ИСПОЛЬЗОВАНИЕ ЕСТЕСТВЕННО-НАУЧНЫХ МЕТОДОВ В АРХЕОЛОГИЧЕСКИХ ИССЛЕДОВАНИЯХ

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ANALYSIS OF A LACQUER CUP FROM THE XIONGNU GRAVE IN TAMIR VALLEY, MONGOLIA*

The Xiongnu Empire (3rd century BCE to the 1st century CE) controlled the Central Asia regions of the Silk Road; the economic and cultural relations with Han are evidenced by the availability of Chinese lacquered objects in the Xiongnu burials in Mongolia. This work focuses on the characterization of materials from fragmented pieces of a lacquer cup excavated in Tamir Valley, Arkhangai province (Central Mongolia), which dates to the Xiongnu period. Multi-analytical spectroscopic techniques were performed, including scanning electron microscopy by X-Ray energy dispersive spectroscopy and FTIR spectroscopy. The results show that the lacquer cup fragments consist mainly of a three-layer structure, which include a lacquer pigment layer, a fiber layer and a wood foundation layer. The lacquer ground layer under study is a mixture of organic materials including urushi, possibly burned ashes and inorganic fillers such as quartz and feldspar. Further, this work will focus on comparing the lacquer ware excavated in different places and from the same period in order to provide information for further studies such as the chronological distribution of lacquerwares in Mongolia.

Key words: lacquerware, Xiongnu, FTIR, SEM-EDX.

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Introduction

Lacquerware was an important invention of ancient Asia. The invention of the raw lacquer coating quickly drew attention with its use as both as a protective layer and for its decorative properties [Wang et al., 2017, p. 105–120]. Lacquer work is today recognized as one of the centrally distinctive components of Han material culture (206 BCE – 220 CE) and the period 206 BC – 8 AD was the apogee of Han Chinese lacquer art [Louis, 2006, p. 48–53]. It is remarkable, that Han lacquers have been found not only in Chinese burials, but in areas far beyond the ancient Han frontier, as far north as Lake Baikal and as far west as Bergam in Afganistan [Louis, 2006, p. 48–53]; these are explained in terms of economic relations and trade along the Silk Road. The confirmations of that are the Han Dynasty Chinese lacquerware cups discovered in the Xiongnu cemeteries between Ulaanbaatar and Lake Baikal [Miniaev, 1998; Torbat, Erdenebat, Seveendorzh, 2003], but also in Altai [Tishkin, Khavrin, Novikiva, 2008]. But the most abundant evidence came from the P.K. Kozlov's expedition of 1924–1925, which discovered the Xiongnu elite burials at Noin Ula and by the Russian-Mongolian expedition in 2009–2012 [Tseveendorj et al., 2010, p. 255–273; Polos'mak et al., 2013, p. 102–118]).

An important part of earlier research focused on the study of inscriptions, since the interpretation of the Chinese characters carved on the bottom of the lacquer cups helped to identify the workshop where it was restored or improved. Based on the inscriptions, the Xiongnu cemetery at Tsaraam complex in Transbaikalia date to the period between 8 BCE and 4 CE [Pirazzoli-t'Serstevens, 2007, p. 56–58]. The lacquerware from the Gol Mod I site in Khairkhan

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sum, Arkhangai (Mongolia) was made in the Gonggong imperial workshop in 16 BCE [Yerul-Erdene and Otani, 2015, p. 104–108]. In Noin Ula, Tomb 6 was dated with regard to the inscription on the lacquer bowl, which was produced in the 2nd year BC, providing a *terminus post quem* [Pirazzoli-t'Serstevens, 2007, p. 56–58], while the lacquerware from the tomb 20 of Noin Ula date to the first century AD [Chistyakova, 2011, p. 83–89].

In addition, based on the independent age for the burial site provided through the lacquered Chinese cups inscribed with Chinese characters, which date back to the fourth year of the Yuanyan Era, i.e., ninth year BCE [Polos'mak et al., 2013, p. 102–118]. First luminescence measurements were carried out on quartz single grains from clay sediment and feldspar fine grains from potteries, and the luminescence results were in agreement with independent age [Solongo et al., 2017] for the Xiongnu burial site at Noin Ula.

Many scholars have made substantial achievements in researching the excavated lacquers, and more attention has been drawn to the lacquer production techniques. The chemical composition and structure of the coatings of the unique Han Dynasty Chinese lacquerware eared cups in the collection of the State Hermitage Museum, Russia were investigated by optical microscopy, cross-section, FTIR and X-ray analysis [Elikhina, Novikova, Khavrin, 2014, p. 93–107]. A multi-analytical investigation was also carried out on the lacquer objects from Noin-Ula excavated by the Mongolian-Russian expeditions that identified the components of the drying oil and urushiol [Karpova et al., 2017, p. 336–344]. Archaeological lacquer that was collected by the Mission Arch´eologique Française en Mongolie (MAFM) during the excavation of the tomb T20 of the Xiongnu necropolis at Gol Mod, Central Mongolia (dated between the 1st century BC and the 1st century AD), was studied by [Le Hô et al., 2013, p. 5685–5685]. In addition, the lacquerwares collected from burials in Transbaikalia, e.g. Ivolga and Altai were studied by [Novikova, Marsadolov, Tishkin, 2018, p. 106–143] using FTIR and TGA methods to characterize the materials used for manufacturing and decoration.

This study investigates the most recent Han lacquer artifacts found from a number of Xiongnu cemeteries in the Tamir River Valley in Mongolia [Turbat, 2004, p. 186–196]. SEM-EDX and FTIR measurements were applied on the multilayered lacquerware fragments with the intention to answer questions such as: What is each layer made of? What are the minerals that make the red and black pigment? How different is this lacquer cup to those earlier findings e.g. at the Xiongnu burials at Noin Ula?

Materials and Methods

Archaeological site and samples. Tamir's Ulaan Khoshu cemetery is located 450 km west of Ulaanbaatar, north of the Tamir River, 10 km west of Orkhon and Tamir confluence; these Xiongnu monuments are among the typical for the social middle layer, but they are extraordinary to other Mongolian monuments [Torbat Ts., 2003]. First of all, in contrast to the fact that most Xiongnu burials are directed to the north, these were directed to the left. The grave goods such as horses and other animals are rare, but also waeponry does not play a significant role among the grave goods. However, there is an unusual high amount of ceramics and vessels; among them ten pottery vessels of every form, iron vessel, iron chests and bronze seals. Chinese lacquer often appears as inventory at this site, as around 14 lacquerware fragments have been found in burials №24, 34, 36, 39 and 40, excavated in 2017 by the Mongolian-French joint archaeogical team. The lacquercup TUK2017, T24 is shown in Fig.1; a kind of "cloud" ornament is typical for the Han style [Elikhina, Novikova, Khavrin, 2014, p. 93–107]. The object was in poor condition at the time of excavation (Fig. 1) and taken for a technical study at the National Center of Cultural Heritage of Mongolia.

Figure 1. Lacquercup TUK2017, T24, L8, N18 found at Tamir Valley, Arkhangai aimag, in 2017

Figure 2. The fragments of lacquer cup:
(a) the lacquer pigmented layer; (b) black fibre layer; and (c) wood foundation layer

Analytical methods. A detailed physical and chemical characterization of the lacquerware has been widely used by recent researchers [Elikhina, Novikova, Khavrin, 2014, p. 93–107; Karpova et al., 2017, p. 336–344; Hao et al., 2017, p. 8446; Novikova et al., 2018, p. 106–143], and focused on the production process and the technique of the ground layer, the lacquer layer and the pigmented layers by the use of multiple analytical methods such as Fourier Transform Infrared (FTIR) Spectroscopy, and Scanning Electron Microscopy by X-Ray Energy Dispersive Spectroscopy (SEM-EDS).

Analysis by FTIR. Infrared analysis has been used to provide a unique spectral pattern of organic and inorganic functional groups present in urushi-oil lacquer components. Three different parts of the lacquer cup were analyzed individually by infrared spectroscopy using an Fourier Transform Infrared Spectrometer, model Shimadzu Prestige-21 with a heat stabilized DLATGS, InGaAs detector in the spectral range of 350–7800 cm^{-1}. The spectra were collected at 128 scans and a resolution of 2 cm^{-1}. Samples were hand pressed and prepared by mixing the sample with KBr at a ratio of 1:100.

Analysis by SEM-EDS. Scanning electron microscope (pSEM) and energy-dispersive X-ray spectroscopy (EDS) measurements were completed using a Hitachi TM-3000 (Hitachi High-Technologies Corporation, Japan) at the Mongolian University of Science and Technology.

Results and discussion

In the following we characterize the lacquer fragments TUK2017, T24 from the Tamir Valley (Arkhangai, Mongolia) and compare them to the aforementioned lacquer samples.

FTIR spectra. FTIR spectra of the lacquer pigmented layer, fibre layer and wood foundation layers are displayed in Fig.3. The main features seen in the three FTIR spectra are almost identical, FTIR analysis identified the characteristic absorption bands for urushiol at 3440 cm^{-1}, 2930 cm^{-1}, 2859 cm^{-1}, 1710 cm^{-1}, 1688 cm^{-1}, 1636 cm^{-1}, 1559 cm^{-1}, 1420 cm^{-1}, and 1030 cm^{-1} which are in good accordance with the infrared spectra of Chinese lacquers [Derrick et al., 1988] and of the Mongolian archaeological lacquerware chariot from the Gol Mod site [Le Hô et al., 2013, p. 5685–5685], whereas the spectrum for a dried film of raw urushi (from the IRUG database) showed certain spectral differences. It is known that a specific peak at 3440 cm^{-1} is due to O-H stretching, and the peaks near 2930 and 2859 cm^{-1} are due to C-H stretching; the absorption peak at 1030 cm^{-1}, along with the absorbance at 776, 785 and 694 cm^{-1} suggested the existence of the Si-O-Si bond [Saran et al., 2016, p. 143–149], indicating the existence of quartz in all three fragments. The bands at 3620 and 3698 cm^{-1} are due to clay, indicating the existence of quartz sand that may have been used as an additive in the manufacturing process. This is also in accordance with the previous results; e.g. in the lacquerwares from burials in Noin Ula [Elikhina, Novikova, Khavrin, 2014, p. 93–107; Karpova et al., 2017, p. 336–344] the absorption bands at 1031, 801, 783, 670, 524 and 467 cm^{-1} were identified and assigned them to the silicates.

As can be seen from the spectra, in the lacquer-pigmented layer, the silicate absorption bands dominate, while the highest amount of organics is present in the black fiber layer, where the absorption bands at 1570 cm^{-1} and 1420 cm^{-1} corresponding to the vibration of carboxylates of calcium soaps [Karpova et al., 2017, p. 336–344] are becoming dominant. One or more binding media, such as drying oil, animal gelatin, or some inorganic minerals, are usually added to the lacquer film and the lacquer ash [Hao et al., 2017, p. 8446]. The additives mixed into the lacquer not only protect and polish the lacquer surface, but also improve the process of coloring the lacquer with various pigments.

It is worth noting, that the FTIR results demonstrate similar compositions of the lacquercup TUK2017,T24 with the spectra of lacquercup 44-31 (lacquer cup from burial 31 Noin Ula), investigated by [Karpova et al., 2017, p. 336–344]. Similarities were also found to the lacquercup MR-2304, studied by J. Elikhina, O. Novikova, S. Khavrin [2014]. First of all, the silicate absorption bands are dominant; furthermore, the absorption bands at 1653, 1453, 1034, 604 and 564 cm^{-1} which are assigned to hydroxyapatite [Karpova et al., 2017, p. 336–344], that are dominant in their samples 77–22 and 44–31, are also pronounced in our sample TUK2017,T24.

J. Elikhina, O. Novikova, S. Khavrin [2014] reported that in the composition of some coatings small amounts of tung oil (the so-called "wooden" oil) were observed, as the absorption band at 712cm^{-1} is detected in the spectra. It is worth mentioning that tung oil was absent in the lacquer coatings from Ivolga and Altai region [Novikova, Marsadolov, Tishkin, 2018, p. 106–143], which excluded the possibility of adding tung oil to those lacquer wares. FTIR analysis provided evidence for the presence of low levels of tung oil in the lacquercup TUK2017, similar to Noin Ula's lacquercup 44-31, and MR-2304, investigated by [Karpova et al., 2017, p. 336–344] and [Elikhina, Novikova, Khavrin, 2014, p. 93–107], respectively.

Even if the durability of lacquer has been demonstrated in Asia with a few well preserved archaeological remains, lacquer objects under study suffered from alteration under

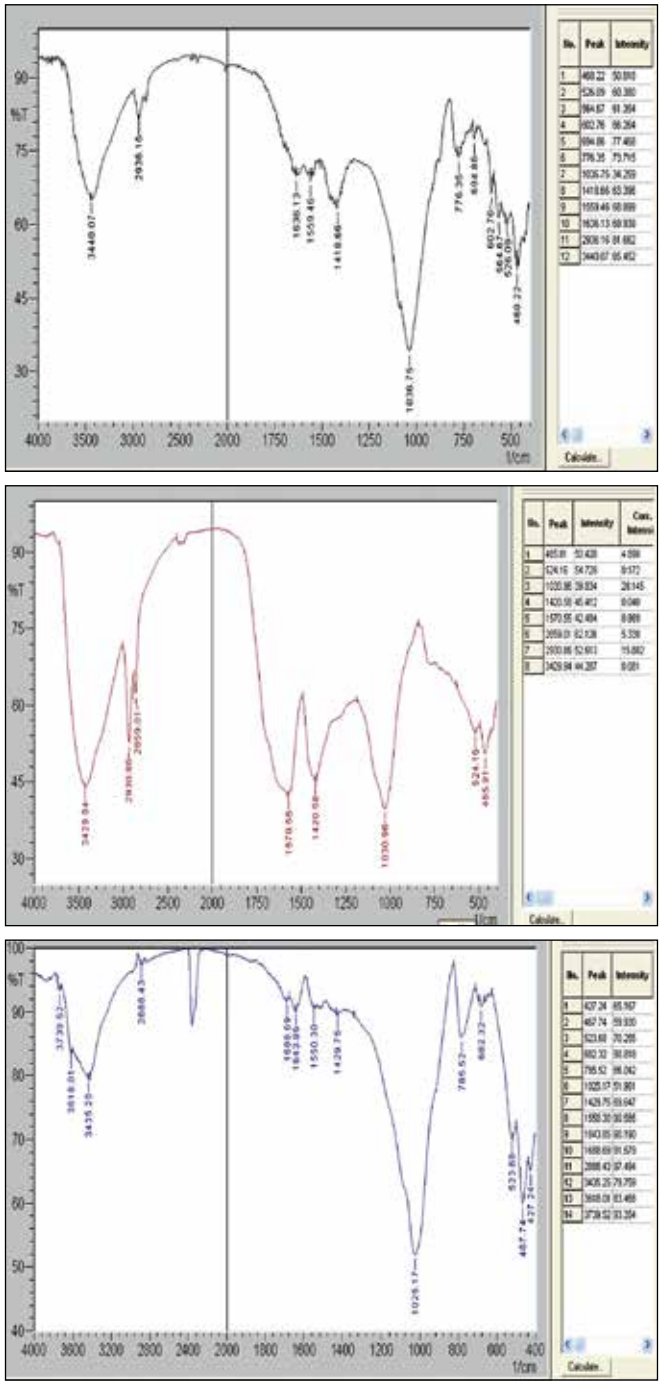

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Figure 3. FTIR spectra obtained on different parts of the lacquer cup: (1) lacquer pigmented layer, (2) fiber layer and (3) wood foundation layer. FTIR spectra of lacquer layer showing bands at 3440 cm^{-1}, 2930 cm^{-1}, 2859 cm^{-1}, 1710 cm^{-1}, 1688 cm^{-1}, 1636 cm^{-1}, 1559 cm^{-1}, 1420 cm^{-1}, and 1030 $cm^{-1}$and traces of 712 cm^{-1}. Hydroxyapatite is identified at 1653, 1453, 1034, 604 and 564 cm^{-1}

the effect of relative humidity or light exposure. The band at 1710 cm^{-1} is due to the carbonyl group vibration [Le Hô et al., 2013, p. 5685–5685], which may be caused by lipidic organic matter. Furthermore, the peak at 1710 cm^{-1} generally has a stronger intensity than the peak at 1630 cm^{-1} in the FTIR spectra of lacquer [Le Hô et al., 2013, p. 5685–5685], which can be seen from the spectra from the lacquerware from Tamir Valley and this is similar to the samples from the archaeological lacquerware from the Gol Mod site; peaks observed at 1710 cm^{-1} are too vague to be even discerned, compared to very strong peaks at 1630 cm^{-1}, indicating the formation of oxidation products. In summary, the FTIR spectra showed the degradation of the archaeological lacquer; we conclude that a careful excavation method is required for such an object that involves protection from light and dehydration for restoration to be successful.

Lacquer pigmented layer. The SEM observations in Figure 4 show quartz particles with different sizes. EDS elemental mapping show high contents of elements Si, Ca, Al, Mg, K, P, Fe and Cu; it clarified that the composition of the lacquer matrix contains quartz and feldspar, as previously confirmed by the FTIR analysis of the lacquer ground layer. The ground layer included both urushiol and silica; this is consistent with previous results [Ma et al., 2017, p. 121–132; Karpova et al., 2017, p. 336–344], which indicate that the ground layer consist of urushi oil and tree oil with inorganic fillers such as quartz, albite and K-feldspar. Fig. 4

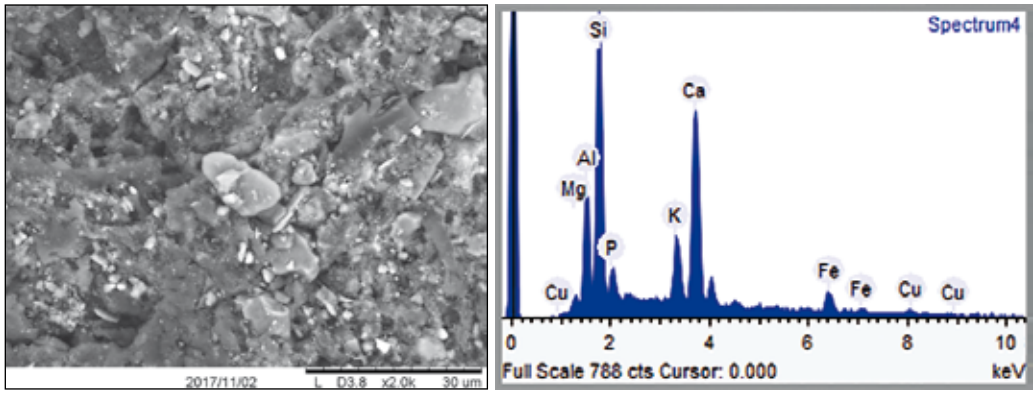

Figure 4. SEM and EDX analyses of the pigment of the lacquer ground layer fragments

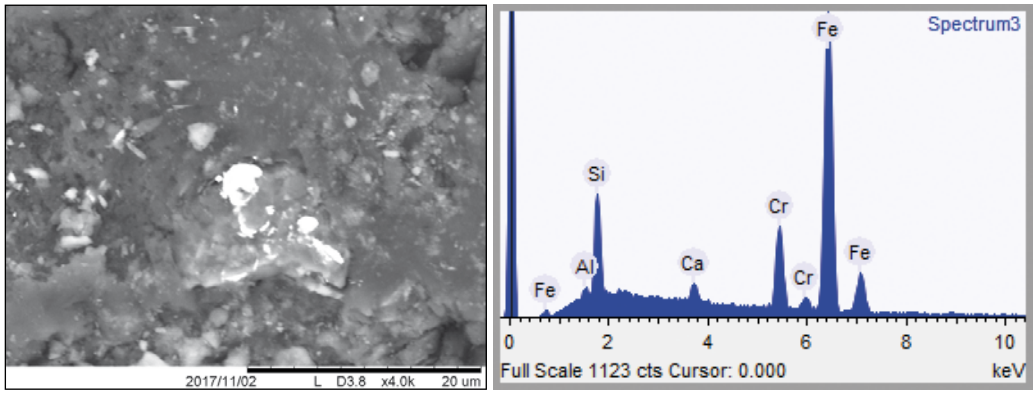

Figure 5. SEM and EDX analyses of the pigment of the lacquer ground layer fragments

illustrates high content of Ca and P which imply, that the white pigment layer consists of hydroxyapatite [Ca10(PO4)6(OH)2] [Wang et al., 2017, p. 105–120], the main inorganic component of bones indicating the possibility of adding animal bone ashes as filler into the ground when making lacquerware, as suggested elsewhere in the literature [Jin et al., 2012, p. 108–111]. The presence of hydroxyapatite was confirmed by FTIR analysis. Fig. 5 shows high content of Fe indicating that the black layer is due to iron oxides as was suggested in the literature [Ma et al., 2017, p. 121–132].

Fiber layer. The SEM observations in Fig. 6 show traces of residuals of silicate clay, quart, albite and potassium feldspar cemented on the lacquerware ground fragment. EDX elemental mapping indicated presence of Fe, Cu and S; the presence of S is mainly associated with the existence of cinnabar, while that of the Fe tends to make the lacquer become dark-colored, or even black [Ma et al., 2017, p. 121–132]. SEM observation of a spot corresponds to Sn (10 μm), indicating that tin was coated on to the ground layer.

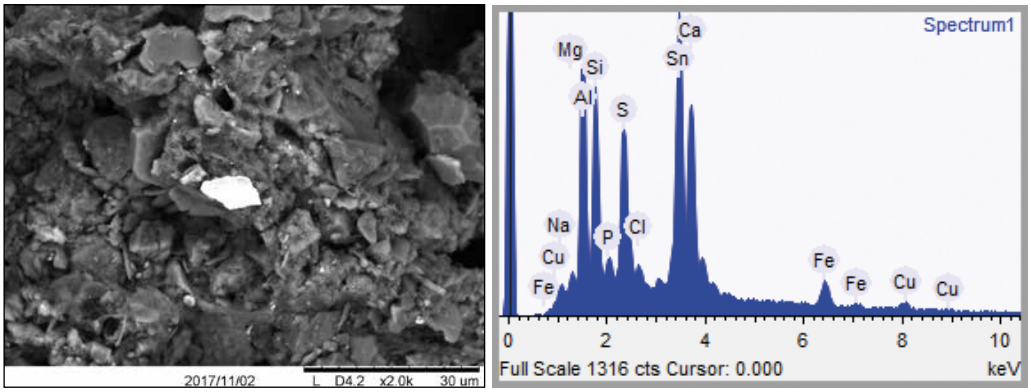

Figure 6. SEM and EDX analyses of the textile fragment

Wood foundation layer. SEM observations of the wood foundation layer showed presence of Si, K, Fe, Al, and Mg and they most likely derive from the clays present in the inner layers of the sample. EDX elemental mapping in Fig. 7 showed the presence of cinnabar (HgS) and minor traces of clay, as detected using SEM-EDX. The SEM shows cinnabar of a particle size 2 to 5 sm.

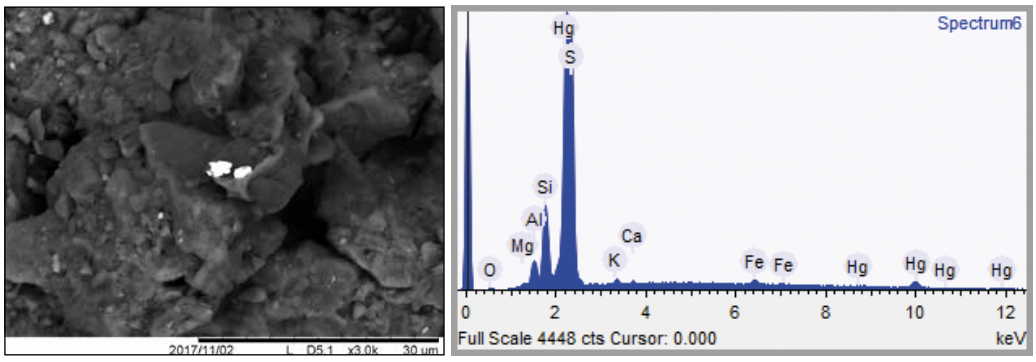

Figure 7. SEM and EDX analyses of the wood foundation layer.
The spot corresponds to cinnabar (HgS)

Conclusion

In this paper, a lacquer TUK2014,T24 excavated from the Xiongnu tomb in Tamir Valley (central Mongolia) was analyzed using FTIR and SEM-EDX and compared with the previous studies on the unearthed lacquerwares in Xiongnu elite burials at Noin Ula. The composition of the lacquer-pigmented layer, fiber layer and wood foundation layer was identified; the lacquer ground layer consisted of urushi oil with inorganic and organic fillers such as quartz, albite and K-feldspar. The Decorative red pigment was identified as cinnabar (HgS) and black pigments as iron oxide. The presence of hydroxyapatite was detected, indicating the possibility of adding animal bone ashes as filler into the ground when making lacquerware. As suggested earlier in the literature, the presence of 712 cm^{-1} lead us to conclude that tung oil may have been added to the lacquer from Tamir Valley during the manufacturing process. The results illustrate the similarity of the composition of the lacquerware TUK2014,T24 from Tamir valley to the sample 44-31 from Noin Ula. However, the studies were limited by the number of samples and the amount of data available for comparison.

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ИЗУЧЕНИЕ ЛАКОВОГО ИЗДЕЛИЯ ИЗ РАСКОПАННЫХ ХУННУСКИХ КУРГАНОВ В ДОЛИНЕ ТАМИРА, АРХАНГАЙ (Монголия)

Империя хунну/сюнну (конец III в. до н.э. – I в. н.э.) контролировала центрально-азиатские регионы Шелкового пути; экономические и культурные отношения с империей Хань подтверждается наличием китайских лакированных изделий в археологических памятниках Монголии. Статья посвящена изучению фрагментов китайского лакового изделия, обнаруженного при раскопках хуннуских курганов в долине Тамира, Архангайский аймак (Центральная Монголия). Применены мультианалитические спектроскопические методы: сканирующая электронная микроскопия, энергодисперсионная рентгеновская спектроскопия и ИК Фурье-спектроскопия. Исследования образцов показывают, что фрагменты лаковой чаши состоят в основном из трехслойной структуры, которая включает слой пигментного лака, слой волокон и слой древесного основания. Исследуемый слой лакового грунта представляет собой смесь органических материалов, включая уруши, сгоревший пепел и неорганические наполнители, такие как кварц и полевой шпат. Сравнение с другими лакированными изделиями хуннуского времени, найденными в разных местах Монголии и изученными ранее другими авторами, проводилось, чтобы получить информацию о хронологическом распределении лакированных изделий в Монголии.

Ключевые слова: лакированные изделия, хунну, FTIR, SEM-EDX.