

# **Investigation on Neolithic adornments using X-ray spectrometry**

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Jade is nowadays understood as two types of minerals: nephrite (dominant chemical elements are Ca, Mg, Fe, Si and O) and jadeite (dominant chemical elements are Na, Al, Si, O), minerals which are similar in appearance (especially as green color). Jade was been used since Neolithic in the production for adornments (amulets, pendants, earrings) because it is polished easily (with wet fine sand). In Romania jadeite Neolithic objects were identified by Pierre Petrequin by spectro-radiometry and published together with Done Serbanescu and others in a synthesis of such discoveries in Romania, Bulgaria and Ukraine, the jadeite coming from the Alps.



**Chapitre 19****Des Alpes à la mer Noire  
(Bulgarie, Roumanie et Ukraine)**

*From the Alps to the Black Sea (Bulgaria, Romania and Ukraine)*

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**Résumé :**

Ce chapitre vient compléter une première étude sur la Bulgarie (Pétrequin, Cassen *et al.* 2012b), où une origine alpine avait été proposée pour 46 lames polies, provenant en particulier du dépôt de Svoboda et des nécropoles de Varna I, Varna II et Durankulak. Cette hypothèse sur les sources italiennes de matières premières, à 1 600 km à vol d'oiseau, surtout depuis le massif du Mont Beigua, a été critiquée à plusieurs reprises, en raison – dit-on – d'une distance excessive qui aurait dépassé les normes habituelles du Néolithique pendant le V<sup>e</sup> millénaire ; cette observation – qui n'est qu'une hypothèse parmi d'autres – nous a conduit d'une part à envisager d'autres sources possibles de jadéite, en particulier dans les Cyclades (île de Syros), et d'autre part à augmenter le nombre de lames polies analysées en prenant en compte le nord de la Bulgarie, l'est de la Roumanie et l'ouest de l'Ukraine. Quatre nouvelles lames ont été étudiées en Bulgarie,

un procédé à peu près absent chez les producteurs d'Italie du Nord. Quant au type Varna, il illustre une forme d'appropriation des importations exotiques, en les repolissant complètement jusqu'à obtenir une forme trapézoïdale originale ; le plus bel exemplaire – et de grandes dimensions – est celui de la tombe 43 de Varna I.

Bien que les lames en jades aient toujours été utilisées comme outils, raffûtés, brisés et recyclés, on ne peut pas les considérer comme de banals outils de travail du bois, car elles ont fait l'objet d'une sélection drastique, où les jadéites les plus claires ont eu la primauté, après un parcours vraisemblable de 700 km à vol d'oiseau depuis la côte adriatique. Cependant, le modèle « occidental » de gestion des jades n'est pas applicable ici, où les lames sont petites à moyennes et rarement, semble-t-il, associées à ce que l'on pourrait considérer comme des contextes de dépôts consacrés et de sanctuaires.



FIG. 9

Le mobilier de la sépulture M17 de Căscioarele - D'ăia Partea (Roumanie), datée de 4887-4547 av. J.-C. et attribuée à Gumelnița A1.  
Photos et DAO A.M. et P. Petrequin.

**Fig. 9 from volume 3, “Jade. Objects-signes et interpretations sociaux des jades alpines dans l’Europe néolithique” editor Pierre Petrequin, is the funerary inventory of tomb 17 - Eneolithic Necropolis at Căscioarele, the “D-aia Parte” point: a jadeite tile, three bracelets from *Spondylus gaederopus* shell (living in Aegean Sea and Mediterranean Seas), beads from *Spondylus* and *Dentalium* shells. This necropolis belongs to the Boian culture, Spanțov stage, the first half of V<sup>th</sup> Millennium BC.**

# **Radiocarbon dating**

- **The analysis carried out at the Arizona University laboratory. Analysis of one human tooth from tomb M. 17 - necropolis Cascioarele-D-aia Parte: 4897-4547 BC cal.**
- **Poznan laboratory. Analysis of a human bone from tomb M. 30 - necropolis Cascioarele-D-aia Parte: 4723-4547 BC cal.**



**Nephrite hair-pin Varna**



## UN PANDANTIV DIN NEFRIT DE LA SĂLCUȚA, JUD. DOLJ

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**Abstract:** *In this paper the author presents an unpublished nephrite pendant discovered in the settlement of Sălcuța–Piscul Cornișorului (Dolj County) over six decades ago. The artefact is a schematized anthropomorphic figurine, and is the only one of its type in Romanian archaeology. We analyze here the possible significance of this artefact and the special material it was made of, as nephrite undoubtedly held special symbolic meaning in prehistory.*

**Rezumat:** *În acest articol autorul prezintă un pandantiv de nefrit, inedit, descoperit în așezarea de la Sălcuța–Piscul Cornișorului (jud. Dolj) cu peste șase decenii în urmă. Piesa reprezintă o figurină antropomorfă stilizată și este una dintre puținele piese de acest tip la nivelul descoperirilor arheologice din România. Cu această ocazie sunt analizate posibilele semnificații ale acestui tip de artefact și a materiei prime speciale din care a fost confecționat, fără îndoială nefritul având o încărcătură simbolică deosebită în preistorie.*

**Key words:** *Late Copper Age, Sălcuța, pendant, nephrite, green colour, symbol, amuletă.*

**Cuvinte cheie:** *Eneolitic târziu, Sălcuța, pandantiv, nefrit, culoarea verde, simbol, amulet.*



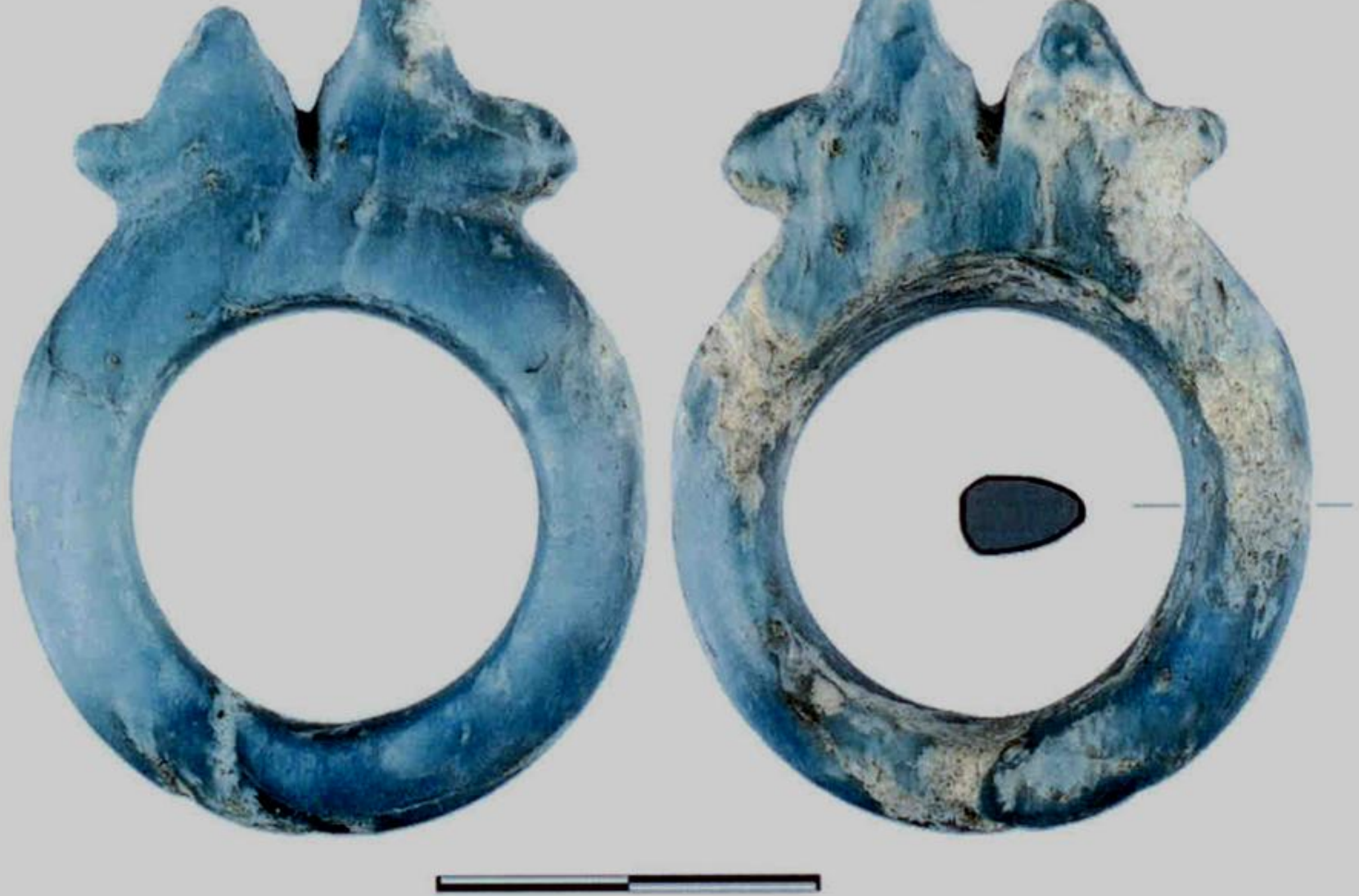
Fig. 3. Pandantivul de nefrit / *The nephrite pendant*  
(desen / *drawing* by C. Georgescu; foto / *photo* C. E. Ștefan).

**The anthropomorphic pendant from the settlement Sălcuța (Piscul Cornișorului) belongs to Salcuta culture and is dated back to the second half of V<sup>th</sup> millennium BC.**

## **THE NEPHRITE AMULET FROM ZMAJEVAC (CEROVAC, CENTRAL SERBIA)**

It is an unwritten rule in Serbian archaeology that the most beautiful artifacts come from the field as chance finds and without an archaeological context. This is also the case with an amulet belonging to the private collection of one of the authors. The artifact was found during the visit of the Zmajevac site in October 2006. It is an amulet of unique shape manufactured in Southeast Europe – worth to be published despite the fact that it is a chance surface find<sup>1</sup>.

The Zmajevac site (**figs 1-2**) is situated in the village Cerovac, 11 km southwest of Smederevska Palanka. It is located near the church at the southwestern edge of the village and lies partially within the churchyard. Today this site is known (and mentioned on maps) as »Lipovac«, while »Zmajevac« is an ancient local name of this area originating from the name of the village Hale which existed at that very location in the 15<sup>th</sup> century. In the archaeological literature, however, the site is mentioned under its ancient name (Katunar 1988). It is situated at 148m a.s.l. on gentle slopes descending towards the river Crkveni potok which transects it. Maize and other crops requiring deep plowing grow in this cultivable soil. It is this deep plowing of the terrain which results in the permanent occurrence of new archaeological artifacts on the surface. A site survey was conducted in 1970, 1972 and 1976. Small-scale sondage excavations under the directorship of Ratko Katunar, keeper in the Narodni Muzej in Smederevska Palanka, were conducted in 1977 within an American-Yugoslav project (see note 1; Katunar 1988, 110). On that occasion, two rather small trenches



**Fig. 3** Amulet from Zmajevac, Cerovac, Central Serbia (chance find). – Max. height 4.5cm. – (Photo Nebojša Borić).

**Anthropomorphic pendant from Zmajevac, Serbia -  
dated back to early Neolithic, Starcevo culture.**

	site	artifact	period	bibliography
1	Zmajevac	ring-amulet	Early Neolithic	unpublished
2	Velesnica	ring	Early Neolithic	Antonović 2003, 122
3	Vinča	palette-pendant	Middle Neolithic	Antonović 1992, 35-37
4	Ribnica	pendant	Early Neolithic	Ljamić 1986, 152 cat.-no. 4
5	Benska bara	tool	Middle-Late Neolithic	Trbuhović / Vasiljević 1983, 41
6	Kusić	small adze	Neolithic(?)	Milleker 1938, 111. 134
7	Srpski Krstur	chisel	Neolithic	Milleker 1938, 134
8	Vršac	chisel	Neolithic(?)	Milleker 1938, 134
9	Divostin	chisels	Early Neolithic	Prinz 1988, 256. 262
10	Grivac	axes, polishers	Early-Middle Neolithic	Antonović 2008, 412-417
11	Čoka	tool	Late Neolithic	Banner 1960, 33
12	Drenovac	axe-chisel	Early Neolithic	unpublished; Narodni Muzej, Belgrade
13	Blagotin	chisel	Early Neolithic	unpublished; Arheološka zbirka, Filozofski fakultet, Belgrade
14	Pločnik	adze, chisel	Middle Neolithic	unpublished; Narodni Muzej, Belgrade

**Tab. 1** Neolithic artifacts made of nephrite from the territory of Serbia (for the location of the sites see fig. 1).

## GEM MINERALS AND MATERIALS FROM THE NEOLITHIC AND CHALCOLITHIC PERIODS IN BULGARIA AND THEIR IMPACT ON THE HISTORY OF GEMMOLOGY

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**Abstract:** Studies of prehistoric (Neolithic to Chalcolithic period) artefacts from the territory of Bulgaria during the past decade revealed a lot of specific gem and decorative minerals and materials: nephrite, malachite, serpentinite, turquoise, jadeite, jet, carnelian, agate and jasper (including heliotrope). Nephrite artefacts in Bulgaria, as well as in some other countries on the Balkans, are widespread during the Neolithic and rare during the Chalcolithic – the nephrite sources are under discussion. A Balkan "nephrite culture" is introduced, which is supposed to be the earliest in the world, compared to the well known Chinese "nephrite cultures". The Varna Chalcolithic necropolis (middle of the V mill. BC) is known with the earliest and largest amount of gold artefacts in the world, including also some copper objects from the copper mines near Stara Zagora. A large amount of beads are also identified as made by malachite (in rare cases with azurite), serpentinite, carnelian, agate, coal (jet), marble and shells. Some of the carnelian beads from Varna display 16+16 facets along their elongation, which is the first record for a constant and complex faceting of hard mineral known so far. An early prehistoric weight system links mineral beads and gold artefacts (the weight unit "van" is introduced, 0.4 g = 2 carats). The first report of turquoise beads for SE Europe is related to the Orlovo prehistoric site (Haskovo district). The "Thracian stone" in ancient sources is identified also as heliotrope, which is known since the Chalcolithic in the Eastern Rhodopes. Some of the artefacts are masterpieces of art and as stage of perfection, thus pointing to the Balkans as a cradle of prehistoric gemmology.

**Keywords:** nephrite, malachite, serpentinite, jadeite, turquoise, carnelian.



**Recently discovered amulet (?) in Bulgaria**

**The fact that many nephrite Neolithic adornments were found especially in South-West Bulgaria and in Serbia suggests the existence of a nephrite deposit in this region. The well-known Jordanow deposit in Poland is too far to be considered as the source of raw material for artifacts identified in the Balkans area, including now in Romania. It is the job of geologists to identify nephrite deposits in the Balkans and compare them with archaeological artifacts.**



# Nephrite and jadeite Neolithic adornments found by Done Serbanescu (Cascioarele and Sultana sites)





**Two  
nephrite  
ear-rings  
from  
Căscioarele  
-“D-aia  
Parte”  
Boian  
Culture  
necropolis,  
Spantov  
stage,  
discovered  
in tomb 6  
and dated  
4897-4547  
BC cal.**



**Jadeite ax pendant (Inv. MCG 8055) discovered in the Eneolithic tell from Vladiceasca, Valea Argovei, Calarasi county, belonging to the Gumelnitza A2 culture - second half of V<sup>th</sup> millennium BC.**



**Jadeite adze (Inv. MCG 8750) discovered at Coconi, Manastirea, Calarasi County, Gumelnitza Culture - the second half of V<sup>th</sup> millennium BC.**

**This necklace consists of bead made from pieces of oxidized native copper and malachite perforated and polished like a rock. People during Boian culture did not know the metallurgical method of copper processing, by beating or melting.**



**Necklace with pendant from Sultana – Valea Orbului  
Beads from bone and nephrite pendant**



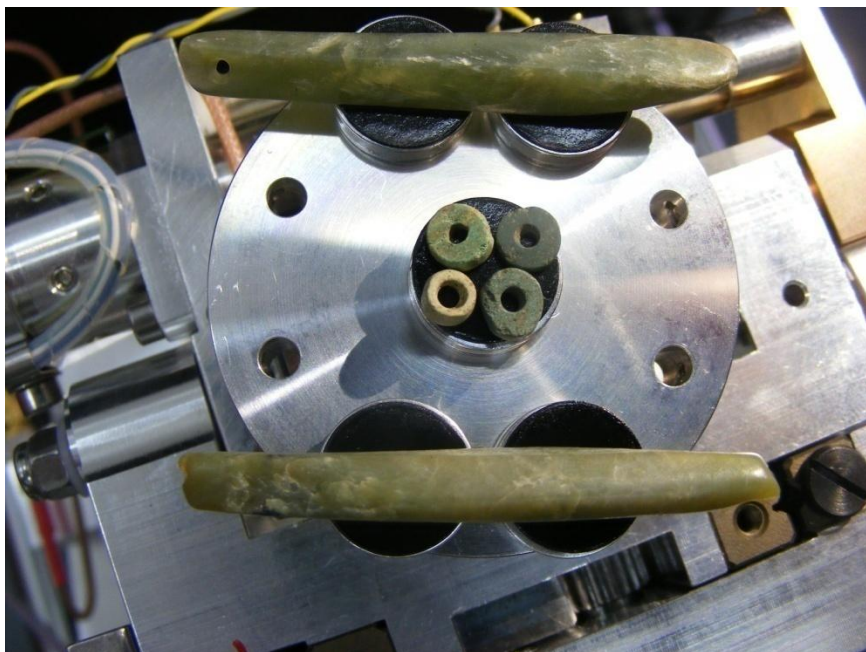
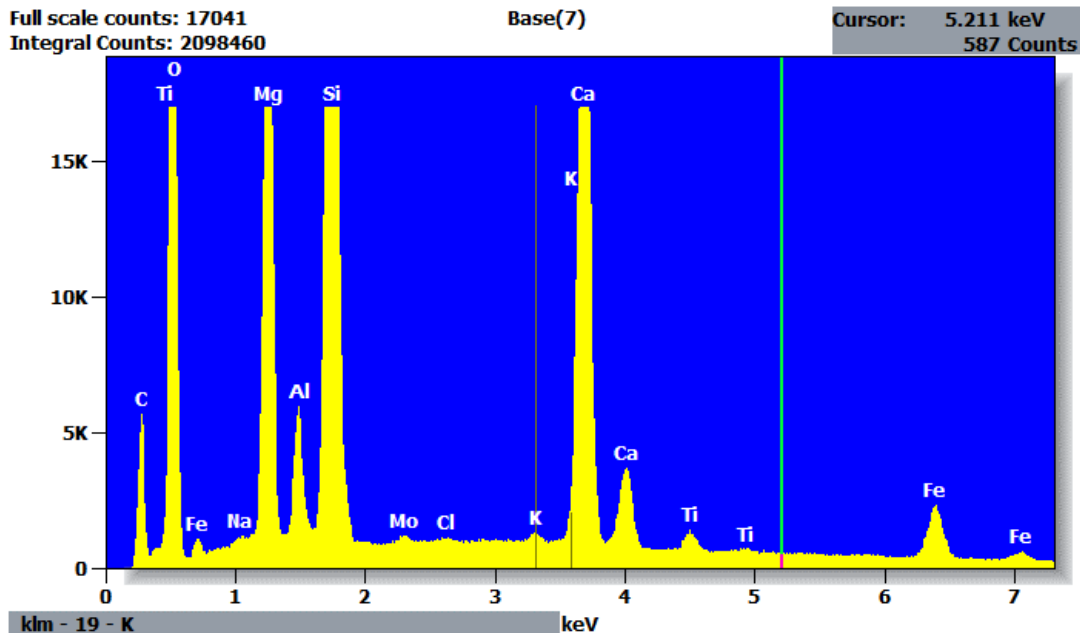
**Found in Sultana – Valea Orbului necropolis (Boian-Bolintineanu culture), radiocarbon dated between 5301-5169 BC cal.**

**Nephrite identification was made by X-ray spectroscopy using a Zeiss EVO MA15 electron microscope, highlighting the dominant presence of Ca, Mg, Fe, Si and O.**

**SEM-EDS (Scanning Electron Microscope - Energy Dispersive X-ray Spectroscopy) analyzes are performed on surfaces between 0.02 and 0.6 mm<sup>2</sup> (diameter about 150-250 μm). The magnification is between 400 and 1500 x. The penetration depth of the electrons is up to 1 μm for the acceleration voltage of 20 kV, depending on sample composition. For a good statistics we used a 1 nA current.**

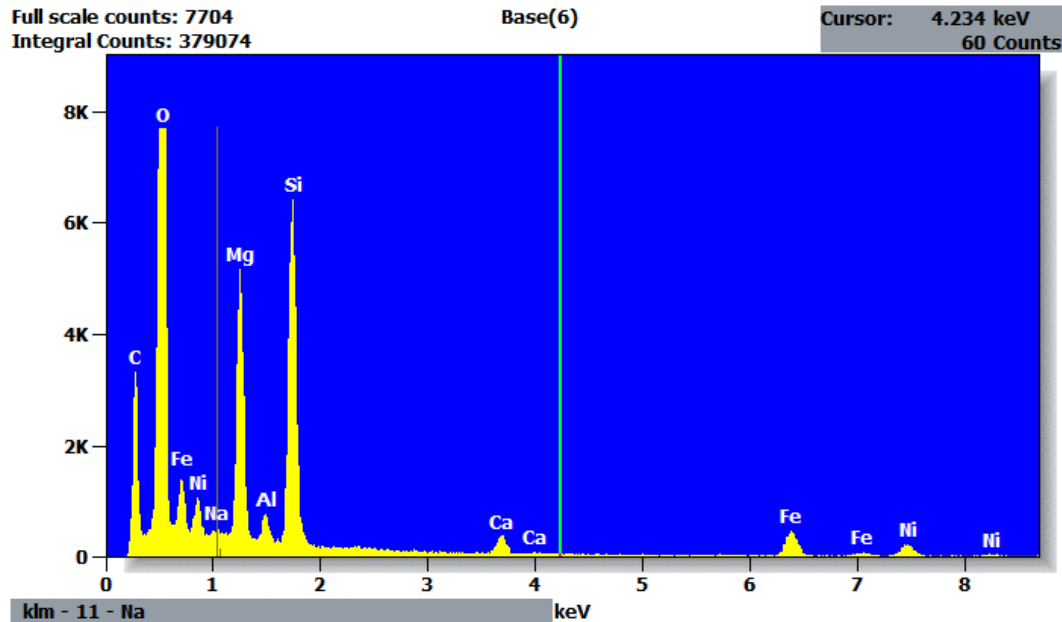


The most important results are the identification of the nephrite - silicate of calcium, magnesium and iron - in the two earrings found in Cascioarele and in a “foot”-pendant found in Sultana.



Both, the earring Inv. 12019 and the earring Inv. 12020 are from nephrite of high purity and homogeneity. There is only a minimal contribution from jadeite - sodium and aluminum silicate (see Na and Al peaks in the spectrum).

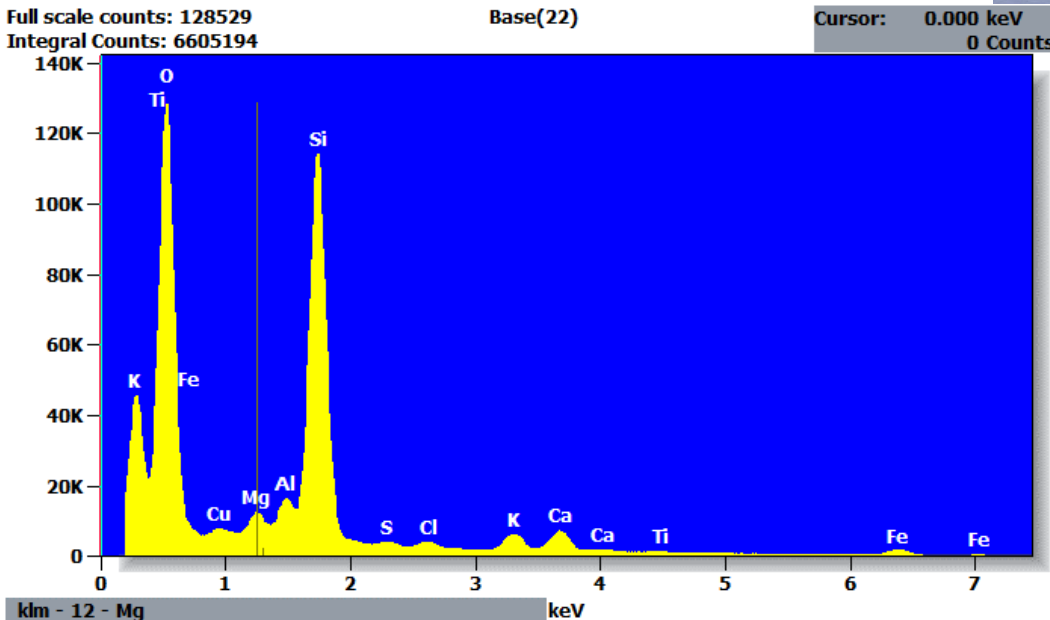
To confirm the identification of nephrite, we analyzed two geologically confirmed nephrite modern pendants. A further check-in consisted in observing of transparency when samples are exposed to a light source, the other form of jade - jadeite - being opaque.



**SEM –EDS spectrum and photo; Nephrite modern pendant - geologically confirmed**

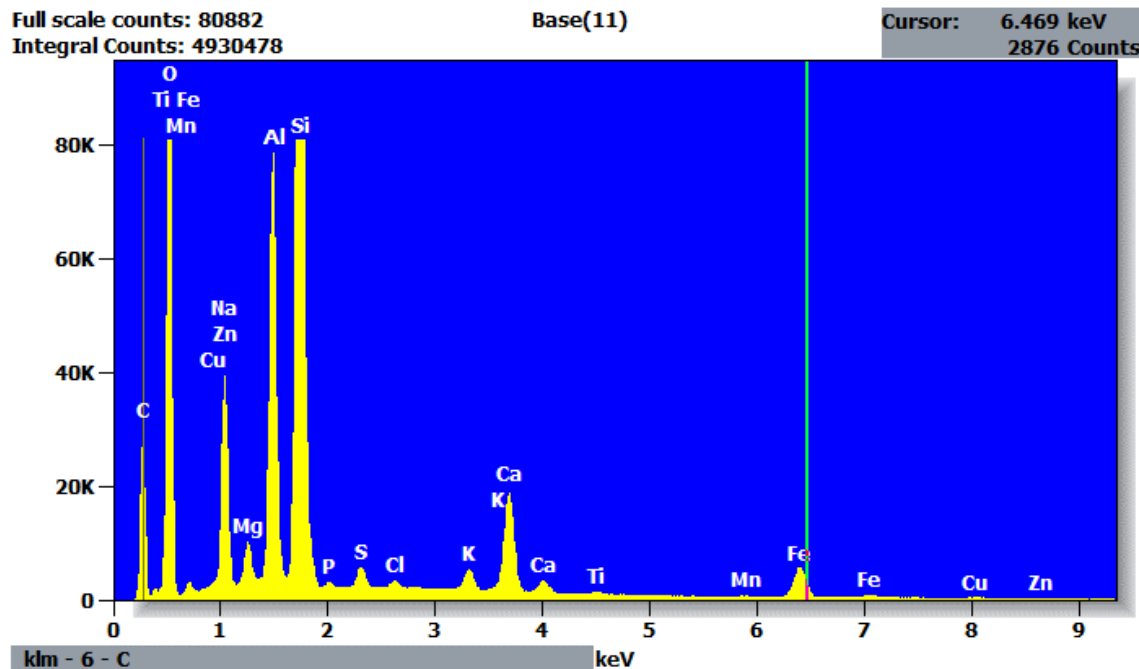


**SEM-EDS analyze of a small pendant in the form of ax (mini-foot?) containing nephrite accompanied by an important component of aluminum silicate - see the spectrum below). Copper traces are responsible for the green-blue color.**



**It was discovered in the necropolis Sultana - the "Valea Orbului" - Boian culture, Bolintineanu phase, it was carbon dated in Poznan laboratory between 5197-5062 BC cal. and according to the data obtained in our institute's AMS laboratory between 5301 -5069 BC cal.**

Our method was verified on two jadeite pendants previously analyzed by Pierre Petrequin where Na, Al, Si, O were highlighted.

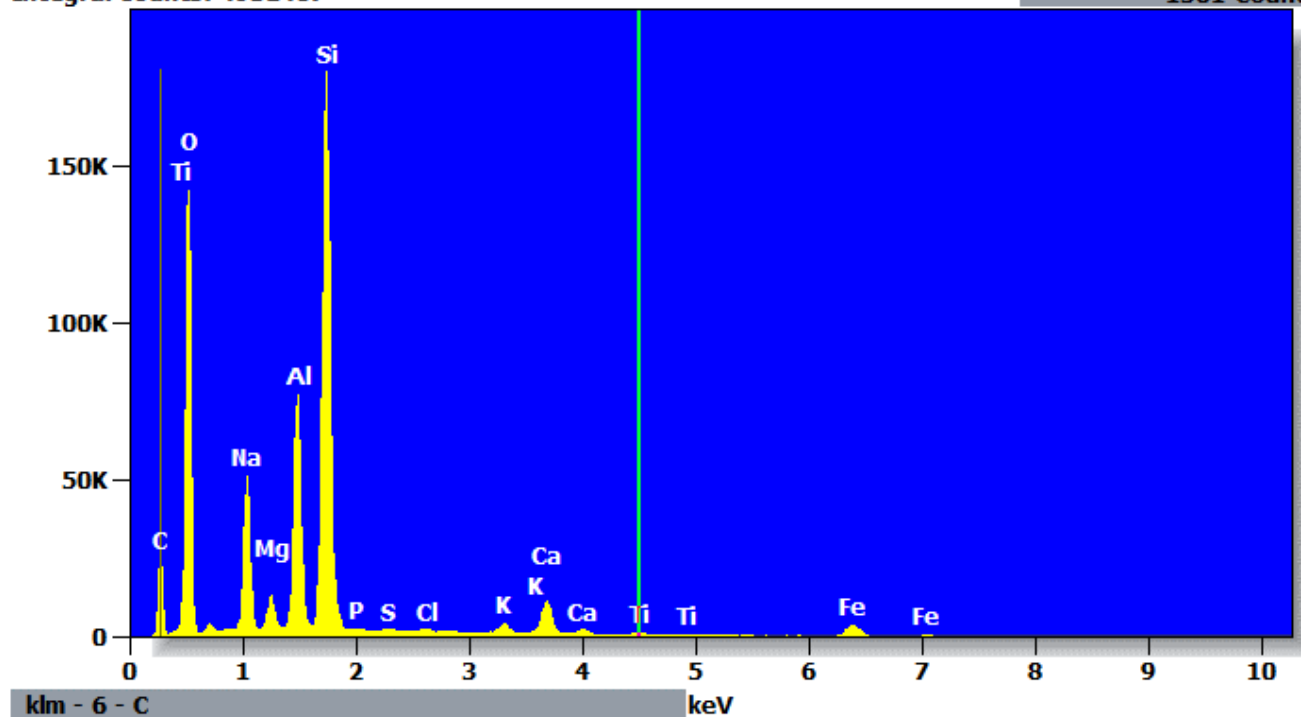


SEM –EDS spectrum and photo  
jadeite adze pendant Inv. 8750

Full scale counts: 180074  
Integral Counts: 4931487

Base(7)

Cursor: 4.500 keV  
1561 Counts



**SEM -EDS spectrum and photo  
Ax pendant Inv. 8055**



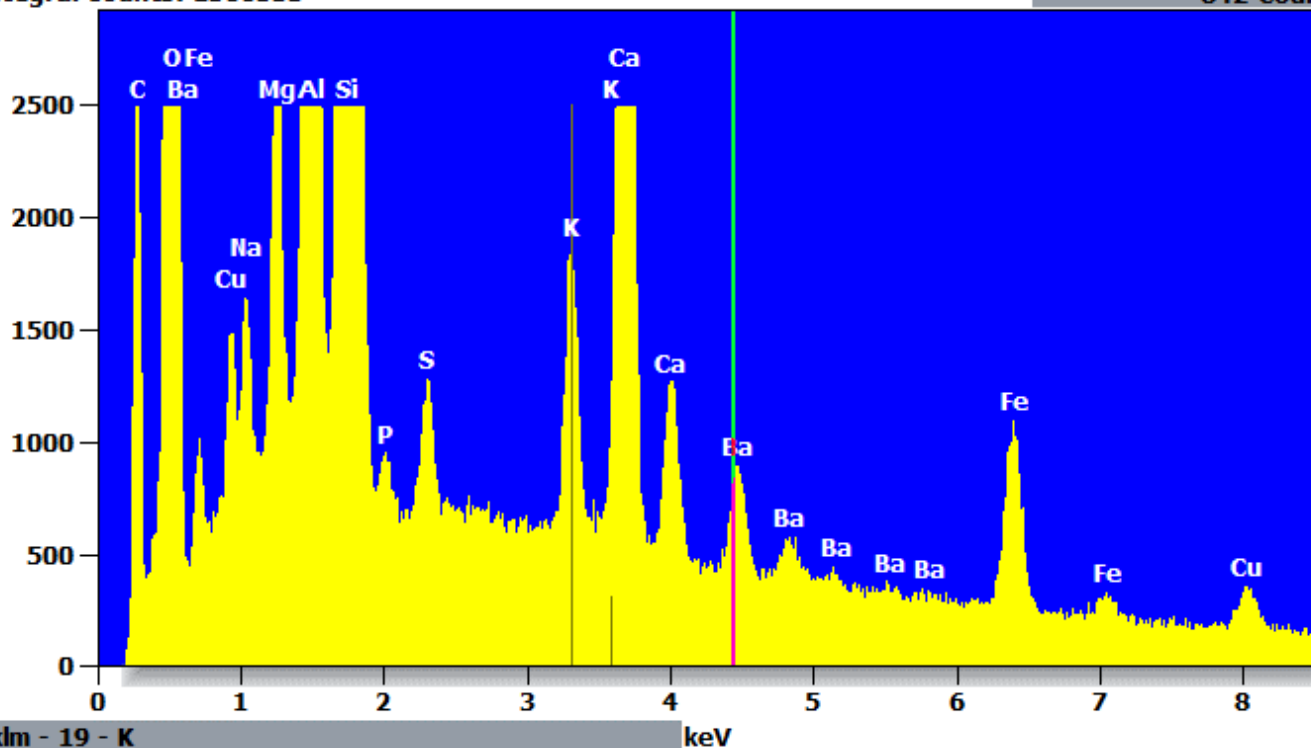
A necklace from nephrite and malachite beads was discovered in the necropolis of Sultana - "Valea Orbului" - Boian-Bolintineanu culture radiocarbon dated between 5301-5069 BC cal.



Full scale counts: 2492  
Integral Counts: 1588533

Base(11)

Cursor: 4.442 keV  
842 Counts



**SEM –EDS  
spectrum for a  
mixed nephrite –  
jadeite –  
aluminium  
silicate**



**Native copper  
accompanied by  
malachite –  
Michigan, USA**

**A special case was the beads containing copper-metal and malachite (copper carbonate). The geological explanation is the presence of native copper and malachite in some geological deposits due to the water-oxidation of copper-iron sulphide (the famous calcopirite - the "gold of fools") - the principal mineral from which copper is obtained. The explanation of the native copper-malachite mix is probably the meaning of green color for the Neolithic people (green=vegetation, spring rebirth)**

**Thank you for your attention!**