# pXRF analyses on Assyrain sculptures

*Ryo Anma[1], Ki-Cheol Shin[2], Chikako Watanabe[3], Akihiro Tuji[4]

1. Graduate School of Technology, Industrial and Social Sciences, Tokushima University, 2. Research Institute for Humanity and Nature, 3. Faculty of International Studies, Osaka Gakuin University, 4. National Museum of Nature and Science

To evaluate a possibility of reconstruction and understanding the provenance of stone materials used in the ancient cities of Western Asia, some of which have been destroyed and/or dismantled, and are moved to different places of the world, we characterized the bulk geochemical composition of gypsum-carbonate rocks, on which the Assyrians carved sculptures to decollate their palaces, using an Olympus VANTA portable X-ray fluorescence spectroscopy (pXRF). To establish the methodology, we started with the Assyrian sculptures with known provenance: i.e. the collections of the British Museum, the Ashmolean Museum, Magdalen College (Oxford), the Okayama Orient Museum and the MIHO museum.
The pXRF analysis was performed using a Rh target at 40 kV and 100 $\mu$A. Beam time was 20 seconds for Beam 1 and 15 and 30 seconds, in later sessions, for Beam 2. The accuracy and precision of the analyses were assessed by analyzing several powdered reference samples of igneous rocks (JB-2 basalt, JA-1, JA-2 andesites, JR-2 rhyolite JG1, JG2 granites and JGb-1 gabbro) and sedimentary rocks (JLk-1 lake sediment, JSd-1 and JSd-2 river sediments, JLs-1 limestone and JDo-1 dolomite) distributed by the Geological Survey of Japan, and gypsum samples (alabaster, selenite and satin spar) distributed by Nichika Inc. We performed the measurement three-times for each spot to assess repeatability of the measurement and/or inhomogeneity of the samples.
Our results showed that ~ 99% of the measured Assyrian sculptures were made of gypsum-anhydrate rocks. Although their distribution fields largely overlap, the gypsum sculptures from Nineveh tend to have more impurities when compared with those from Nimrud and Khorsabad. Visually, the stone materials used in the sculptures in Nimrud often have homogeneously distributed large gypsum crystals, whereas those from Nineveh are rather inhomogeneous and finer in grain size, and often exhibit developed bedding planes. Together with the textural changes, we provisionally conclude that degraded stone materials became used over time.
The Til-Tuba sculpture from Room 33 of the Southwest Palace of Nineveh has higher Ca and lower S contents compared with others. Visually, it contains numerous foraminifera shells of *Alveolinidae Sp*. The different chemical composition could be attributed to the amount of the included foraminifera shells. Stones from Room 36 of Nineveh Southwest Palace have slightly higher Al and Fe contents than others. Although, few such examples have somewhat different compositions, the exact provenance would not be able to identify from a single chemical data. More information obtained through destructive analyses would be necessary for the reconstruction and understanding the provenance of stone materials, such as textural and mineralogical information through thin-section observation, crystallographic information through XRD analysis and analyses of micro-fossils.
Nevertheless, our observation from a material science point of view can provide evidence to what Sennacherib (705-681 BCE) described in his text. The king states that he used "$Na_4$.$^{d}$Še.tir" stone from Nipur (Judi Dagh in SE Turkey) that looks like cucumber seeds to construct the statues of Aladlammû and *Apsātu*. Based on this, Mitchel and Middleton (2002) speculated that this same stone was used for the reliefs in Room 33 of the Southwest Palace, Nineveh. If their speculation is correct, evaporites of the middle to late Miocene Fars formation should have comprised the quarry.

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