

Provenance of archaeological wool textiles: new case studies

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Abstract

In the last two decades, measurements of strontium (Sr) isotopes in archaeological bone tissues/skeletons have shown to be an effective technique for the characterisation of human and animal mobility in prehistory. More recently, this tracing system is also being applied to the investigation of archaeological textile's provenance. The importance of ancient textiles has been often underestimated, however research of archaeological textiles is currently experiencing an extremely increasing interest as the development of new methodologies, conducting experimental studies and launching of new projects are providing an unreached amount of new information, knowledge and impressive data sets which together build the basis of novel thinking and interpretations. This manuscript aims at summarising two of the most recently developed methods that focus on the extraction of Sr from ancient non-dyed and organic-dyed wool threads from archaeological textiles in an attempt to identify if the raw materials are local or non-local to the sites. In particular, this study presents two case studies which rely on the use of these chemical protocols. The first example deals with a wool/fur sample from a modern Greenlandic Musk ox. The purpose of this study is to characterise wool from an exotic animal on the one side, and to try to establish a link between this wool and a geologically-seen ancient and very *special* terrain (Archaean basement rocks from the Kangerlussuaq area of Western Greenland) on which this musk ox was grazing. Our interest was focused on whether the bio-available Sr fraction from this terrain impacted on the composition of the wool from the animal. The second case study deals with three thread samples from four ancient wool textile pieces recovered from one and the same pre-Roman Iron Age peat bog site at Krogs Mølle (Denmark). Some of these textiles have proven to be dyed with organic dyestuffs. This study therefore aimed at applying a novel pre-cleaning methodology developed for dyed (by organic dyestuffs) wool threads from ancient textiles. The outcome of these two particular studies revealed both the potential of these novel methodologies for retrieving the original Sr isotope signature of the raw material wool, and their limitations.

Introduction

Ericson (1985) was the first who proposed the idea of using the variations of the strontium (Sr) isotopic ratios in archaeological materials and to relate these to the bedrock and soil characteristics of specific geological areas and thus to constrain their potential geographical origin. Since then, many studies of human and animal migration have been conducted on the base of archaeological bone tissue/skeletons (Evans *et al.*, 2006; Grupe *et al.*, 1997; Knudson *et al.*, 2005; Montgomery *et al.*, 2003; Price *et al.*, 1998, 2010, 2011). The base for performing such tracing studies lies in the fact that Sr isotopic ratios ($^{87}\text{Sr}/^{86}\text{Sr}$) do not change within their pathway throughout the food chain (Graustein, 1989). Furthermore, the age and the type of lithology of the bedrocks (magmatic, metamorphic and sedimentary rocks) on which respective soils are developed impose a control on the $^{87}\text{Sr}/^{86}\text{Sr}$ ratios of a particular geological area and thereby creating necessary variations in this signature which are measurable and traceable. However, studies have shown that the knowledge of the local bedrock is not always sufficient; hence there is the need to characterise the so-called bio-available Sr isotope composition of the targeted area. There are several ways to characterise such a bio-available signature: by bone tissue of small animals, by plants, by water and/or soil samples (Evans *et al.*, 2010; Frei and Frei, 2011; Price *et al.*, 2002). However, it should be noted that the spatial resolution of the bio-available $^{87}\text{Sr}/^{86}\text{Sr}$ ratios can be similar in different geographical areas, therefore setting limits to the discrimination between different areas with the same or very similar bio-available Sr characteristics. Several book chapters and review articles have recently been written on the subject of the application of Sr isotopes to study human and animal migration, thus this manuscript will concentrate on the novel methods developed for applying this tracing system to ancient wool and refers to the following articles in which more information regarding the parameters controlling the Sr isotopic system are contained (Bentley, 2006; Montgomery, 2010; Price and Burton, 2011; Price *et al.*, 2002).

Wool textiles, and hence the raw material *wool*, are often regarded as rare finds in archaeological contexts. Nevertheless, there are a number of notable exceptions. For example, in northern Europe, and most specifically in Denmark, there is an assemblage of extremely well preserved pre-Roman Iron Age textile finds recovered from peat bogs and contained in oak coffins from Bronze Age graves. These finds comprise a rich variety of garments (Andersson Strand *et al.*, 2010; Bender Jørgensen, 1986; Broholm and Hald, 1940;

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Brøndsted, 1960). In case of the peat bog finds, these textiles have been preserved due to the acidic conditions present in their burial contexts. The importance of ancient textiles, such as garments, blankets, floor-mats or even sails, has been somewhat underestimated within the archaeological world (Gleba, 2008). However, textile research is today a growing field within archaeology, proving to be an important source of information for prehistoric societies. Moreover, textile production is a craft that needs meticulous planning and the crafts people must have possessed many different skills and *a priori* know-how. In Northern and Central Europe, the Mediterranean and the Near East the most common fibre materials were animal fibres from sheep and goats as well as plant fibres from flax, hemp and nettle

(Barber, 1991). Despite the increasing interest in archaeological textiles, one of the most important questions, namely the one of provenance, remains often unclear, as only comparative studies were possible. However, there have been a few attempts to fill this gap both for plant and animal fibres (Benson *et al.*, 2006; Frei, 2009; Frei *et al.*, 2009b; Von Carnap-Bornheim *et al.*, 2007).

Case Studies

Modern unspun wool fibers from Greenlandic Musk ox were collected during the summer 2009 in the western Greenlandic town of Kangerlussuaq (Sønderstrømfjord) in an area close to the inland ice sheet. Most of the native population of Musk ox in Greenland originally stems from the northeastern part of Greenland, and individual animals were moved to other parts of Greenland later on, so to the area around Kangerlussuaq. Musk ox feed on willow and various grasses. Musk ox fur consists of two layers, an inside part composed of fleece (which is the actual wool) and an outer part which consists of extremely long hair. The fleece naturally falls during the summer period, where people usually collect it to produce a very soft yarn. The sample analysed in this study was collected in a similar way. The wool was neither treated nor cleaned in any way prior to the pre-cleaning methods described in the analytical techniques section. The wool had a natural brownish pigmentation colour.

The ancient samples consisted of four yarn pieces all unearthed from the same Danish peat bog near Krogens Mølle and belong to the Danish pre-Roman Iron Age (500 BC-AD 1; National Museum of Denmark nr D 1310). The research leading to these results has received fundings from the Danish National Research Foundation D NRF 64. Recently some of these textiles have been ^{14}C dated to 399-207 BC (cal) (Mannering *et al.*, 2010). Textiles and skin fragments were discovered in the Krogens Mølle peat bog site located on the northern part of Jutland in 1878. The skins and textiles were found together with some skeletal remains, identified as female (Hald, 1980). The textiles consist of several woven pieces all made of wool. The three textiles specified below were chosen for this study and small thread samples (less than 20 mg) from each was removed from the respective woven textile pieces.

Sample D 1310 A was collected from a torn off strip with dimensions 40×32 cm. The yarn from which this textile piece is made of is fairly finely spun and woven as a tabby with a checkered pattern. The wool has been identified as sheep wool. From this piece two samples were investigated, one thread from the

darker wool and one from the lighter wool.

Sample D 1310 D was collected from a badly damaged textile piece with dimensions 84×43 cm which appears to originally have been part of a bag. It is also made of sheep wool.

Sample D 1310 J was separated from an irregular strip with a tubular woven selvedge and with dimensions 50×60 cm. Some of the loose ends of this textile piece are tied in knots, apparently done in antiquity.

Recent dye analyses [high performance liquid chromatography (HPLC)] conducted on a wool thread samples belonging to one of the textiles of the Krogens Mølle find (Vanden Berghe *et al.*, 2009) showed that they had been dyed in Antiquity with luteolin-containing-dyestuff. Ancient textiles that can be shown to contain organic dyestuff, by *e.g.* HPLC analyses, need to be specially treated in order to ensure the recovery of the true wool's nutritional information. It is tenable to believe that the threads studied herein from this location were also dyed with an organic dyestuff. Therefore, in this study, we introduced an additional extra pre cleaning step of the sample before its final dissolution, particularly aimed at recovering a dye-free fraction of Sr from the fibers which then closely approximates the composition of the true bio-available Sr. Therefore, this additional step allows for a dyestuff decontamination of the sample material prior to ion chromatography procedures.

This is important as dyestuff has been shown to potentially mask/contaminate the Sr isotopic signature of a textile's raw material (Frei *et al.*, 2010). The organic dye-removal step has shown to be able to remove up to 100% of organic dyestuff without the use of mordants, and up to 99.5% when mordants have been used.

Materials and Methods

Pre-cleaning/decontamination

The unspun wool sample from the Musk ox was washed in 20% dilute cold hydrofluoric (HF) acid under ultrasonic treatment for 1 h in a 7 mL Teflon beaker (Savillex™; Savillex, Eden Prairie, MN, USA) prior to dissolution.

The hydrofluoric acid wash was subsequently pipetted off from the actual wool sample.

The wool sample was deeply rinsed (several times) with 1 mL of deionised water (MilliQ™; Millipore, Billerica, MA, USA) and dried down.

A 1N hydrochloric acid (HCl) wash followed the HF treatment step.

The remaining wool sample was again deeply rinsed (several times) with 1 mL of deionised water (MilliQ™; Millipore) and finally dried down.

Two extra pre-cleaning steps should follow the HF-pre-cleaning step for organic

dyed wool.

The rinsed wool sample was emerged in 3 mL of 0.2 M ammonium peroxodisulfate ($(\text{NH}_4)_2 \text{S}_2\text{O}_8$ [a strong oxidant (APDS)] on a hotplate (preferentially at 130°C) for *ca.* 15-30 min, depending on the degradation of the sample.

The residual wool sample is then deeply rinsed with 1 mL of deionised water (MilliQ™; Millipore) several times and subsequently dried down.

Dissolution and ion chromatographic procedures

In order to be able to keep track of the Sr mass budget during the different pre-cleaning steps applied to the samples, a ^{84}Sr enriched (94%) spike was added to the respective leachates and to the finally dissolved sample. This provides a measure to also control the concentration of Sr in the pre-cleaned wool residue, which should be in the range of 0.05-15 ppm usually characteristic of natural animal hair.

The residual wool fraction is dissolved in a 1:1 mixture of 30% HNO_3 (Seastar, Jacksonville, FL, USA) and 30% H_2O_2 (Seastar). The samples tend to decompose within 30 minutes. After decomposition the solution is dried down on a hotplate at *ca.* 80°C .

Samples are then taken up in a few drops of 3N HNO_3 and loaded on either glass extraction columns or on disposable pipette-tip columns containing 0.2 mL, intensively pre-cleaned SrSpec™ (Eichrome Inc., Lisle, IL, USA), mesh 50-100 resin. The elution recipe essentially follows that of Horwitz *et al.* (1992).

The Sr fraction is eluted by ultra pure deionised water and subsequently dried down on a hotplate. Organic compounds are often retained on the resin, causing a brownish staining of the originally white resin.

Thermal ionisation mass spectrometry

The samples are dissolved in 2.5 μL of a $\text{Ta}_2\text{O}_5\text{-H}_3\text{PO}_4\text{-HF}$ activator. The samples are then loaded onto outgassed 99.98% single rhenium filaments. Samples are measured in so-called dynamic mode on a VG 54 Sector IT mass spectrometer, at temperatures between 1300 and 1450°C , and keeping the ^{88}Sr signal at least at 300 mV over at minimum 6 blocks with 10 scans each.

Results and Discussion

The study of textiles probably goes as far back as to the first Egyptian antiquarians (Good, 2001). Today, archaeological textiles and textile production in Antiquity can be

investigated by several means, e.g. by comparative studies, textile tools, written sources, fiber counting/recognition techniques, ^{14}C dating techniques, context analyses, dye analyses, visual quality analyses, weaving techniques, and function analyses. However, one of the most important questions remained unanswered: Where did these textiles, particularly the raw materials they were made of, originate? Until recently, this question was not possible to answer, but the development of several new methodologies designed to fill this gap, can potentially provide new important information regarding the provenance of an ancient textile's raw material. These methodologies apply the Sr isotope tracing system as an archeometrical tool and include deep pre-cleaning procedures to ensure the removal of contaminants (Frei *et al.*, 2009a, 2010). This contribution aims at presenting the results of such methodological approaches using $^{87}\text{Sr}/^{86}\text{Sr}$ ratios for an attempt to define the provenance of textiles.

One of the important differences between analyzing Sr isotopic ratios in e.g. tooth enamel and hair (wool) lies in the difference in their Sr concentration (Kohn *et al.*, 1999), as Sr levels are typically low in non-skeletal tissues. Concentrations of Sr in teeth enamel and bone tissues range often between ca. 50-1000 ppm [Bentley (2006) and references therein]. On the other hand, hair has much lower Sr concentrations of ca. 0.05-15 ppm (Frei *et al.*, 2009a). Consequently, the pre-cleaning protocol, the ion chromatographic separation of the

Sr and the final mass spectrometrical analyses are more difficult because they create generally a greater methodological and analytical challenge than those for tooth enamel and bone tissues. Thus, the aim is to recover the bio-available $^{87}\text{Sr}/^{86}\text{Sr}$ ratios of the textiles fibers in order to potentially pinpoint possible areas of origin for the textiles' raw material. Moreover, the protocols have been developed with the perspective of consuming as little as possible of the material. The protocols are now routinely applied to samples in the order of only 10-20 mg.

The first case study, the unspun wool sample of modern Greenlandic Musk ox, is expected to recover relatively elevated $^{87}\text{Sr}/^{86}\text{Sr}$ ratios of the cleaned material, as the feeding grounds are located on very old rocks of an Archaean terrain in Western Greenland. This area is predominantly composed of Archaean gneisses and granitoids (Figure 1) therefore characterised by high $^{87}\text{Sr}/^{86}\text{Sr}$ ratios. As the unspun wool fibers had not been dyed, the decontamination procedure followed was the one for undyed wool (Frei *et al.*, 2009a).

Samples from the second case study, the archaeological textiles from the pre-Roman Krogens Mølle site were chosen because previous studies of sample material from this site have shown that at least three other samples had been dyed in antiquity with luteolin and/or indigotin dyestuffs (Vanden Berghe *et al.*, 2009). The samples studied herein were expected to be dyed as well and the pre-cleaning procedure therefore followed the one

described above for wool organic-dyed textiles (Frei *et al.*, 2010).

A total of 9 $^{87}\text{Sr}/^{86}\text{Sr}$ ratios (leachates/fractions recovered from the pre-treatment steps included) from the five samples studied herein are presented in Table 1. The $^{87}\text{Sr}/^{86}\text{Sr}$ ratios from the unspun wool sample of the Greenlandic Musk ox yielded the highest $^{87}\text{Sr}/^{86}\text{Sr}$ ratio of all the five samples analysed herein. The Musk ox residual wool has a $^{87}\text{Sr}/^{86}\text{Sr}$ ratio of 0.71419, a ratio which resulted after the pre-cleaning methods were applied. The pre-cleaning removed a considerable amount of Sr from mass budget calculations it can be seen that a relative amount of 118 ppm from a total relative concentration of ~140 ppm (sum of Sr from HF and HCl leachates, and the residual material) was leached away during the decontamination procedures. These calculations do not consider the fraction of Sr which was potentially removed by the deionised water (MilliQ™) rinsing steps interspersed after the acid leaching steps. This implies that the concentration of Sr in the final residue is a maximum concentration. The elevated Sr isotope value of the residual fraction of the Musk ox wool fibers seems only partially to reflect the radiogenic Sr of the Archaean rocks exposed in the Kangerlussuaq area. Even higher values ($^{87}\text{Sr}/^{86}\text{Sr} > \sim 0.75$) could be expected in such geologically ancient areas, but the proximity of the grazing area to the fjord shore, and thus to seawater, enabled the potential contamination of the soils by seawater-spray, and thus by relatively unradiogenic Sr

Table 1. Strontium concentrations and isotope ratios of wool.

Sample description	Treatment	Weight (mg)	Sr (ppm)	$^{87}\text{Sr}/^{86}\text{Sr}$	2 SE (abs)
Greenland Musk ox	HF leach	77.35	93.58	0.71382	0.00003
Greenland Musk ox	HCl leach	77.35	25.09	0.71389	0.00003
Greenland Musk ox	Residue (wool)	77.35	18.50	0.71419	0.00006
Krogens Mølle D 1310 A (dark yarn)	HF leach	11.93	19.47	0.70896	0.00006
Krogens Mølle D 1310 A (dark yarn)	HCl leach	11.93	292.67	0.70863	0.00004
Krogens Mølle D 1310 A (dark yarn)	Ammonium peroxodisulfate	11.93	20.09	0.70894	0.00004
Krogens Mølle D 1310 A (dark yarn)	Residue (wool)	11.93	0.23	0.71030	0.00007
Krogens Mølle D 1310 A (light yarn)	HF leach	5.50	1.46	0.70946	0.00002
Krogens Mølle D 1310 A (light yarn)	HCl leach	5.50	1.47	0.70988	0.00004
Krogens Mølle D 1310 A (light yarn)	Ammonium peroxodisulfate	5.50	1.44	0.70946	0.00002
Krogens Mølle D 1310 A (light yarn)	Residue (wool)	5.50	0.03	0.71195	0.00003
Krogens Mølle D 1310 D	HF leach	14.25	13.78	0.70998	0.00004
Krogens Mølle D 1310 D	HCl leach	14.25	9.07	0.71121	0.00005
Krogens Mølle D 1310 D	Ammonium peroxodisulfate	14.25	7.29	0.70920	0.00005
Krogens Mølle D 1310 D	Residue (wool)	14.25	2.07	0.71013	0.00005
Krogens Mølle D 1310 J	HF leach	13.29	22.85	0.70822	0.00006
Krogens Mølle D 1310 J	HCl leach	13.29	197.58	0.70816	0.00004
Krogens Mølle D 1310 J	Ammonium peroxodisulfate	13.29	37.82	0.70811	0.00004
Krogens Mølle D 1310 J	Residue (wool)	13.29	3.44	0.70878	0.00005

SE, Standar error; HF leach, hydrofluoric leach; HCl acid leach, hydrochloric acid leach.

with a $^{87}\text{Sr}/^{86}\text{Sr}$ ratio of ~ 0.7092 . Such seawater-spray contamination has been recognised and discussed in previous studies (Frei *et al.*, 2009a; Price and Gestsdottir, 2006). It seems likely therefore that the composition of bio-available Sr in the grazing area of the Musk ox is substantially depressed/lowered by seawater spray contamination.

Results of the four Krogens Mølle samples studied herein are also presented in Table 1. Two samples belong to the same textile piece, D 1310 A. One sample was taken from the darker yarn and one from the lighter yarn, with the purpose of investigating potential differences in the Sr isotope composition of the two yarns. The darker yarn yielded a residual $^{87}\text{Sr}/^{86}\text{Sr}$ ratio of 0.71030 which can be considered as local to the site if one uses the baseline established for Denmark which constrains local composition to a range between $^{87}\text{Sr}/^{86}\text{Sr} = 0.708\text{--}0.711$ (Frei and Frei, 2011; Frei and Price, 2012). On the other hand, the lighter wool yarn yielded a more radiogenic $^{87}\text{Sr}/^{86}\text{Sr}$ ratio of 0.71195. Using the same dis-

crimination as above, this yarn sample seems to indicate a non-local provenance as its Sr isotope value falls outside the range typical of Danish bio-available Sr. Bornholm excluded. Similar recent investigations have shown that another well preserved pre-Roman Iron Age textile, the Huldremose II find, from another peat bog site in Jutland, is composed of yarn from both local and non-local provenance (Frei *et al.*, 2009b).

The other two yarn samples from two different textile pieces, D 1310 D and D 1310 J, have $^{87}\text{Sr}/^{86}\text{Sr}$ ratios of 0.71013 and 0.70878 respectively. Thus, both Sr isotopic values fall within the isotopic range of bio-available Sr from Denmark established by (Frei and Frei, 2011; Frei and Price, 2012) and thus can be considered as local. Nevertheless, the significant difference in their Sr isotopic composition implies that they do not seem to come from the same area. Interestingly, the yarn sample D 1310 J, which has the lowest Sr isotope ratio reported in this study, could come from a site to south of the Krogens Mølle area, where sim-

ilar Sr isotope values of surface water have been reported (Frei and Frei, 2011). In general, the Sr isotopic values measured on the four yarn samples of wool textiles from the Krogens Mølle site seem to point to different, but distinctive localities. Two of the localities seem to be local and constrained to Jutland (Denmark), the third one seems to be non-local to Denmark (Bornholm excluded). The two yarns with significantly different, but still Danish signatures could potentially be explained by differences in the Jutlandic bio-available signatures. The less radiogenic $^{87}\text{Sr}/^{86}\text{Sr}$ ratio of 0.70878 for sample D 1310 J could potentially indicate an area south of the Krogens Mølle site and in the vicinity of the Limfjord, where Eocene ash layers apparently have the effect of lowering of the bio-available signal of Sr (Frei and Frei, 2011). It is however, too premature to elaborate on the provenance of the textile D 1310 A, which has a non-local (non-Danish) signature. One possibility would be to deduce such values from the southernmost part of Sweden (Skåne) or from some regions of northern Germany, as similar values have been reported for mineral waters from these regions (Voerkelius *et al.*, 2010).

Conclusions

The study presented herein shows the importance of removing dust, and dyestuff of ancient wool textiles prior Sr isotope analyses for provenance investigations. Modern unspun wool from Musk ox that grassed in an area dominated by Archaean gneisses was expected to show a strongly radiogenic $^{87}\text{Sr}/^{86}\text{Sr}$ composition that would mirror the ancientness of the terrain. Our analyses showed that this wool has indeed an elevated $^{87}\text{Sr}/^{86}\text{Sr}$ ratio of ~ 0.714 , but lowering of this ratio by on-site contamination of the grazing area by seawater-spray might explain a depression of the bio-available fraction of the soil in this area, which then translates into the food chain of the Musk ox.

The four ancient wool yarn samples belonging to textiles from a pre-Roman Iron Age site in the northern part of the Danish peninsula of Jutland, yielded significantly lower values than the one recovered for the Musk ox wool. Three samples seem to be local to Jutland, but probably from two different sites; one near the Krogens Mølle area (two threads) and one from an area more towards the south, where less radiogenic Sr isotope values of bioavailable fractions have been reported and mapped. The last textile sample falls out of the Sr isotopic range characterising bio-available compositions typical of Denmark (Bornholm excluded) and thus can be interpreted as non-local.

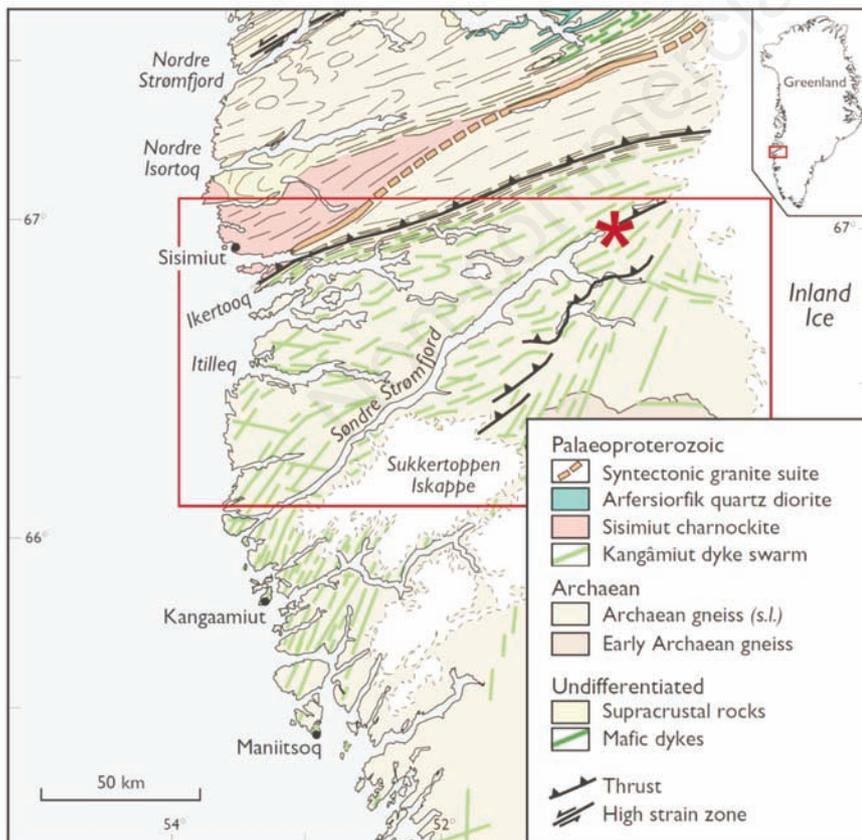


Figure 1. Geological map covering the region of Kangerlussuaq/Søndrestrømfjord in southwest Greenland, modified after Jensen *et al.* (2002). The red star shows the location where the wool sample was collected, situated near Kangerlussuaq.

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