	0.03	0.05	0.03	1.22	1.38	2.85	3.55	2.84
Tm	0.01	0.04	<0.01	<0.01	0.01	<0.01	<0.01	0.01	0.05	0.01	0.02	<0.01	0.19	0.21	0.405	0.56	0.48
Y	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	<0.5	10.2	11.2	27	31.8	
Yb	0.04	0.08	0.05	0.04	<0.03	<0.03	0.05	<0.03	<0.03	<0.03	0.08	<0.03	1.25	1.38	2.82	3.29	3.06
Lu	<0.01	0.01	<0.01	<0.01	<0.01	0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	0.19	0.21	0.58	0.58	0.46

STL 1A – 7B: siliceous raw material; STL 16 and 18: representative analyses of underlying argillically altered shale

¹REE composition of Post-Achaean Australian sedimentary rock (PAAS) after McLennan (1989); ²Average European Shale after Haskin and Haskin (1966); ³ North Atlantic shale composite (NASC) after Gromet et al. (1984) and Haskin and Haskin (1966).

0 2cm

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1)

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4)

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A.

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STL131B

Operations: X Offset 0.017 | Import

00-033-1161 (D) - Quartz, syn - SiO₂ - Y: 78.39 % - d x by: 1. - WL: 1.5406 - Hexagonal - I/Ic PDF 3.6 - S-Q 100.0 % -