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HYDROMETALLURGICAL PROCESSING OF BRONZE PRODUCTION WASTE WITH SELECTIVE EXTRACTION OF COPPER AND ZINC

S.T. Yermukhanova*[1], D.S. Nazarova[1], F.Zh. Akhmetova[1],
M. Zh. Amangeldiyev[2], Sh. Ye. Atuova[1]

[1] Zhangir Khan West Kazakhstan Agrarian and Technical University, Uralsk, Kazakhstan
[2] «West Kazakhstan Machine Building Company» JSC, Uralsk, Kazakhstan

**Corresponding author e-mail: svetok_88-88@mail.ru*

Abstract: This article presents a study on the selective extraction of copper and zinc compounds from metallurgical bronze production slags using a hydrometallurgical approach. The relevance of the work is associated with the depletion of mineral resources and the increasing environmental burden of bronze production slags. The aim of the study was to evaluate efficient and environmentally friendly methods for recovering copper and zinc from these slags. Leaching experiments were conducted using nitric acid, ammonia solutions, and sulfuric acid, and their efficiencies were compared. The chemical and phase compositions of the slags were determined using inductively coupled plasma optical emission spectrometry, infrared spectroscopy, and X-ray phase analysis. The "hot leaching" method with 25% sulfuric acid at 80°C was applied for the selective separation of copper and zinc. The results demonstrated that sulfuric acid provides the most economical and environmentally effective separation. The process yielded tin precipitate, high-purity metallic copper via chemical and electrochemical methods, and zinc-rich solid residues. The proposed technological scheme reduces metal consumption, enhances the recyclability of bronze slags, and offers a practical, resource-saving approach to processing metallurgical waste.

Key words: bronze production slag, hydrometallurgical processing, sulfuric acid leaching, electrodeposition of metals, resource-saving technology.

Yermukhanova Svetlana Tasbolatovna	*Senior Lecturer; Email: svetok_88-88@mail.ru*
Nazarova Dauriya Sagyndykovna	*Senior Lecturer; Email: nazarova.ria@mail.ru*
Akhmetova Firuza Zhantaskyzy	*PhD; Email: firuza.92@mail.ru*
Amangeldiyev Meirambek Zhumamuratuly	*Engineer; Email: mejrambekamangeldiev@gmail.com*
Atuova Shugula Yelemeskyzy	*Master's student; Email: atuovashhh03@mail.ru*

1. Introduction

The rapid development of technology has significantly expanded the applications of non-ferrous, rare, and precious metals, as well as their alloys. However, the extraction of these metals from ore raw materials presents several complex challenges. In particular, ore reserves of many metals are limited and

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irreplaceable, the metal content in mineral raw materials is decreasing, large capital investments are required for developing new deposits, and bronze production slag from industrial operations poses a negative environmental impact. These factors collectively hinder the expansion of metal production [1].

Non-ferrous and ferrous metals are present in large quantities in industrial bronze slag from the mining and metallurgical industries. Due to the lack of economical and environmentally friendly technologies for processing and recycling, these secondary raw materials are often stored and accumulated [2]. Comprehensive processing of bronze slag, aiming for maximal extraction of valuable components, is particularly important when it contains non-ferrous and rare metals, given the diverse composition of secondary raw materials [3,4]. A clear classification of secondary bronze slag is necessary to ensure efficient processing and utilization of metal-containing materials [5].

Bronze slag containing precious metals can be classified according to its origin: either as slag from the production of a specific product or as slag formed during a particular process. Non-ferrous metals in the original raw materials may be present in metallic form, as simple or complex oxides, or in various salts, among which sulfates, chlorides, chalcogenides, and arsenides are most common, with sulfides being particularly representative [6-9]. Typical raw materials with metals in elemental form include ores containing native metals (gold, silver, platinum group metals, and less commonly copper), secondary raw materials, and semi-finished products such as cementation precipitates and cinders after reducing roasting [10-13].

The chemical properties of electronegative metals (e.g., zinc, cadmium, aluminum) determine their high solubility in acidic and alkaline solutions:

$$Me + nH^{+} \rightarrow Me^{n+} + \frac{n}{2}H_2$$

$$Me + 2nOH^{-} \rightarrow MeO_{n/2} + \frac{n}{2}H_2$$

Aeration is used to prevent the accumulation of explosive hydrogen concentrations. Electropositive metals dissolve only in the presence of both a solvent and an oxidizing agent. When the solvent has complexing or acidic properties and exhibits oxidizing activity (or when a mixture is used), the leaching process is significantly accelerated:

$$Cu + 2H^{+} + 0.5O_2 \rightarrow Cu^{2+} + H_2O$$

$$Ni + nNH_3 + 0.5O_2 + H_2O \rightarrow [Ni(NH_3)_n]^{2+} + nOH^{-}$$

Certain metals (e.g., nickel, cobalt) are prone to passivation in air due to the formation of oxide films; therefore, leaching efficiency is higher for freshly reduced cinders [14,15]. The dissolution rates of alloys decrease in the presence of components that form insoluble films. The most rational method to prepare metallized raw materials for leaching is melting and spraying the melt, which removes non-metallic inclusions and ensures homogeneity in chemical and phase composition, as well as a developed surface area [16].

In this study, the object of investigation was bronze slag obtained after smelting operations at a machine-building enterprise. The material represents a heterogeneous metallurgical residue consisting of oxidized phases and residual metallic inclusions. Throughout the manuscript, the term “slag” refers exclusively to this material.

Bronze slag was obtained from [Company/Plant]. Chemical reagents included sulfuric acid (H_2SO_4, 98%), hydrogen peroxide (H_2O_2, 30%), and deionized water. Copper cathodes (99.9%) and lead or chrome-plated steel anodes were used for electrolysis.

2. Experimental part

The main alloying element in bronze is tin, while in brass it is zinc; however, both alloys are copper-based. Depending on their composition, bronzes may be tin-containing or tin-free, whereas brasses may be binary or multicomponent alloys. Copper–tin bronzes can be produced by various methods, including smelting, co-smelting, and cementation.

Due to the depletion of high-grade mineral resources, complex and low-grade ores, as well as their processing products (slags, cakes, dusts, and sublimates), are increasingly involved in metallurgical production. In the present study, slag obtained from bronze smelting at a machine-building enterprise was used as the raw material.

To determine the chemical composition, the initial samples were crushed and divided into two fractions: crushed material (sample 1a) and uncrushed metallic phase (sample 1b). Photographs of the samples are shown in Figure 1.

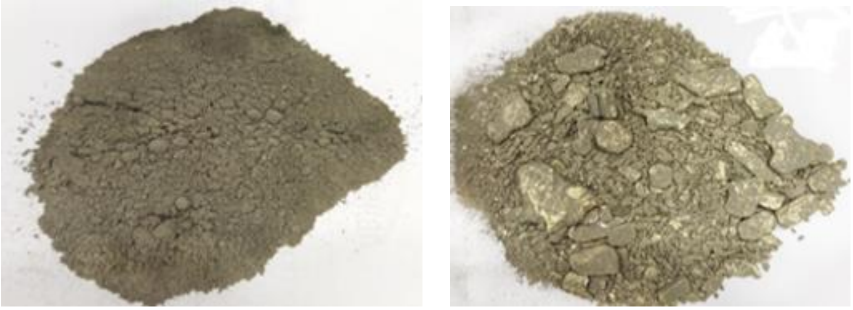

Figure 1 – Bronze slag samples: 1a – abraded and 1b – non-abraded (metal phase).

The compositional differences between the crushed (1a) and uncrushed (1b) fractions confirm the heterogeneous distribution of metallic phases in the slag. Therefore, preliminary particle-size classification and controlled grinding are important steps in the processing scheme. Finer fractions enriched in copper are more suitable for oxidative sulfuric acid leaching, while coarser fractions containing zinc-rich phases may require additional grinding or thermal treatment to improve metal accessibility.

The chemical and phase compositions of the slag were determined using inductively coupled plasma optical emission spectrometry (ICP-OES), infrared spectroscopy, and X-ray phase analysis (XRD).

Based on the analytical results, a technological scheme for the extraction of copper and zinc was developed (Figure 2).

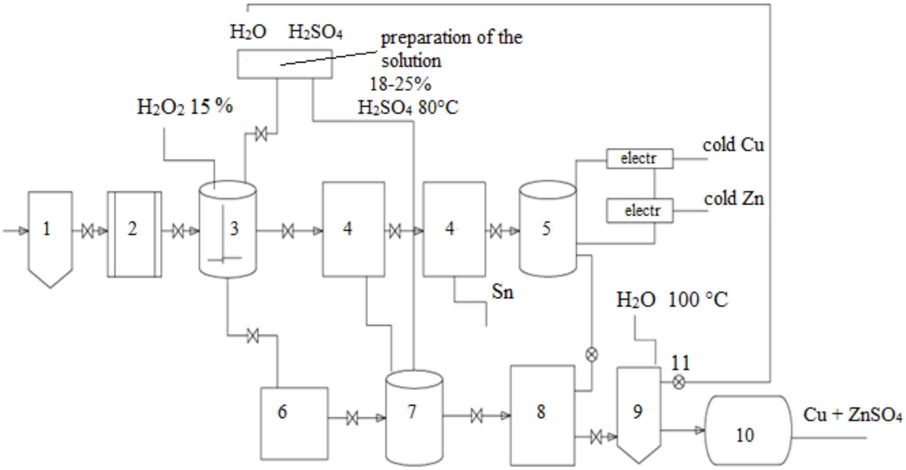

Figure 2 – Hot leaching flow chart for bronze bronze production slag: 1 – classifier, 2 – calcination furnace, 3 – leaching reactor, 4 – filter, 5 – electrolyte collector, 6 – cake collector, 7 – leaching reactor, 8 – filter, 9 – rinse tank, 10 – dryer, 11 – circulation pump.

The process for extracting copper and zinc from bronze slag included the following stages:

Classification of slag to a particle size not exceeding 0.315 mm.

Calcination at 600°C for 8 hours to remove organic impurities.

Leaching in a reactor with 18–25% sulfuric acid at 80°C for 1 hour under constant stirring (400 rpm). After 20 minutes, 9–15% hydrogen peroxide was added as an oxidizing agent. Hydrogen peroxide oxidizes Fe^{2+} to Fe^{3+}:

$$2Fe^{2+} + H_2O_2 + 2H^+ \rightarrow 2Fe^{3+} + 2H_2O$$

Under strongly acidic conditions (25% H_2SO_4), Fe^{3+} remains soluble; therefore, hydrogen peroxide primarily promotes oxidative dissolution of copper rather than iron precipitation.

After leaching, the suspension was allowed to settle for 20–30 minutes at 80°C. Metallic copper formed via cementation was separated by hot filtration.

The primary filter cake was retained, while the filtrate was allowed to cool for 30–40 minutes, resulting in tin precipitation. The tin precipitate was filtered, washed with water, and dried at 80°C.

The primary cake was returned to the reactor for secondary leaching with fresh 18–25% sulfuric acid. The deep blue coloration of the solution indicated further copper dissolution. After refiltration, the secondary filtrate was collected for electrolyte preparation.

The concentrations of free acid, copper sulfate, and zinc sulfate were monitored and adjusted prior to electrolysis. Wash waters were recycled back to the leaching stage.

The electrolyte was prepared from the filtrate containing copper and zinc sulfates in 18–25% H_2SO_4 and adjusted to 80 g/L Cu^{2+} and 20 g/L Zn^{2+}.

Electrolysis was performed at a current density of 5 A/dm^2 using a copper cathode and a lead or chrome-plated steel anode for 1 hour at room temperature.

Selective copper deposition occurred due to the difference in standard reduction potentials:

$$E^0 (Cu^{2+}/Cu) = +0.34 \text{ V}$$
$$E^0 (Zn^{2+}/Zn) = -0.76 \text{ V}$$

Under these conditions, copper was deposited on the cathode, while zinc remained in solution. The cathode deposit contained 98.5% Cu with minor Zn and Sn impurities (<1%), confirming high copper purity. If higher copper purity is required, additional purification steps may be applied. These include electrolyte purification using solvent extraction or ion-exchange methods to remove trace metal ions, as well as secondary electrorefining under optimized electrochemical conditions. Such approaches are widely used in hydrometallurgical processing and can increase the purity of cathode copper to industrial standards.

The zinc-rich solution may be further processed for zinc recovery.

All leaching experiments were performed in triplicate. Extraction efficiencies are presented as mean values ± standard deviation. The analytical error of the ICP-OES method was ±0.5%, confirming reproducibility and reliability of the results.

3. Results and discussion

The chemical composition of the combined bronze slag sample showed the following contents (wt.%): TiO_2 – 0.07; MnO – 0.46; Na_2O – 0.53; K_2O – 0.14; SiO_2 – 2.66; MgO – 0.93; CaO – 4.40; Fe_2O_3 – 1.94; Al_2O_3 – 2.82; P_2O_5 – <0.1; Zn – 28.49; Ni – 0.38; Cu – 55.69; Cr – 0.087; W – 0.006.

The results indicate the predominance of copper and zinc as the principal metallic components, while other oxides and alloying elements are present in minor quantities.

Solutions obtained after microwave digestion of crushed (1a) and uncrushed (1b) samples in a mixture of hydrochloric and nitric acids were analyzed by ICP-OES. Chemical analysis showed that in sample 1a, zinc (Zn) was 17.08%, nickel (Ni) 0.37%, copper (Cu) 72.97%, chromium (Cr) 0.036%, and tungsten (W) 0.007%. In sample 1b, zinc was 25.63%, nickel 0.26%, copper 41.57%, chromium 0.027%, and tungsten 0.005%. These results indicate that sample 1b contains a higher zinc content and a lower copper content, reflecting the heterogeneity of the slag and uneven distribution of its phases.

Sample 1b contains significantly more zinc and less copper than sample 1a, indicating heterogeneity of the slag and uneven phase distribution.

X-ray diffraction suggested the possible presence of minor phases containing Ge, Se, and Co. However, chemical analysis showed that these elements were either below the detection limit or present only in trace amounts.

Therefore, identification of phases such as Cu_3Ge and Co- or Se-containing compounds should be considered tentative. Their appearance in the diffractogram may be explained by:

- trace micro-impurities,
- overlapping diffraction peaks,
- limitations of automatic database matching (PDF/ICDD).

Phase identification was refined by prioritizing phases consistent with the bulk Cu–Zn–Sn composition. Minor phases were interpreted cautiously.

Component composition of bronze samples:

- Without calcination: Cu 54%, Zn 34%, Sn 5%, Pb 6%;
- Calcined 1 h: Cu 31%, Zn 56%, Sn 1.5%, Pb 4%;
- Calcined 8 h: Cu 26%, Zn 62%, Sn 1.3%, Pb 4%.

Prolonged calcination leads to redistribution of metallic components and relative zinc enrichment.

Sulfuric acid was selected as the leaching agent because it forms soluble sulfates of copper and zinc, while avoiding formation of poorly soluble by-products.

Since bronze is an alloy, dissolution must be considered as independent reactions of its components.

Zinc dissolves readily:

$$Zn + H_2SO_4 \rightarrow ZnSO_4 + H_2\uparrow$$

Tin may react as:

$$Sn + H_2SO_4 \rightarrow SnSO_4 + H_2\uparrow$$

Copper does not dissolve in non-oxidizing sulfuric acid. In the presence of hydrogen peroxide:

$$Cu + H_2O_2 + H_2SO_4 \rightarrow CuSO_4 + 2H_2O$$

Thus, zinc dissolves through a direct acid attack, reacting readily with sulfuric acid to form soluble zinc sulfate and release hydrogen gas. In contrast, copper dissolves via an oxidative mechanism, requiring the presence of an oxidizing agent such as hydrogen peroxide, which enables the formation of soluble copper sulfate.

If Cu^{2+} forms, cementation may occur:

$$Cu^{2+} + Zn \rightarrow Cu\downarrow + Zn^{2+}$$

Therefore, metallic copper powder observed during leaching is attributed to cementation rather than direct acid dissolution.

Leaching was performed at 80°C in 25% H_2SO_4 under constant stirring.

Figure 3 shows the solid products obtained.

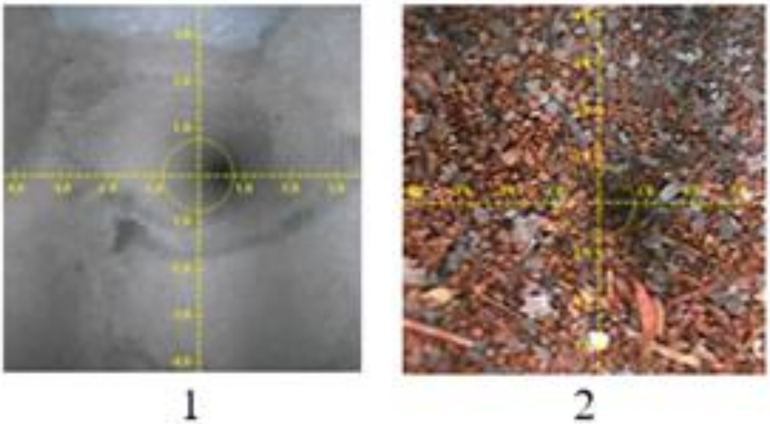

Figure 3 – Precipitates formed during leaching of bronze slag with sulfuric acid:
1 – tin powder formed after leaching bronze with sulfuric acid and hydrogen peroxide;
2 – metallic copper obtained after leaching bronze slag with sulfuric acid without hydrogen peroxide.

Metallic copper formed as a brown powder with minor zinc inclusions, as confirmed by XRD.

The study demonstrates that copper and zinc are the dominant components of bronze slag, while the particle size and thermal treatment of the material significantly influence the distribution of its phases. Oxidative leaching with sulfuric acid allows for the selective dissolution of copper, and the formation of metallic copper can be explained by the cementation process. Overall, the hot leaching method provides an effective and practical approach for obtaining high-purity copper, tin precipitates, and zinc-rich residues.

These findings establish a direct relationship between compositional analysis and the development of a resource-saving hydrometallurgical process for bronze slag recycling.

4. Conclusion

The results of this study showed that bronze slag primarily consists of copper (Cu) and zinc (Zn), with other impurities present in minor amounts. The multicomponent structure of the slag significantly influences the leaching rate, as different metals respond differently to chemical treatment. When ammonium hydroxide and nitric acid were tested as leaching agents, several limitations were observed. In ammoniacal systems, copper dissolution occurs through the formation of ammine complexes, which requires strict control of pH and ammonia concentration. In addition, ammonia volatilization may lead to atmospheric emissions and requires additional gas-capture systems. Nitric acid, although effective as an oxidizing agent, produces nitrogen oxide gases (NOx) during the leaching process, which require catalytic gas-treatment systems and increase operational costs. In contrast, sulfuric acid proved to be a much more efficient reagent, enabling selective extraction of copper and zinc while minimizing the formation of by-products.

To evaluate the efficiency of metal recovery, a material balance was established for copper, zinc, and tin. The results indicated that 81% of the total copper in the slag was recovered as a chemical precipitate, 70% of zinc remained in the solid residue after leaching, 59% of tin was obtained as a precipitate, and electrochemically deposited copper reached a purity of 98.5%. A total material balance accounting for all phases, including losses, chemical precipitates, electrochemical products, and residues, confirmed the transparency and effectiveness of the selective separation process. Furthermore, the washing solution from filtration can be reused to prepare fresh sulfuric acid, enhancing the sustainability of the process. Overall, these findings demonstrate that the proposed hot leaching method allows for the effective, resource-saving, and environmentally safe recovery of copper and zinc from metallurgical bronze production slag.

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Conflict of Interest: The authors declare no conflicts of interest that require disclosure in this paper.

ҚОЛА ӨНДІРІСІНІҢ ҚАЛДЫҚТАРЫНАН МЫРЫШ ҚОСЫЛЫСТАРЫН БӨЛІП АЛУДЫ ЗЕРТТЕУ

С.Т. Ермуханова[1*], Д.С. Назарова[1], Ф.Ж. Ахметова[1], М.Ж. Амангелдиев[2], Ш. Е. Атуова[1]

[1] Жәңгір хан атындағы Батыс Қазақстан аграрлық-техникалық университеті, Орал, Қазақстан
[2] «Батыс Қазақстан машина жасау компаниясы» АҚ, Орал, Қазақстан

Түйіндеме. Бұл мақала қола мен жезді өндіру кезінде пайда болған металлургиялық қалдықтардан мырыш пен мыс қосылыстарын гидрометаллургиялық әдіспен селективті бөлу технологиясын зерттеуге арналған. Зерттеудің өзектілігі минералды шикізаттың азаюымен және металлургиялық қалдықтардың қоршаған ортаға тигізетін экологиялық жүктемесінің артуымен байланысты. Жұмыстың мақсаты - қола мен жез шлактарын өңдеу кезінде мыс пен мырышты тиімді және экологиялық таза жолмен алу мүмкіндігін анықтау. Зерттеу барысында азот қышқылы, аммиак ерітінділері және күкірт қышқылы қолданылды, олардың шаймалау тиімділігі салыстырмалы түрде бағаланды. Шлактардың химиялық және фазалық құрамы индуктивті байланысқан плазмалық оптикалық эмиссиялық спектрометрия, инфрақызыл спектроскопия және рентгендік фазалық талдау арқылы анықталды. Жаңа әдістемелік тәсіл ретінде 80 °C температурада 25% күкірт қышқылын қолданатын «ыстық шаймалау» әдісі қолданылды. Зерттеу нәтижелері күкірт қышқылының мыс пен мырышты бөлуде экономикалық және экологиялық тұрғыдан тиімдірек екенін көрсетті. Бұл процесс қалайы тұнбасының, химиялық және электрохимиялық жолмен алынған жоғары таза металл мысының және мырышқа бай қатты қалдықтардың пайда болуына әкелді. Ұсынылған технологиялық схема металл шығынын азайтады, қалдықтарды қайта өңдеу мүмкіндігін арттырады және аз қалдықты, ресурстарды үнемдейтін металлургиялық технологияларды әзірлеу үшін практикалық маңызға ие.

Түйінді сөздер: металлургиялық қалдықтар, гидрометаллургиялық әдіс, күкірт қышқылымен шаймалау, металдарды электродтау, ресурс үнемдеуші технология.

Ермуханова Светлана Тасболатовна	*аға оқытушы*
Назарова Даурия Сағындыковна	*аға оқытушы*
Ахметова Фируза Жантасқызы	*PhD*
Амангелдиев Мейрамбек Жұмамұратұлы	*инженер*
Атуова Шұғыла Елемесқызы	*магистрант*

ИССЛЕДОВАНИЕ ВЫДЕЛЕНИЯ СОЕДИНЕНИЙ ЦИНКА ИЗ ОТХОДОВ ПРОИЗВОДСТВА БРОНЗЫ

С.Т. Ермуханова[1*], Д.С. Назарова[1], Ф.Ж. Ахметова[1], М.Ж. Амангелдиев[2], Ш. Е. Атуова[1]

[1] Западно-Казахстанский аграрно-технический университет имени Жангир хана,
Уральск, Казахстан
[2] АО «Западно-Казахстанская машиностроительная компания», Уральск, Казахстан

Резюме. Данная статья посвящена изучению технологии селективного разделения соединений цинка и меди из металлургических отходов, образующихся при производстве бронзы и латуни гидрометаллургическим методом. Актуальность исследования связана с истощением минерального сырья и возрастающей экологической нагрузкой металлургических отходов. Цель работы – определить возможность эффективного и экологически безопасного получения меди и цинка при переработке бронзовых и латунных шлаков. В ходе исследования использовались азотная кислота, растворы аммиака и серная кислота, а также проводилась сравнительная оценка их эффективности выщелачивания. Химический и фазовый состав шлаков определялся методами оптической эмиссионной спектрометрии с индуктивно связанной плазмой, инфракрасной спектроскопии и рентгенофазового анализа. В качестве нового методического подхода использовался метод «горячего выщелачивания» с применением 25% серной кислоты при температуре 80 °С. Результаты исследования показали, что серная кислота является более экономически и экологически эффективной при разделении меди и цинка. В результате процесса образовался осадок олова, химически и электрохимически извлеченная высокочистая металлическая медь, а также твердые отходы, богатые цинком. Предложенная технологическая схема снижает расход металла, увеличивает возможности переработки отходов и имеет практическое значение для развития малоотходных, ресурсосберегающих металлургических технологий.

Ключевые слова: металлургические отходы, гидрометаллургический метод, выщелачивание серной кислотой, электроосаждение металлов, ресурсосберегающая технология.

Ермуханова Светлана Тасболатовна	*старший преподаватель*
Назарова Даурия Сагындыковна	*старший преподаватель*
Ахметова Фируза Жантасовна	*PhD*
Амангелдиев Мейрамбек Жумамуратович	*инженер*
Атуова Шугыла Елемесовна	*магистрант*

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