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V.M. Belskyi [1], https://orcid.org/0000-0001-7990-1386
H.O. Kulchytska [1], https://orcid.org/0000-0002-7206-4797
D.S. Chernysh [1, 2], https://orcid.org/0000-0001-5390-2591
L.I. Kanunikova [1], https://orcid.org/0000-0002-7530-969X

[1] M.P. Semenenko Institute of Geochemistry, Mineralogy and Ore Formation of the NAS of Ukraine, Kyiv, Ukraine
[2] Institute of Renewable Energy of the NAS of Ukraine, Kyiv, Ukraine
E-mail: belskyi.vm@gmail.com

Discovery of a rare earth mineral in an archaeological artefact

Presented by Corresponding Member of the NAS of Ukraine V.P. Semenenko

The chemical and mineral composition of an archaeological artefact — Bronze Age ceramics from a settlement near the city of Kaniv was studied. It was established that red pottery clay mixed with sand was used for its formation, most likely from the deposits of the Kaniv Paleogene suite. An increased concentration of phosphorus (1—3 wt. % P_2O_5), a reduced alkalis content, and the absence of REE characterised the clay. In one of the ceramic samples, a solid-phase cluster ~100 μm in size was found, which, in reflected electrons, stands out with a bright glow against the background of a grey clay matrix and a contrasting chemical composition. The dominant role in the cluster belongs to cerium (Ce_2O_3 36—49 wt. %), subordinated to oxides of Si, Al and P (on average 23, 9 and 6 %, respectively). Other REE, except for a small amount of La oxide (1 %), are practically absent. Within the cluster, at least two phases with blurred hexagonal outlines are distinguished, which visually appear homogeneous, which also confirms the relatively uniform distribution of other components — oxides of Si, Al, P, Fe and Ca. Identified as a Ce and Al phosphosilicate with the empirical formula $Ce_{1.78}La_{0.03}Ca_{0.24}Al_{1.29}Fe_{0.43}Si_{2.65}P_{0.58}O_{12.3}$, the phases have no analogues among known mineral species. In terms of the ratio of components, the phases are simultaneously close to species in both the allanite and brytholite groups, and with such a ratio of components, they can only be an amorphous mineral close to brytholite. Various possible sources of Ce for mineral formation and the reason for its purely cerium composition are considered. Most likely, the phosphosilicate was formed in the deposits of the Kaniv suite due to Ce ions physically adsorbed on clay minerals. The ions easily passed into solution and were precipitated by phosphorus compounds according to the chelation scheme. The migration of the element under oxidising conditions in the Ce^{4+} form caused its separation from other trivalent rare earth elements.

Keywords: *clay, cerium, REE adsorption, Kaniv suite, ceramics.*

Introduction. Archaeological artefacts — objects or phenomena artificially created or modified by humans in the past, possessing historical, cultural, and scientific value, and providing insights into human history (household utensils, tools, cult objects, etc.) — are of interest to researchers not only from the perspective of their functionality. The material composition of the objects is

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studied with equal scrutiny, enabling the elucidation of the source of the material and the logistical pathway of its transit to the site of the archaeological find. Typically, such investigations are conducted by mineralogists [1, 2]. One of these artefacts became the subject of our study.

Purpose of the research. To determine the chemical and mineral composition of Bronze Age pottery and to identify the distinctive features of its composition.

Object and methods of research. In accordance with Contract No. 70 dated 01 July 2025, the M.P. Semenenko Institute of Geochemistry, Mineralogy and Ore Formation of the National Academy of Sciences of Ukraine conducted a study of a collection of Bronze Age pottery fragments from a settlement in the vicinity of the town of Kaniv [3]. Polished sections for the research were provided by Artem Serhiyovych Derhai, a postgraduate student at the Faculty of History of Taras Shevchenko National University of Kyiv. According to the technical specification, it was required to determine the mineralogical and geochemical features of the ceramic fragments. One of these fragments (No. 13) stood out among the others due to the presence of aggregates enriched in the rare-earth element cerium within the ceramic matrix. According to the data provided by the collection owner, the sample was collected by M.M. Bondar in 1961 at excavation site "B" in square 1 at a depth of 0.4—0.6 m within the Iskovshchyna settlement of the Middle Dnipro culture [4, pp. 12-91].

The chemical and mineral composition of the pottery was determined using a JXA-733 microprobe (JEOL, Japan) equipped with an energy-dispersive spectrometer. Analysis conditions: accelerating voltage 20 kV, probe current 20 nA, probe diameter 10 μm. Minerals with verified chemical component contents (monazite, diopside, augite, etc.) were used as reference standards.

According to archaeological research data, the components for the pottery paste (clay, sand) were obtained by the settlers near their place of residence [5], which allows for the localization of the area of mineralogical investigation.

Results of the pottery characterisation. Typically, the primary component of potting clay for ceramic production is kaolin, with a SiO_2:Al_2O_3 ratio of ≈ 2:1. In kaolinite, which is the principal mineral of kaolin, this ratio is even lower. In the analysed samples, this value is higher — ranging from 2.4 to 3 (Table 1), which indirectly indicates the addition of sand (SiO_2) to the clay, furthermore being poorly blended. The presence of a fairly significant amount of Fe_2O_3, alongside noticeable impurities of TiO_2 and CaO in the ceramic composition, indicates that the so-called red potting clay was used for firing. Apart from kaolinite and quartz, this clay contains iron-micaceous minerals or products of their disintegration. Conversely, the alkali content (Na_2O i K_2O) is rather low. The elevated phosphorus content is noteworthy. Generally, the source of kaolin clays is the weathering crust of granitoid rocks that has undergone leaching and partial desilicitisation. The elevated phosphorus content indirectly points to the sediments of the Kaniv Suite of the Paleogene, the finest outcrops of which are situated near Kaniv [6]. The sediments of the Kyiv Suite, developed in the Middle Dnipro region, could also have served as a source. A characteristic feature of both suites is the presence of layers with phosphorite nodules within the section. These are predominantly sandy sediments, though clayey interlayers also occur in the upper horizons of the suites.

Results of the investigation of Ce-bearing phases. In one of the pottery samples (sample No. 13), an aggregate of solid phases was detected, at least two of which exhibit blurred hexagonal outlines (Figure, analyses No. 19-4 and 19-6 in Table 2). In backscattered electrons, the aggregate stands out with a bright luminescence against the background of the grey clay matrix and displays a contrasting chemical composition in comparison. The dominant role in the phases belongs to

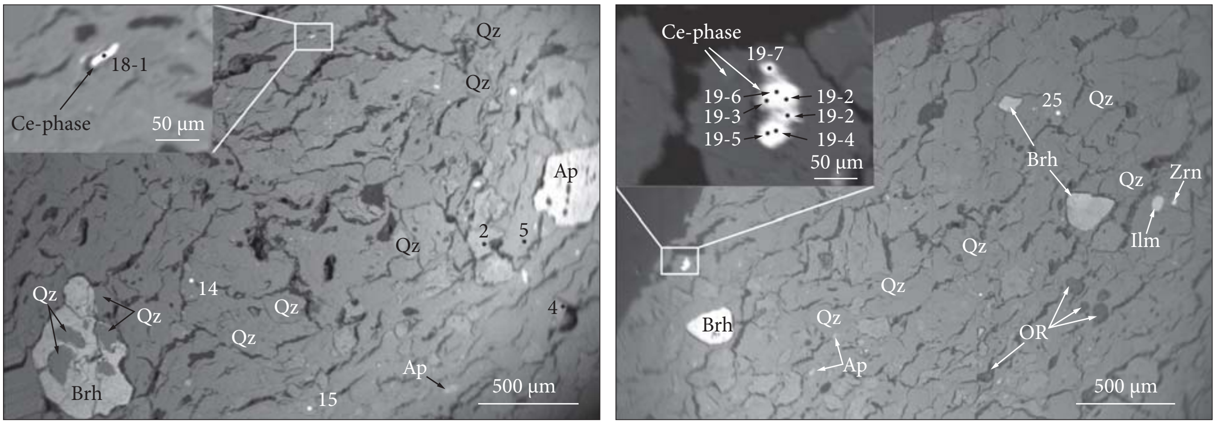
Ce_2O_3, against the background of a negligible content of other determined *Ln*. The contents of Y and F were not determined. Undoubtedly, this is a rare-earth mineral, and specifically a selectively cerium one. In the image, the phases appear homogeneous; whether they are crystalline or amorphous (metamict) remains unknown. The homogeneous composition of the phases is further confirmed by weak fluctuations in the concentrations of the main chemical components, particularly the oxides of Al, Fe, and Ce, for which the deviation of the content from the mean value is around 10 %. Minor fluctuations are also characteristic of Ca, Si, and P, with the exception of two points. The P_2O_5 content at these points doubles, while simultaneously there is a sharp decrease in the SiO_2 content, which provides grounds to suggest the presence of solid-phase inclusions at these points, such as monazite — $Ce[PO_4]$ or apatite — $Ca_5[PO_4]_3(OH, F)$. Excluding the results at the two "anomalous" points, the variation in the content of these oxides as well, namely CaO, SiO_2 and P_2O_5, in the mineral is also small — 10, 6, and 15% respectively. Similar "spikes" in U and Th concentrations can likewise be attributed to potential inclusions of uraninite (UO_2), thorianite (ThO_2), or thorite ($ThSiO_4$). At points 19-4 and 19-6, where a correlation between Th and Ce content is observed, the likely phase could be a thorium variety of monazite-(Ce) — $(Ce,Th)[PO_4]$. The content of other lanthanides, except for Ce and La, is at the detection limit. The fluctuation in La content is also attributed to its proximity to the detection limit.

Thus, we obtain six main chemical elements distributed relatively evenly within the mineral, the content of each exceeding 1 wt. %. At least three of them (Ce, Si i Al) definitely belong to the formula-forming elements, while the others (P, Ca, Fe) may represent isomorphous impurities of solid solutions. According to the chemical composition, the detected phases can be interpreted as a cerium aluminosilicate or phosphatoaluminosilicate with the empirical formula $Ce_{1,78}La_{0,03}Ca_{0,2}Al_{1,3}Fe_{0,4}Si_{2,6}P_{0,6}O_{12,3}$, or a cerium and aluminium phosphosilicate with the

Table 1. **Chemical composition of the pottery (clay matrix) of sample No. 13, wt. %**

Oxides	Measurements						Average ± avedev
	2	4	5	14	15	25	
Na_2O	N/d	0.16	0.01	N/d	N/d	N/d	0.09 ± 0.08
MgO	0.91	0.89	0.7	0.7	0.8	0.71	0.79 ± 0.08
Al_2O_3	22.25	16.59	15.33	20.4	18.18	17.57	18.39 ± 2
SiO_2	54.03	49.13	46.9	48.93	49.37	48.38	49.46 ± 1.5
P_2O_5	0.66	3.06	3.06	2.76	1.99	0.94	2.09 ± 0.9
SO_3	0.42	0.25	0.3	0.31	0.33	0.22	0.31 ± 0.05
K_2O	0.32	0.81	0.24	0.21	0.41	0.29	0.38 ± 0.15
CaO	1.11	1.39	1.28	1.52	1.18	1.73	1.37 ± 0.18
TiO_2	1.29	0.71	0.69	1.2	0.55	0.94	0.9 ± 0.25
V_2O_5	N/d	N/d	N/d	N/d	N/d	N/d	N/d
MnO	N/d	N/d	N/d	N/d	N/d	0.04	0.01
Fe_2O_3	8.54	6.35	6.12	7.36	8.06	6.87	7.22 ± 0.8
Summ*	89.67	79.4	74.76	83.5	80.97	77.69	81.05 ± 3.72

Note. The numbering of the analyses corresponds to that shown in Figure. N/d — not detected. *The deficit in the total indicates the potential presence of hydroxyl groups (OH), which is characteristic of sorbed complexes.



Aggregate of cerium-bearing phases (Ce-phase) within the clay matrix of sample No. 13. Ap — apatite, Brh — iron hydrosilicates (berthierine/chamosite), Ilm — ilmenite, Qz — quartz, Zrn — zircon, OR — organic residues (Organic Residues). COMPO-image

Table 2. **Chemical composition of the cerium-bearing phases detected in sample No. 13, wt. %**

Oxides	Measurements								Average ± avedev
	18-1	19-1	19-2	19-3	19-4	19-5	19-6	19-7	
Al_2O_3	10.79	9.18	9.15	7.23	9.23	10.41	9.58	8.27	9.23 ± 8
SiO_2	22.53	27.42	22.91	24.05	17.27	26.32	14.84	26.19	22.69 ± 15
P_2O_5	2.82	4.32	4.98	3.42	10.93	5.48	11.23	4.06	5.91 ± 44
CaO	1.84	1.47	1.53	2.06	2.71	1.63	2.23	1.71	1.90 ± 17
Fe_2O_3	4.10	4.52	4.97	4.22	5.48	5.99	4.56	5.13	4.87 ± 11
La_2O_3	1.26	1.44	N/d	N/d	1.12	1.09	1.03	0.32	0.78 ± 65
Ce_2O_3	37.96	35.61	42.50	45. 00	43.88	36.22	48.59	43.60	41.67 ± 9
Nd_2O_3	N/d	N/d	N/d	N/d	N/d	N/d	N/d	N/d	0.00
Sm_2O_3	0.02	N/d	N/d	N/d	N/d	N/d	N/d	N/d	0.00
Eu_2O_3	N/d	0.08	0.03	0.11	0.1	N/d	0.03	0.03	0.05 ± 78
ThO_2	N/d	N/d	0.6	0.37	1.32	N/d	0.93	N/d	0.40 ± 102
UO_2	N/d	N/d	0.01	N/d	N/d	1.14	0.38	N/d	0.19 ± 149
Summ*	81.32	84.04	86.66	86.45	92.03	88.29	93.4	89.29	87.69 ± 3
Crystallochemical coefficients calculated on the basis of seven cations									
Al	1.560	1.253	1.283	1.041	1.250	1.360	1.325	1.238	1.289
Si	2.764	3.176	2.725	2.937	1.985	2.918	1.742	2.936	2.648
P	0.293	0.424	0.502	0.354	1.063	0.514	1.116	0.385	0.581
Ca	0.242	0.182	0.195	0.270	0.334	0.194	0.281	0.205	0.238
Fe	0.379	0.394	0.445	0.388	0.474	0.500	0.403	0.433	0.427
La	0.057	0.062	0.000	0.000	0.047	0.045	0.045	0.013	0.034
Ce	1.705	1.510	1.851	2.012	1.846	1.470	2.088	1.790	1.784

Note. N/d. — not detected. *The deficit in the total indicates the potential presence of hydroxyl groups (OH) or fluorine.

formula $(Ce, Ca, La)_2(\square, Al, Fe)_{1,7}(Si, P)_{3,2}O_{12}$[1]. The results of the analysis at the two “anomalous” points have little effect on the average composition of the phases.

The webmineral.com search engine found no matches among the two hundred mineral species currently known to contain formulaic Ce. The only mineral in which it is theoretically possible to combine all the components is alumino-cerite-(CeCa) from the cerite supergroup, the theoretical formula of which is $(Ce, Ca)_9(\square, Al, Fe^{3+})(Si, P)_7O_{24}(OH)_4$. Isomorphous substitutions between Si and P in cerites are possible, since the cerite subgroup is isostructural with phosphates from the whitlockite subgroup. However, it is evident that for the formation of cerite, the mineral from the pottery contains too little Ce and too much Al.

The composition of the Ce-bearing phases from the pottery is closer to allanite-(Ce) with the theoretical formula $(Ca, Ce)_2(Al, Fe)_3Si_3O_{12}(OH)$. This is particularly plausible given that species from the allanite group, as members of the epidote—törnebohmite homologous series, can contain less Al. However, there is no site for phosphate groups within the allanite structure. On the other hand, isomorphism between the oxygen radicals of silicon and phosphorus is characteristic of the britholite structure — a fairly frequent paragenetic companion of allanite in geological formations. For the composition of the detected phase to approach that of britholite — $Ce_5Si_3O_{12}OH$, all excess components must occupy the same position as Ce; that is, the formula of the Ce-phase must take the following form: $(Ce, La, Ca, Al, Fe)_{4,7}(SiO_4, PO_4)_{3,2}O_{12}(OH)$. Understandably, in the event of such substitutions, the britholite structure cannot possibly be crystalline; thus, the diagnosed Ce-phases, intermediate in composition between allanite and britholite, represent an amorphous mineral.

Formation of the amorphous cerium mineral. The question arises: where did the cerium mineral in the clay pottery come from? Conventionally, there are three sources of *REE* in clays:

a) residual primary *REE* minerals, damaged only partially as a result of weathering of the parent rock;

b) secondary *REE* minerals formed during weathering;

c) hydrated REE^{3+} ions physically adsorbed on the negatively charged surface of clay minerals such as kaolinite, montmorillonite, halloysite, and dioctahedral micas with a deficit of interlayer cations. The ions are held by weak electrostatic forces and transfer relatively easily into solution as a result of ion exchange.

The residual primary mineral is an *REE* mineral inherited by the clay or sand from intrusive rocks. Monazite-(Ce) comes to mind first — a stable phosphate characteristic of felsic rocks. It typically has an idiomorphic shape or is rounded in placers, which does not coincide with the irregular shape of the aggregates. The presence of other components remains unexplained. The firing of pottery occurs at temperatures of around 900—1,000 °C, which makes melting impossible, and the duration of the process is too short for solid-phase segregation of components to take place. Furthermore, the firing process promotes the exsolution of distinct crystalline phases from an amorphous mixture, rather than the reverse.

The secondary mineral could have been supergene cerianite CeO_2 adsorbed by the clay, which formed this unique substance after firing. Such an assumption allows for the explanation of the purely cerium composition of the investigated phase. Almost all known *REE* minerals simultane-

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[1] ☐ — The generally accepted notation in crystallochemical formulae for vacant positions of cation deficit required for charge balancing.

ously contain the entire spectrum of lanthanides, differing only in their ratios. The investigated phase, apart from a minor amount of La, does not contain other *LREE*. A similar find of supergene cerianite, composed almost 100% of CeO_2, oxide, was first described in the pegmatites of Madagascar [7]. The authors of the aforementioned work believed that the separation of cerium is influenced by its ability to form CeO_2 oxide, whereas all other lanthanides tend to migrate in the form *REE*$(OH)_3$. A similar phenomenon has also been recorded in the weathering crust of granitoids in Madagascar [8]. The transition of Ce into the tetravalent state requires oxidising conditions, and in this manner, *REE* fractionation and the separation of Ce from other elements of the group occur.

The most attractive hypothesis is that of clay-adsorbed Ce^{3+} ions serving as the source for the formation of the phase. The Ce-bearing phase was formed back in the clay as a result of the segregation of components with *REE* and P according to the nodule formation scheme. Phosphorus in this case is considered as a precipitant and concentrator of Ce. Phosphates promote the sorption of *REE* on the surface of clay minerals; the ability of phosphorus to form ternary silicate-phosphate-*REE* complexes on the kaolin surface has been sufficiently studied [9]. In an acidic environment (pH 4), these are hydroxo-complexes like $\equiv SiHPO_4$*REE*+, while in a neutral one, they are oxide complexes without a hydroxyl group. Cerium, as a strong luminophore, is capable of rapid oxidation. The replacement of Ce^{3+} by Ce^{4+} nder oxidising conditions promoted *REE* fractionation and the separation of Ce from the trivalent elements of the group. After the firing of the clay, an amorphous mineral of intermediate composition between britholite and allanite was formed, with inclusions of U and Th oxides, as well as Ce and Ca phosphates. Taking into account the oxidation of Ce, the approximate crystallochemical formula of the mineral will take the following form: $(Ce^{4+}, Ce^{3+}, La^{3+}, Ca, Al, Fe^{2+}, Fe^{3+})_5(SiO_4, PO_4)_3O_{12}(OH)$.

Conclusions.

1. According to chemical analysis data, red potting clay enriched with phosphorus, most likely from the sediments of the Kaniv Suite of the Paleogene, was used for the manufacture of the Bronze Age pottery.

2. After the firing of the pottery paste, an aggregate of solid phases formed within the ceramic matrix — namely cerium and aluminium phosphosilicates with the empirical formula $Ce_{1,78}La^{3+}_{0,03}Ca_{0,24}Al_{1,29}Fe_{0,43}Si_{2,65}P_{0,58}O_{12,3}$. The composition of the phases does not correspond to any of the currently known mineral species. In terms of the ratio of components, the mineral simultaneously approaches species within both the allanite and britholite groups. The approximate crystallochemical formula of the mineral is: $(Ce^{4+}, Ce^{3+}, La^{3+}, Ca, Al, Fe^{2+}, Fe^{3+})_5(SiO_4, PO_4)_3O_{12}(OH)$.

3. A characteristic feature of the amorphous mineral is its purely cerium *REE* composition.

4. Both primary and secondary *REE* minerals inherited by the clay from crystalline rocks could have served as the source for the formation of the Ce-bearing phase aggregate. The most likely hypothesis involves the leaching of Ce^{3+} ions adsorbed on the surface of clay minerals, their oxidation to Ce^{4+}, and subsequent precipitation by phosphorus according to the nodule formation scheme.

5. The migration of Ce in its oxidised form led to its separation from the rest of the *REE*, which determined the purely cerium composition of the phosphatosilicate nodules.

6. The discovered Ce mineral can serve as a “marker” for prospecting rare-earth mineralisation in clayey sediments.

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В.М. Бельський [1], https://orcid.org/0000-0001-7990-1386
Г.О. Кульчицька [1], https://orcid.org/0000-0002-7206-4797
Д.С. Черниш [1, 2], https://orcid.org/0000-0001-5390-2591
Л.І. Канунікова [1], https://orcid.org/0000-0002-7530-969X

[1] Інститут геохімії, мінералогії та рудоутворення ім. М.П. Семененка НАН України, Київ, Україна

[2] Інститут відновлюваної енергетики НАН України, Київ, Україна

E-mail: belskyi.vm@gmail.com

ЗНАХІДКА РІДКІСНОЗЕМЕЛЬНОГО МІНЕРАЛУ В АРХЕОЛОГІЧНОМУ АРТЕФАКТІ

Досліджено хімічний і мінеральний склад археологічного артефакту — кераміки бронзової доби з поселення в районі м. Канів. Встановлено, що для її виготовлення використано червону гончарну глину, змішану з піском, імовірно, з відкладів канівської світи палеогену. У кераміці виявлено скупчення розміром ~100 мкм з вмістом Ce_2O_3 36—49 мас. %. Інших рідкісноземельних елементів, за винятком незначної кількості оксиду La (1 %), майже немає. У межах скупчення виділяються фази з гексагональними обрисами, які візуально здаються однорідними, що підтверджує також відносно рівномірний розподіл інших компонентів — оксидів Si, Al, P, Fe і Ca. Фази, ідентифіковані як фосфатосилікат церію й алюмінію з емпіричною формулою $Ce_{1,78}La_{0,03}Ca_{0,24}Al_{1,29}Fe_{0,43}Si_{2,65}P_{0,58}O_{12,3}$, не мають аналогів серед відомих мінеральних видів. За співвідношенням компонентів фази одночасно наближені до видів у групах як аланіту, так і бритоліту, і за такого співвідношення компонентів можуть бути лише аморфним мінералом. Розглянуто різні можливі джерела Ce для утворення мінералу. Найімовірніше, фосфатосилікат утворився ще в глині. Фізично адсорбовані на глинистих мінералах іони Ce легко переходили в розчин і осаджувалися сполуками фосфору за схемою жовноутворення. Міграція в окиснювальних умовах у формі Ce^{4+} зумовила відокремлення елемента від інших тривалентних рідкісноземельних елементів.

Ключові слова: *глина, церій, адсорбція рідкісноземельних елементів, канівська світа, кераміка.*