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PROVENANCING ARCHAEOLOGICAL CHERT FINDS WITH pXRF: INITIAL RESULTS FROM THE EASTERN COAST OF THE BOTHNIAN BAY

Abstract

We present the initial results of a research combining non-destructive chemical analyses with a quantitatively and chronologically representative research assemblage – 52 specimens from five sites – to examine the provenance of Late Neolithic and Bronze Age chert finds from the cluster of sites located near the city of Oulu on the eastern coast of the Bothnian Bay. The results confirm the previously observed transition in the use of raw material sources: eastern Carboniferous cherts high in iron were replaced by calcium-rich Cretaceous flints of Scandinavian or southern Baltic origin. We also consider the overall applicability of pXRF as a non-destructive research method to determine the provenance for archaeological chert finds recovered from the coniferous boreal zone, characterized by the impact of post-depositional weathering on the chemical composition of objects found in the soil matrix.

Keywords: Bronze Age, Bothnian Bay, chert, provenance, pXRF, weathering

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INTRODUCTION

This paper presents the initial results of a research program combining non-invasive chemical analyses with a quantitatively and chronologically representative assemblage of chert finds from two site clusters located on the eastern coast of the Bothnian Bay in northwest Finland to answer not only methodological but also archaeological research questions. The research method, an X-ray fluorescence analysis performed with a portable analyzer (pXRF), has become very common over recent years in archaeology, but the ways the method is applied and the results interpreted have also been intensively debated (e.g., Frahm 2013; Speakman & Shackley 2013). Here, the intention is to assess the applicability of pXRF as a non-invasive method to establish a provenance for a quantitatively significant number of archaeological chert finds recovered from the coniferous boreal zone, where the impact of post-depositional alteration can possibly have an effect on the chemical composition of objects found in the soil matrix.

From the archaeological standpoint, the aim of the article is to revitalize the Finnish scholarship focusing on the provenance of chert, which is an umbrella term for a group of sedimentary rocks consisting primarily of microcrystalline quartz (Luedtke 1992: 5). Especially in Scandinavia, these rocks are commonly discussed in archaeological literature under the 'folk category' term flint (e.g., Johanson 2021 et al.: 123–4). However, as this term is also used in more restricted sense to define a black, nodular subcategory of highly siliceous chert associated with Cretaceous deposits (e.g., Stow 2005: 184), the term chert is preferred throughout this article. The specific archaeological research question to be dealt with here concerns the provenance of Late Neolithic and Bronze Age chert finds of northern Finland. As indicated below, this subject has only been touched upon in previous Finnish archaeological scholarship.

To sufficiently succeed in communicating various aspects related to these two goals, the remainder of the paper is structured as follows. First, a concise literature review is offered on the state of chert studies in northernmost Europe, the area of Fennoscandia and adjacent regions in particular. Here, special attention will be paid to the recent scholarship focusing on geological deposits and the geochemistry of local cherts. Next, the two prehistoric activity areas yielding the research material for this study will be introduced together with arguments justifying their selection and the characterization of archaeological finds. As the authors are well aware of the advantages and pitfalls that the use of pXRF incorporates (see, e.g., Drake et al. 2009; Shackley 2010; 2012), materials and methods will be described in detail, and special attention has been paid to the analyses of research materials. The results and their interpretation will be followed by a discussion about their wider implications that touches upon both the suitability of pXRF in the analysis of archaeological chert finds from the boreal zone and the picture regarding long-distance contacts of trade and exchange in the research are during the Bronze Age.

LITERATURE REVIEW

Virtually every study touching upon the use of chert and related siliceous rock types in prehistoric Finland begins with a laconic statement pointing out the absence of chert – "sedimentary rocks of biogenic, biochemical or chemogenic origin" (Stow 2005: 184, see also Luedtke 1992: 5; Burke 2018: 1) – from the local bedrock (e.g., Huurre 1986: 53). Thus, when found in an archaeological context, these siliceous rocks bear evidence of long-distance contacts that might have taken the form of gift-giving, exchange, or trade. Prehistoric chert imports in Finland are traditionally thought to fall into two main groups according to their geographic and lithologic origin (Fig. 1). First, siliceous nodules that occur in Cretaceous chalk are available in an east-west oriented, ca. 1400 km long belt that extends from southern Sweden and northern Denmark through Lithuania to Russia (e.g., Baltrūnas et al. 2006; Hughes et al. 2012). On the other hand, Carboniferous deposits containing chert form a north-south oriented and ca. 1100 km long belt in northwest Russia that extends from the Valdai region to the White Sea (e.g., Zhuravlev 1982; Zhilin 1997; see also Kinnunen et al. 1985: 7, fig. 1). Minor deposits of Ordovician and Silurian chert have also been identified in Estonia and Latvia (Yurgenson 1958; Kröger 2007; Kriiska et al. 2011: 67; Johanson et al. 2021: 124–5). Thus, the geological setting corresponds well with Shackley's (2008: 205–6) observation concerning the wide distribution of chert deposits and the potential elemental and isotopic variation within them.

Reflecting this reasoning, in order to use quantitative elemental analysis on provenance, the research activity on various chert deposits of Scandinavian and Baltic origin has intensified significantly during the past decade. This is mainly due to the extensive chemical characterization program carried out by Anders Högberg, Richard E. Hughes, and Deborah Olausson. While mainly focusing on geological outcrops of Scania (southern Sweden) and Denmark (Hughes et al. 2010; 2012; Högberg et al. 2012), these scholars have not only studied additional deposits on the Swedish islands of Gotland and Öland (Högberg et al. 2016) but also extended their activity to the Baltic and beyond (Högberg et al. 2013; 2014). The main outcome of their research program is the observation that geochemical methods, the determination of Ca/Fe-ratio in particular (Hughes et al. 2010: 21–2; Olausson et al. 2017; see also Johanson et al. 2021: 127), can be used to distinguish various chert deposits

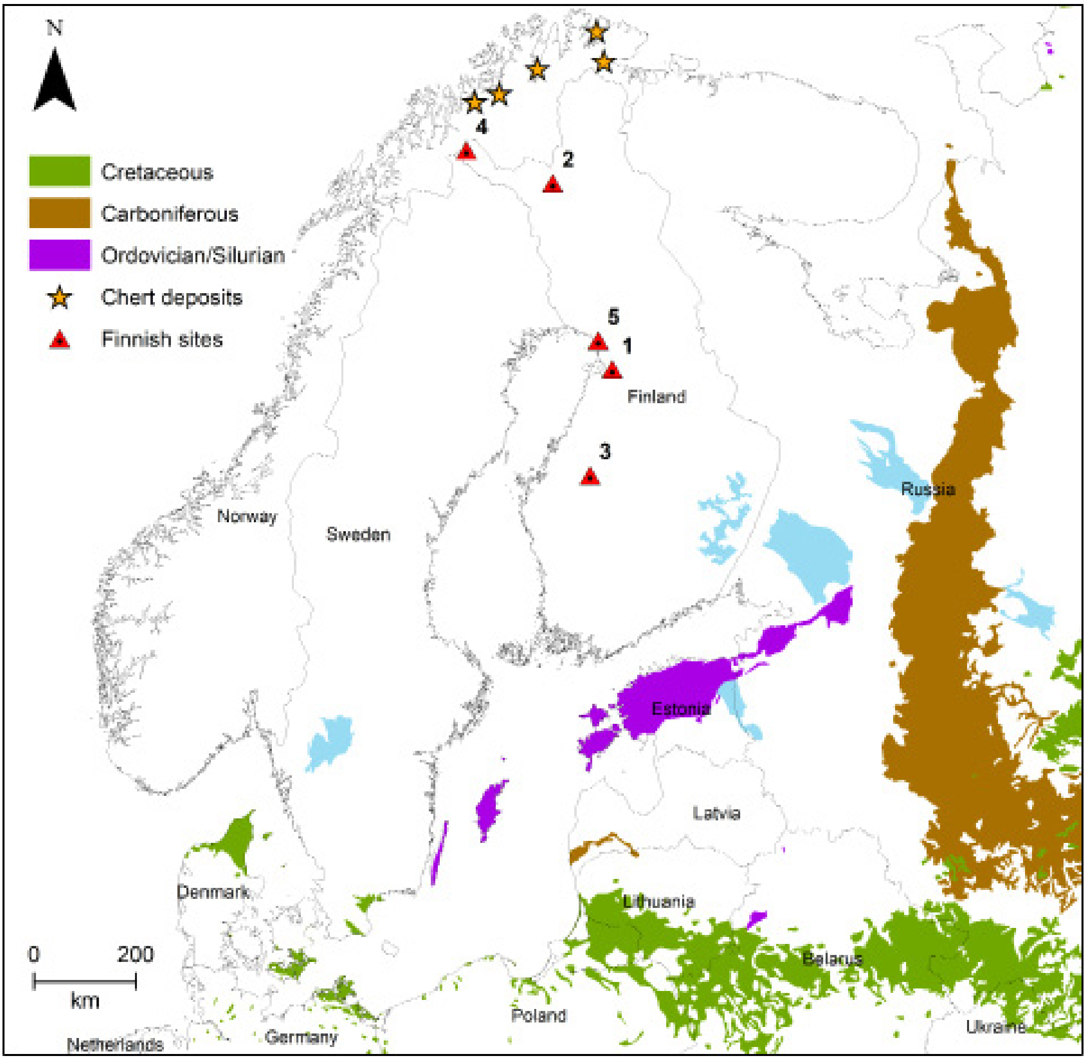

Figure 1. Overview map of the Rautajärvi area. Dots mark archaeological sites (black) and stray find (white) locations. The Järvensuo 1 site is marked with a star. Background data by Finnish Heritage Agency (2020a; 2020b) and National Land Survey of Finland. (Map: Janne Ikäheimo.)

from one another. Therefore, the results of their research have been adapted as the framework for this paper, which is further complemented with the observations of other scholars that have recently touched upon the topic in Scandinavia, the Baltic, and northwestern Russia (e.g., Olofsson & Rodushkin 2011; Zariņa et al. 2014; Zariņa & Segliņš 2017; Sinitsyna & Kolokol'tsev 2018).

It is also worth pointing out here that while the last Ice Age deprived the Finnish bedrock of Phanerozoic formations, save the southernmost part and the northwesternmost tip of the country, deposits of metamorphosed Precambrian cherts are known from the area. The best-known case to Finnish archaeologists is the occurrence of red jasperoid in the Kittilä area in Finnish Lapland (Kinnunen 1982; Vartiainen 2017), but deposits of red jasper have recently been also reported from Vimpeli, southern Ostrobothnia (Kinnunen 2008). In addition, several geological research reports contain references to other deposits of metamorphosed Precambrian cherts than jasperoid (e.g., Lehto & Niiniskorpi 1977; Sipilä et al. 2008; Öhman 2017), which might be of interest for the future research. Moreover, traces of small-scale quarrying of Cambrian sedimentary deposits have been discovered in northwestern Lapland by the Norwegian border at Kuonjarvarri (Guonjarvárri) in Enontekiö (e.g., Halinen 2005: 27–8). It is uncertain, however, whether the quarried rock is actually chert or quartzite, because recrystallized chert and fine-grained quartzite are difficult to tell apart (Luedtke 1992: 27) without petrographic analyses.

While cherts and related rock types (e.g., flint, chalcedony, and jasper) in Finnish archaeological assemblages have been studied quite intensively (for research history, see e.g., Manninen et al. 2003: 162–8); the provenance of the raw materials has been studied with scientific research methods rather sparingly. Nonetheless, two important contributions were published already in the late 1980s. Kinnunen et al. (1985) explored various properties of chert, such as texture, mineralogy, and microfossil content, whereas Matiskainen et al. (1989) applied atomic absorption spectrometry (AAS) to carry out the only sufficiently comprehensive geochemical study of chert-like materials published to date in Finland. Most samples they examined, 70 in total, were archaeological finds that were destroyed in the analysis along with a selection of geological reference materials. The results suggested a rather clear-cut and temporally significant division between the provenance of Neolithic and Bronze Age cherts with a chronological shift from the exploitation of the eastern to the western chert sources (Matiskainen et al. 1989: 636–7).

Thereafter, only a re-examination of Matiskainen's research group results incorporating some new data has been published by Costopoulos (2003). This new data was acquired with electron probe microanalysis (EPMA), but no further information was provided about the conditions under which these analyses had been performed. The analyses were carried out on six fragments chipped off from two chert finds – a late Neolithic basal biface fragment from Ii Hiidenkangas and an early Bronze Age flake from Muhos Halosentörmä – found at two dwelling-sites in northern Finland. Based on these new results, Costopoulos (2003: 52) concluded that contrary to the southern parts of the country, no synchronous re-alignment of the trade networks could be observed with the introduction of bronze metallurgy to northern Finland.

As Costopoulos based his far-reaching conclusions on quantitatively limited research material, it was deemed appropriate to re-examine his hypothesis about the change in chert supplies from the Late Neolithic to the Bronze Age in northern Finland using a larger research assemblage. Simultaneously, it was also essential to form the research assemblage in a way that the methodological goals of this article – assessing the applicability of pXRF as a non-destructive research method to determine the provenance for archaeological chert finds recovered from the coniferous boreal zone – could be reached. This twofold objective, in addition to the small number of excavated archaeological sites pertaining to the period of interest, left very few options regarding the choice of materials to be studied. The rationale behind their selection will be described next.

MATERIALS

The Sites

Only a few Bronze Age dwelling sites are known from northern Finland, comprising the provinces of Lapland, Kainuu, and Northern Ostrobothnia, as they customarily lack any features that could be detected as visible anomalies on the ground surface. Instead, these sites are characterized by tightly clustered scatters of lithic debitage, charred bone, and fire-cracked rocks found within few hundred square meters of space. Due to these reasons, "pure" Bronze Age dwelling-sites are seldom spotted in archaeological surveys unless the ground has been recently disturbed by earth-moving activity. In addition, a great amount of the existing material evidence on the Bronze Age in northern Finland pertains to multi-period inland sites located in the provinces of Kainuu and Lapland (e.g., Huurre 1986: 56, fig.) that were used either periodically or permanently from the Stone Age to the Iron Age. When some of these sites were excavated between the 1950s and 1970s, not enough attention was paid to their stratigraphy. Thus, while the resulting find assemblages may contain a fair amount of chert finds, the absence of sufficiently precise information about their chronological position enforces their exclusion from this study.

The situation is drastically different on the coastal area of the Bothnian Gulf characterized by active and still-ongoing land uplift caused by the post-glacial isostatic rebound. There, prehistoric dwelling-sites were often used for a relatively short period of time, because the people exploiting marine resources had to relocate their settlements once in a while closer to the "escaping" seashore (e.g., Hakonen 2017). The current rate of land uplift in this area can be utilized to estimate the vertical position of the seashore in the past, indicating the altitudes of 20–40 meters above the current sea level as those corresponding roughly with the late Neolithic and Bronze Age shorelines.

The area of Hangaskangas – a 7 km^2 wide glacial sand esker located by the Oulujoki River ca. 20 kilometers southeast of the city of Oulu in the province of Northern Ostrobothnia – comes here to the fore. It is the sole locality on the coast of the Bothnian Gulf with several dwelling sites that are chronologically successive and datable either to the Late Neolithic or the Bronze Age based on their location altitude above the sea level. Furthermore, the archaeological excavations that have taken place at these sites in recent years were carried out with modern archaeological methods thus yielding significant amounts of datable chert finds.

This archaeological evidence from the area of Hangaskangas pertains to two main clusters (Fig. 2, Table 1), one belonging administratively to the city of Oulu and another to the municipality of Muhos. Located by the southeastern tip of the Hangaskangas esker, the Muhos Halosentörmä site was subjected to several campaigns of excavation between 1968 and 2012 (Kopisto 1968; Ikäheimo 1999; 2001a; 2003; 2015), and the site of Muhos Hangaskangas, excavated in 2000 (Ikäheimo 2001b), is located a few hundred meters west of it. The Oulu Hangaskangas E site cluster, excavated in its entirety (Pesonen 2013; Mikkola 2015), on the eastern flank of the esker turned out to consist of several activity areas located on different altitudes and commonly referred to as dwelling-sites. Radiocarbon dates obtained from the sites in the area of Hangaskangas corroborate, although in a somewhat ambiguous manner, the positive correlation between the vertical position of the site and its date (Table 1). As indicated by the table, the Oulu Hangaskangas E1 site on the highest elevation was probably in use during the last centuries of the 3rd millennium BC, while the lowermost radiocarbon dated site of Oulu Hangaskangas E2 falls with high probability to the early 1st millennium BC. Thus, the chronological coverage of these sites extends from the Late Neolithic period at least to the early Late Bronze Age, assuming that the beginning of the Bronze Age is placed around 1950/1900 cal. BC (Kristiansen 2018) and that the Late Bronze Age begun ca. 1000 cal. BC.

Finds and specimens

The 118 chert and related siliceous rock finds recovered in various archaeological excavations form just a fraction of the total lithic assemblage found from the sites located in the Hangaskangas

Table 1. The sites with chert finds discussed in the article.

Site	Elevation	^{14}C-dates	cal.BC	Material
Oulu Hangaskangas E1	40.85–.90	Ua-45450 3695+35BP1)	2200–1972	charred bone
Muhos Halosentörmä	36.25–.75	Hela-154 3420+105BP	2019–1463	chewing resin
		GrA-63888 3000+35BP	1386–1121	charred bone
Muhos Hangaskangas	33.00	GrA-63520 3195+35BP	1518–1410	chewing resin
Oulu Hangaskangas E2	30.50–.75	Ua-45447 2775+40BP	1013–824	pot crust
		Ua-45451 2710+35BP	920–806	charcoal
Oulu Hangaskangas E8	26.00	–	–	–

Elevation: meters above sea level in Finnish N2000 vertical coordinate reference system.
Cal. BC= dates calibrated to 2σ with OxCal 4.4 on-line calibration program (Bronk Ramsey 2009) using IntCal20 calibration curve (Reimer et al. 2020).
1) Date from Oulu Hangaskangas E3 -site located at the same elevation Oulu Hangaskangas E1.

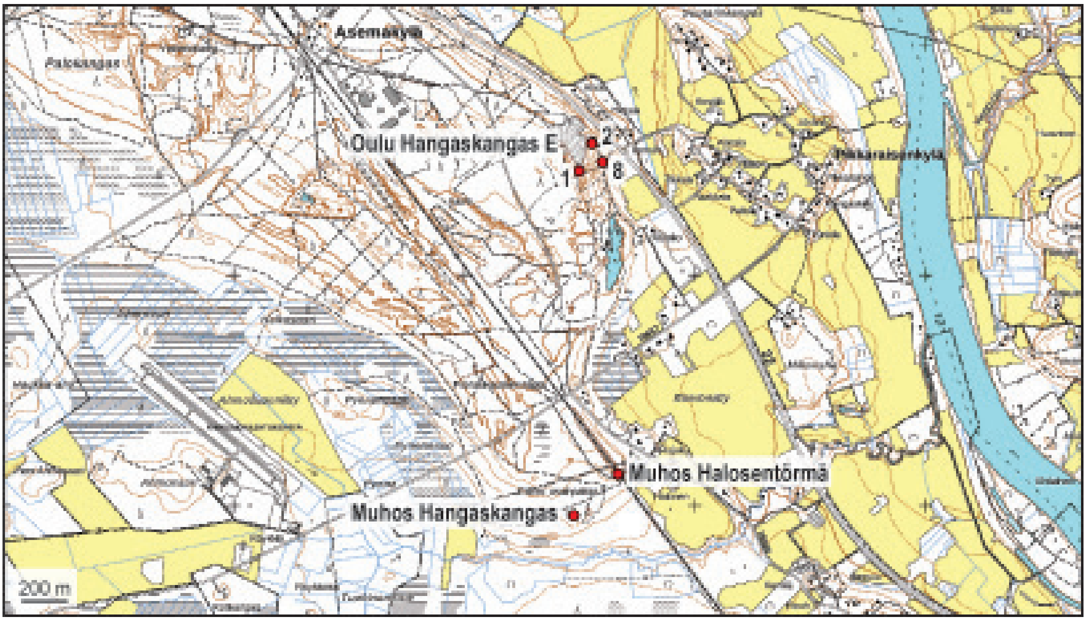

Figure 2. The location of archaeological sites with chert finds in the Hangaskangas area. Base map: National Land Survey of Finland, CC BY 4.0.

area (Table 2). Most likely due to its availability with several venous outcrops located within a 10-kilometer radius from the two site clusters, quartz was the most common raw material used for the making of small stone artifacts followed in importance by various types of quartzite. While the quartz assemblage totals nearly 25 kilograms in weight, the chert assemblage weighs only 82.69 grams in total. A considerable number of the chert finds (Fig. 3) can be classified as flakes detached from the artifact as it was gently rejuvenated to suit better for the intended purpose. Besides the size and formal attributes of these fragments, this is reflected by the average weight of the specimens in the assemblage; half of the finds weigh 0.1 grams or less. Of the proper artifacts identified, most are scrapers or their fragments, while a nearly intact bifacial arrowhead of black chert (Appendix 1: 14; Fig. 3) is the only find standing out in the assemblage. However, the chert assemblage is both quantitatively large and visually heterogeneous enough – ranging from fine-grained translucent black and greenish specimens with traces of chalky cortex to coarser, opaque reddish-brown or brown pieces with layered structure – to potentially include finds pertaining to several geological outcrops and source areas.

The research material is currently stored by the Finnish Heritage Agency in Helsinki, the Museum of Northern Ostrobothnia in Oulu, and the Archaeology Laboratory of Oulu University. All these institutes granted a swift access to the materials in their storage only after finding out that the aim was to apply a non-destructive research method on a large sample of archaeological finds. Therefore, the physical modification of the finds to fit them better over the instrument's analysis window was excluded at the outset. Yet, common recommendations for performing elemental analysis of lithics with a pXRF (e.g., Williams-Thorpe 2008: 181) were followed as closely as possible.

While flat and substantially large sample surfaces offering uniform conditions for analysis would have been desirable, they were infrequently available. In such cases, convex surface geometry was preferred over concave, to minimize the distance variations between the sample and the analyzer window. Catalog numbers inked on some specimen surfaces were intentionally avoided (see also Hughes et al. 2012: 787). Each sample was also visually assayed for

Table 2. Excavated sites and their chert assemblages.

Site	Year	m²	CHERT FINDS N	x wgt	pXRF
Oulu Hangaskangas E1	2012	52	4	0.66	3
Muhos Halosentörmä	1968	180	14	2.73	14
	1998–9, 2012	47	57	0.20	16
Muhos Hangaskangas	2000	62	2	5.04	2
Oulu Hangaskangas E2	2012	44	40	0.42	16
Oulu Hangaskangas E8	2012	24	1	8.26	1

pXRF= the number of finds analyzed for this article

other impurities and defects that could have affected the results. Compensation for the possible chemical heterogeneity known as the nugget effect (see Burke 2018: 3) that results from mineral inclusions potentially present in the sample due to the differences in the deposition of the parent material was sought by analyzing each specimen three times.

Sample diameter and thickness can also influence the analysis results, as previously demonstrated by experimental studies of archaeological basalt and obsidian finds (Lundblad et al. 2008; Davis et al. 2011). For example, the analyses of the lightest elements like MgO, Al_2O_3, and K_2O are most prone to a bias resulting from sample thinness (Lundblad et al. 2008: 7–8; see also Desroches et al. 2018: 38, 40). The heaviest element to be included in the present analysis was iron (Fe), for which the depth of analysis in aluminosilicates is ca. 200 microns, whereas it is much less for the lighter elements (see Grave et al. 2012: 1676, fig. 2) implying that the many specimens in the assemblage met well the requirement for infinite thickness (e.g., Ferguson

Figure 3. A selection of chert finds from the Muhos Halosentörmä and Muhos Hangaskangas sites (finds by row [see Appendix 1 for details] – top : 11, 20, 24, 12, 6, 19; middle: 1, 14, 9, 8; bottom: 31, 23, 30, 29). (Photo: Janne Ikäheimo.)

2012: 413–4). After all the variables potentially influencing the results had been considered with each find, altogether 52 archaeological specimens were analyzed for the study (see Appendix 1).

It goes without saying that as the chert assemblage has been exposed to chemical processes through soil alteration and weathering, the chemical stability of these lithics is not comparable to samples that have been recently hammered off from respective geological deposits. This is especially evident at the area of Hangaskangas. While the two site clusters are located only 1.4 kilometers apart from one another (Fig. 2) in environmentally uniform conditions, the finds have been embedded for several millennia in a matrix formed by podzol soil that typifies subarctic coniferous forests. It is characterized by extensive leaching and enrichment of various elements that become visually discernible in the soil profile as distinct vertical layers over the time (e.g., Tyler 2004).

In podzol, the grayish-white eluvial layer underneath the topsoil is depleted from many compounds, while the underlying illuvial layer is visually discernible from the bottommost stratum of 'sterile soil' (parent material) due to its reddish-brown color resulting from the enrichment of iron and aluminum. Therefore, the compositional data measured from a chert specimen might be influenced by its position in the soil matrix and this must be taken into account when drawing any conclusions about the assemblage. As any invasive chemical or mechanical procedure (e.g., acid treatment or ultrasonic bath) to remove or to significantly diminish the effect potentially caused by weathered outer surface was not allowed due to the status of these specimens as archaeological finds (see also Gauthier & Burke 2011: 270), each of them was carefully wiped with a cotton pad soaked in pure ethanol before the analysis.

METHODS

Instrumentation

The Bruker Tracer IV-SD (S/N T4S1945) portable XRF-analyzer (pXRF) manufactured by Bruker AXS Elemental Inc. was used for this study. This instrument has a rhodium target X-ray tube and is equipped with a Peltier-cooled 10mm^2 X-Flash silicon drift detector. Instrument-specific analytic parameters were controlled with X-ray Ops -software (version 1.2.21), while the spectra used to establish an instrument-specific chert calibration and the subsequent chert analysis were acquired with S1 PXRF S1 MODE -software (version 3.8.32), both designed and distributed by Bruker AXS Elemental Inc. The instrument was operated under vacuum (<1 torr) for improved light element performance (see Shackley 2011: 30) at 15 kV and 55 µA without tube filters from an external power source.

An alternative investigative approach suggested by Conrey et al. (2014: 292) with the voltage set to 45 KeV and tube current to 25 µA was also briefly explored. Higher voltage would have enhanced intensities for heavy elements while simultaneously diminishing intensities of the lighter elements, and it would have also yielded Compton scattered RhK_α intensities for matrix correction. However, as these settings resulted in a serious instrument instability over prolonged times of operation, this line of investigation had to be terminated before respective data on all samples had been collected.

All analyses, including both reference materials to be described shortly and archaeological finds, were performed 'from below' as the instrument was sitting in a stand with the beam pointing upward. The sample was in atmospheric conditions under the safety shield of the instrument with an Ultralene™ gridded window (P/N 485315-400) separating it from the vacuum. In all cases, the instrument registered spectra using 2048 channels with an average resolution of 20.03eV and with the time of analysis fixed to 240 live seconds. Previous research (Newlander et al. 2015: 542–5) has shown that while the counting error does not generally decrease significantly when the count time exceeds 180 seconds, the analysis of cherts can be enhanced by using longer count times. This is because other elements than silicon are normally present in cherts, particularly in flints, in low concentrations.

Reference materials

The calibration of the chert matrix was performed with the use of appropriate certified reference

Table 3. Certified reference materials and the elements used in the calibration.

	SiO_2	Al_2O_3	Fe_2O_3	MnO	MgO	CaO	K_2O	TiO_2	P_2O_5	S	Cl
AC-E	70.35	14.7	2.53	0.058	0.03	0.34	4.49	0.11	0.014	0.007	0.018
AMIS0305	96.7	1.2	1.36	–	0.04	–	0.27	0.07	0.01	–	–
FK	88.2	6.18	0.261	0.004	0.15	0.11	4.23	0.058	0.077	–	–
FLX-13	46.93	3.63	0.42	0.42	2.28	4.96	4.93	0.45	0.53	0.42	0.37
GSD-10	88.98	2.84	3.86	0.13	0.12	0.7	0.125	0.21	0.62	0.009	0.0053
GSD-8	82.92	7.71	2.2	0.04	0.25	0.25	2.83	0.61	0.03	0.01	–
GSD-9	64.89	10.58	4.86	0.08	2.39	5.35	1.99	0.92	0.15	0.015	0.005
GSR-4	90.36	3.52	3.22	0.02	0.082	0.3	0.65	0.26	0.22	0.086	0.0042
JCh-1	97.81	0.734	0.356	0.017	0.754	0.0449	0.221	0.032	0.017	0.0004	0.0014
J-Gb1	43.44	17.66	15.16	0.17	7.83	11.98	0.24	1.62	0.05	0.195	–
NBS-91	67.53	6.01	0.081	0.008	0.008	10.48	3.25	0.019	0.022	–	–
NIM-L	52.4	13.64	9.96	0.77	0.28	3.22	5.51	0.48	0.06	–	0.12
Q1	99.9	–	–	–	–	–	–	–	–	0.002	0.018
Q2	99.9	–	–	–	–	–	–	–	–	0.002	0.022

Origin and type: Institute of Geophysical and Geochemical Exploration, China (sediment powders GSD-8, GSD-9 and GSD-10); National Research Centre of Geoanalysis, China (sandstone powder GSR-4); Geological Survey of Japan (chert powder JCh-1 and gabbro powder JGb-1); African Mineral Standards, South Africa (blank silica chips AMIS0305); National Institute for Metallurgy, South Africa (lujavrite powder SARM3 NIM-L); Zentrales Geologisches Institut, Germany (felthspatic sand powder FK); Centre de Recherches Petrographiques et Geochimiques, France (granite powder AC-E); National Bureau of Standards, USA (opal glass NBS-91), FLUXANA, Germany (XRF monitor glass FLX-13), in-house monitor glasses (Q1 and Q2) measured with a Malvern Panalytical benchtop XRF.

materials and additional in-house parallel method determined calibration samples (see Donais & George 2018: 54–7) that would sufficiently cover the needed concentration range and match the silicon matrix of chert. A previous study focusing on the application of a hand-held XRF analyzer to monitor the quality of quartz in an industrial setting has shown that this matrix type is quite uncomplicated to analyze (Desroches et al. 2018: 37). The sixteen calibration samples selected for this study comprised both pressed powder pellets and glass discs (Table 3). While potentially running the risk of being affected by matrix effects (see Shackley 2010: 19; 2011: 18–21; Ferguson 2012: 408–9), the use of solid glass (amorphous SiO_2) and pressed pellets of powders (chert, sediments, etc.) was necessitated by the general unavailability of suitable reference materials for pXRF, particularly those high in silica (see Conrey et al. 2014: 292; Burke 2018: 3; Desroches et al. 2018: 38).

The certified reference materials chosen for the study, for which the origin, type, and the reported standard composition of elements relevant for the present study, are shown in Table 3. All glass discs were analyzed per se, while 10 % of wax (Hoechst) was used in the preparation of pressed powder pellets. In two cases (GSR-4 and JCh-1), another pellet was prepared from the same reference material using 12 % of wax to increase the stability of the sample due to minute edge crumbling observed in the 10 % pellet. At least one reference material was chosen for each element of interest with concentration well above or below the usual range present in chert and other siliceous rocks to produce a meaningful concentration estimate for extended range (see, e.g., Ferguson 2012: 406–7; Shackley 2011: 34; Conrey et al. 2014: 292).

In addition, five geological samples of known general provenance – two from Denmark and Russia and one from southern Sweden – obtained from our study collection and through collegial solidarity were analyzed together with the archaeological finds. Due to the explorative nature and limited scope of the study, the use of quantitatively and geographically more representative geological reference collection was left for the future. Yet, the inclusion of these samples in the study was necessary to determine the consistency of analysis results between pressed powder and glass samples used in calibration

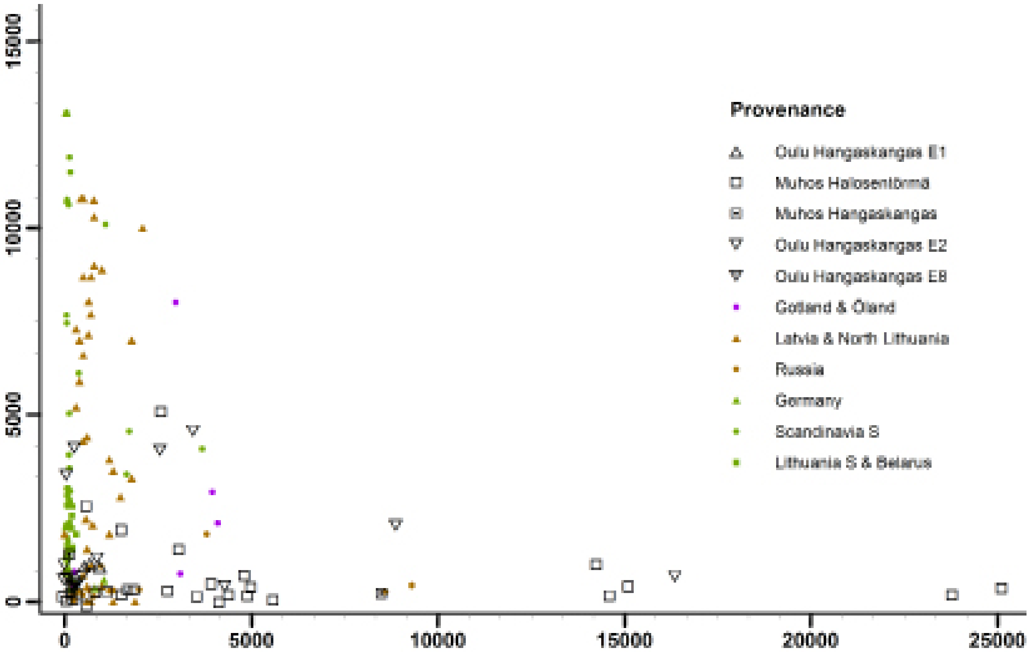

Figure 4. The Ca/Fe-ratio of all analyzed samples with reference values from published geological deposits. All values in ppm. Find chronology: up-pointing triangle= Late Neolithic, square= Early Bronze Age, down-pointing triangle= Late Bronze Age.

and irregular geological and archeological finds targeted to be measured with developed calibration.

Calibration

The authors agree on a conceptual level with Shackley's (2008: 196) statement that "nothing is ever really 'sourced'" and are aware of the challenges that the characterization of cherts with pXRF may present due to variation in lithological and chemical compositions (Newlander et al. 2015: 544). Yet, in the light of recent successful lithic provenance studies executed with a pXRF (Forster & Grave 2012; Grave et al. 2012; Newlander 2012), the authors took a positive stand in developing a calibration suited for archaeological chert finds recovered from the northern boreal zone instead of applying general rock calibrations not specifically matched to a chert matrix provided by the instrument manufacturer (cf., e.g., Carvalho & Pereira 2017).

CloudCal 3.0 software (Drake 2018) was used to calibrate each element of interest by establishing the closest fit between the spectra acquired with S1 PXRF S1 MODE -software and the published values of standard reference materials. The elements included in the calibration using K_α lines were the following: Mg, Al, Si, P, S, Cl, K, Ca, Ti, Mn, and Fe. Measured spectra were normalized by time averaging. For the two elements of interest in this article, calcium, and iron, linear calibration was the most reasonable approach, while non-linear regression was needed for some other elements.

RESULTS

The spectra resulting from the analysis of archaeological specimens were translated into relative element concentrations with the afore-described calibration using CloudCal 3.0. software. The analyzed finds are listed in Appendix 1. To make the results easily comparable with previous studies focusing on the geochemistry and provenance of chert around the Baltic Sea and adjacent areas, iron and calcium values were plotted (Figs. 4–6) together with previously published values for geological specimens and archaeological finds (Matiskainen et al. 1989; Hughes et al. 2011;

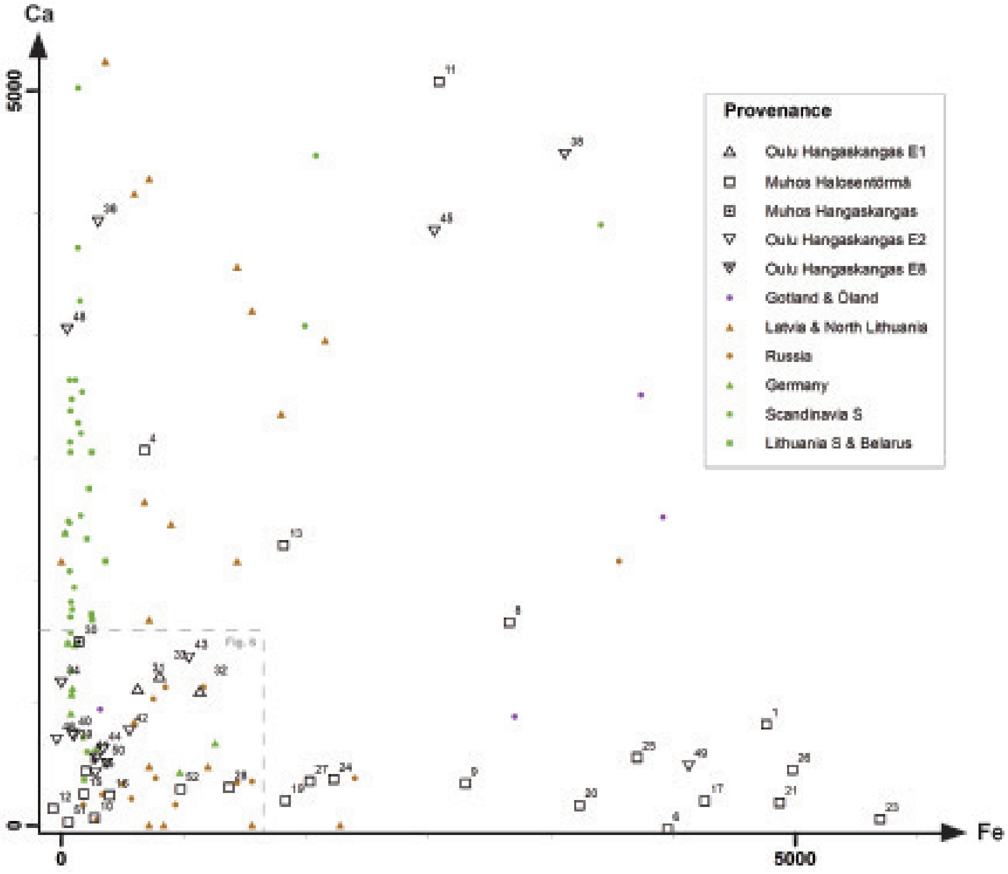

Figure 5. The Ca/Fe-ratio of samples in < 5000 ppm range with reference values from published geological deposits. Find chronology: up-pointing triangle= Late Neolithic, square= Early Bronze Age, down-pointing triangle= Late Bronze Age.

2012; Olofsson & Rodushkin 2011; Olausson et al. 2012; Högberg et al. 2014; 2016; Zarina et al. 2014; Sinitsyna & Kolokol'tsev 2018). The plotting was executed with ArcMap 10.7.1 GIS-software for improved visual scalability necessitated by the considerable variation in element concentration both in the actual results (Ca ≤5,062 ppm; Fe ≤23,781 ppm) and the reference values (Ca ≤215,000 ppm; Fe ≤50 000 ppm). Before reviewing the outcome and comparing it with previously published results, it is worth underlining here that due to variation in research methods used and analysis routines applied, their inter-comparability can reasonably be questioned. Yet, in spite of possible discrepancies, the overall pattern seems to be distinct enough for drawing generalizing conclusions.

This being said, several observations can be made regarding the results themselves (Fig. 4). First, the calcium content of many geological specimens, especially the ones from Gotland, Öland, and North Lithuania, exceeds significantly the values measured from archaeological finds. While this could be taken at face value as an indication that no chert from these sources reached the Hangaskangas area during the Late Neolithic and the Bronze Age, surface weathering must also be considered as a potential contributing factor to low calcium values (see Gauthier & Burke 2011: 278). Moreover, as all specimens yielding under 400 ppm concentration for calcium in various geological analyses pertain to the eastern deposits of Moscow, Valdai, and the White Sea, this value might be used as a threshold for assigning general provenance between the east and the west.

On the other hand, several finds from the area of Hangaskangas yielded substantially high values for iron that still fall within the same concentration range as other archaeological finds from Finland and northern Sweden. The enrichment of iron on the object surface (Gauthier & Burke 2011: 278; Hughes et al. 2012: 787; Olausson et al. 2017: 106) could explain some of this variation, but as with calcium values, the provenance and potentially also the specific rock type can be put forth as additional explanatory factors. The find in the assemblage yielding the highest value for iron, for example, can be tentatively identified as jasper or jasperoid (Kinnunen 2008: 11) by its visual properties. High concentrations of iron also characterize some Silurian and Carboniferous chert deposits (Johanson et al. 2021: 127). For example, the reported maximum value for the Valdai region (Sinitsyna & Kolokol'tsev 2018: 451, table 5) is no less than 50,000. Such cherts are often opaque with a color palette ranging from black through brown to various hues of red (Dolukhanov et al. 2017: 68; see also Kinnunen et al. 1985: 19). Apart from these extremes, the concentration of both calcium and iron values fall below 5,000 ppm in the majority of the finds (Fig. 5) while a further cluster is formed by the finds with values not exceeding 1,200 ppm (Fig. 6).

DISCUSSION AND CONCLUSIONS

When the results are projected against the temporal framework offered by the two site clusters in the Hangaskangas area, the following observations emerge. First, all the specimens analyzed from the Oulu Hangaskangas E 1, which is purportedly the oldest of the sites examined, are tightly associated with the geological samples of the Valdai-Moscow region. This supports the previous observation by Matiskainen et al. (1989: 636–7), according to which cherts of eastern origin were predominant during the Neolithic period in Finland. The picture remains quite invariable regarding finds from the Muhos Halosentörmä site with some exceptions. The most notable of them is a scraper (Appendix 1: 11; Fig. 3) of jet black chert with a Ca/Fe-ratio comparable to geological sources of southern Sweden. The presence of an artifact made of Scandinavian chert is not by any means a surprise, as also other find classes at the Halosentörmä site, most notably ceramic crucibles (see Ikäheimo 2020), contain finds of this origin. The nearby Muhos Hangaskangas site with its finds falling into both main categories, eastern and western chert, is also indicative of a transition period with overlapping chert supplies.

The finds analyzed from the Oulu Hangaskangas E 2 site, on the other hand, suggest a definitive change in the supply chain. Approximately 80 % of the material analyzed is now clustered with the reference values from specimens belonging to southern Scandinavian or southern Baltic Cretaceous deposits, and the rest of the finds are only disputably of eastern origin. Moreover, the results published on chert finds from the Bronze Age dwelling-sites located in southwestern Finland (see Matiskainen et al. 1989: 827, Map 1, open circles) fall predominantly within the same cluster. A stray find tentatively identified as a strike-a-light (for Bronze age strike-a-lights, see e.g., van Ginj 2010), the only chert object found at the lowermost, and thus chronologically youngest site of Oulu Hangaskangas 8, completes the group.

In all, the results obtained by analyzing the finds from various sites located at the area of Hangaskangas seem to confirm the hypothesis formulated by Matiskainen et al. (1989: 636–7) by suggesting a transition from the use of eastern Carboniferous to western Cretaceous chert sources during the Bronze Age. Neither do the results necessarily contradict Costopoulos' idea about the stability of the trade networks in the early Bronze Age, but as it was based on the analysis of a solitary find from the Muhos Halosentörmä site, additional samples analyzed here indicate that by that time chert imports reached the eastern coast of the Bothnian Bay both from the east and the west.

The other research question concerning the effect of weathering and other post-depositional processes on chert finds from archaeological contexts can also be evaluated here by examining the clustering or dispersal of Ca/Fe-ratios measured from finds pertaining in all likelihood to the same geological source. The two examples that can be seen in Fig. 5 are both distinct groups

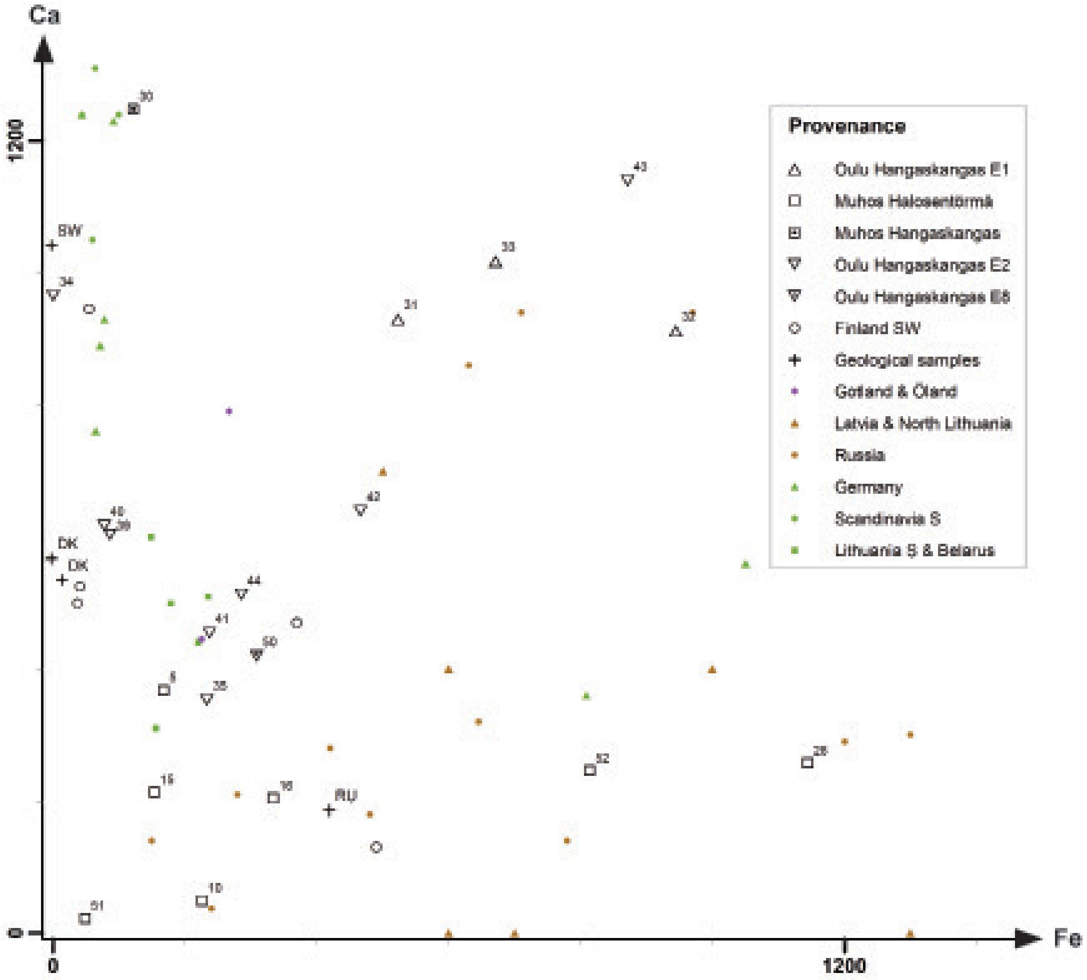

Figure 6. The Ca/Fe-ratio of samples in < 1200 ppm range with reference values from published geological deposits and Bronze Age finds from southwest Finland. Find chronology: up-pointing triangle= Late Neolithic, square= Early Bronze Age, down-pointing triangle= Late Bronze Age.

of small flakes, substantially uniform in size and thickness: one of jet black chert (Fig. 5: 19, 24 , 27) and the other of brownish-grey chert (Fig. 5: 6, 20, 21, 25). In both cases, rather than being dispersed, the results are clustered, although clustering is in both cases quite loose. While this confirms the observation about weathering as a factor increasing the chemical heterogeneity of the finds (see, e.g., Lundblad et al. 2011: 70), the clusters are still compact enough to form interpretatively significant patterns.

From this methodological point of view, these initial results suggest that calibrated and matrix-matched pXRF-analyses provide sufficiently precise information on the elemental composition of chert finds. By classifying them into meaningful groups and pairing them with chemical reference data obtained from known geological chert sources, a general provenance for archaeological chert finds may be tentatively assigned. These identifications can be strengthened by complementing the two-dimensional compositional data with analyses of other chemical elements known to be indicative in chert sourcing as well as more traditional observations regarding their macroscopic and microscopic characteristics such as color, texture, and possible microfossil content (see Lundblad et al. 2011; Olausson et al. 2017). Yet, to really be an applicable option for archaeological finds recovered from the podzol soil of the coniferous boreal zone, which is a famously geochemically harsh environment, increased attention should be paid to the immediate soil matrix from which such finds have been recovered.

Therefore, the documentation of the soil matrix should be carried out at archaeological excavations with a similar routine as one records today the precise location of every find. Because pedological conditions between the eluvial and illuvial layer in the podzol soil are starkly different, the position and orientation of a chert, or other find type, in soil is decisive for the extent and gravity of post-depositional processes it will be subjected to. For instance, as the orientation of the find in the soil can potentially impact the results of chemical analysis, logical considerations suggest that the up-facing side of a lithic fragment in the podzol soil matrix is more heavily weathered than the downfacing one. As modern survey equipment have customizable data collection interfaces, these variables are easy to record along with the positioning data on the field, while non-destructive analysis techniques such as X-ray diffraction and Raman spectrometry can provide further information about the weathering layer in laboratory conditions (Capel Ferrón et al. 2015).

In addition, the knowledge about raw material flows in the research area would clearly profit from long-lived and systematic research efforts focusing on provenance studies of chert and other siliceous rocks. For instance, due to its considerable mining potential for various metals and minerals, northern Finland continues to be the target of intensive geological surveys that produce indirectly a lot of relevant information for archaeological research. At the very least, this should promote the survey of domestic metacherts, the kind of which have constituted an important raw material resource in northern Norway from the Mesolithic period onwards (e.g., Hood 1992; Niemi 2019). The expansion in the range of geological comparison materials to be included in the investigative framework together with new samples from previously known sources and archaeological sites would likely lead into an augmented perception of prehistoric raw material flows. With the use of a non-invasive analysis method like pXRF, while simultaneously understanding its analytical limitations, the scope of research may easily be broadened to incorporate other chronological periods and geographical areas.

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APPENDIX

Appendix 1. Catalogue of analyzed finds.

#:	KM-cat. n:o	Item type	Munsell color[1]
Muhos Halosentörmä			
1.	17646:104	scraper	GLEY1 8/N
2.	17646:157	flake	GLEY1 8/N
3.	17646:15	flake	10R 3/4
4.	17646:164	scraper	7.5YR 8/1
5.	17646:167	flake	7.5YR 8/1
6.	17646:168	flake	10R 4/6
7.	17646:171	flake	2.5YR 6/1
8.	17646:31	scraper	10R 4/6
9.	17646:50	scraper	7.5YR 5.5/3
10.	17646:56	flake	10YR 7/1
11.	17646:8	scraper	GLEY2 4/10B
12.	17646:86	scraper	2.5Y 4/2
13.	17646:9	flake	GLEY2 4.5/10B
14.	17646:95	arrowhead	GLEY1 8/N
15.	30888:11	flake	GLEY1 5/10Y
16.	30888:15	flake	GLEY1 4/N
17.	30888:33	point frg.	GLEY1 7/N
18.	30888:56	flake	10YR 4/6
19.	30888:90b	flake	5YR 3/3
20.	32048:115	flake	3/N
21.	32048:136	flake	7.5YR 5/6
22.	32048:1478	flake	10YR 4/4
23.	32048:167	scraper	10YR 5/6
24.	32048:176	flake	7.5YR 4/7
25.	32048:209	flake	GLEY1 2.5/N
26.	32048:262	flake	7.5YR 5/6
27.	32048:292	flake	2.5YR 4/6
28.	32048:387	flake	GLEY1 2.5/N
29.	32048:894	cutter	2.5YR 4/4
Muhos Halosentörmä			
30.	32171:23	cutter	10R 4/3
31.	32171:24	scraper	7.5YR 7/1
Oulu Hangaskangas E1			
32.	39158:1016	flake	GLEY1 4/N
33.	39158:1018	flake	2.5YR 6/2
34.	39158:1019	flake	2.5YR 6/2
Oulu Hangaskangas.E2			
35.	39158:114	flake	5YR 5/1
36.	39158:115	flake	10R 5/3
37.	39158:120	flake	GLEY1 8/N
38.	39158:121	scraper	10R 3/3
39.	39158:122	flake	2.5YR 3/2
40.	39158:124a	strike-a-l.	GLEY1 5/10YR
41.	39158:124b	strike-a-l.	GLEY1 3/N
42.	39158:125	flake	GLEY1 6/N
43.	39158:126	flake	GLEY1 5/10Y
44.	39158:128	scraper	2.5Y 4/1
45.	39158:134	flake	2.5YR 4/6
46.	39158:135a	flake	GLEY1 6/10Y
47.	39158:135b	flake	7.5YR 5/6
48.	39158:138	flake	10YR 7/1
49.	39158:139	flake	10R 3/4
Oulu Hangaskangas E3			
50.	39158:995	strike-a-l.	GLEY1 7/N
Muhos Halosentörmä			
51.	39187:19	flake	10YR 4/2
52.	39187:24	flake	GLEY1 2.5/N

[1] Munsell soil color charts 2000: washable edition