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Extraction of selenium from substandard non-ferrous metallurgical feedstock

Abstract: In this paper, an innovative process for the extraction of selenium from substandard selenium-containing raw materials obtained at copper smelters is proposed. The process has been tested on an enlarged scale under the developer's conditions and implemented on an industrial scale for the first time in the Republic of Kazakhstan. The implementation of this development has enabled the Republic of Kazakhstan to transition from a country reliant on raw materials to a new level: a country producing high-tech products in demand on all global metal markets. This work was awarded a high Government award in 2025 – the Al-Farabi Prize in Science and Technology.

Keywords: selenium, refining, rough selenium, vacuum distillation, introduction, impurities, purification.

Introduction

The use of selenium by humankind has a history spanning more than one hundred and thirty years (Kudryavtsev, 1961). Selenium has found extensive applications in radio electronics, phototechnology, metallurgy, the rubber industry, organic synthesis, and glass production (Analytical review, 2018). Selenium with a main-component content of no less than 99.5%, corresponding to grade ST1 (GOST 10298-2018, 2018), is in particularly high industrial demand and is primarily intended for export.

According to USGS estimates (U.S. Geological Survey, 2019), global selenium consumption is distributed as follows: 40% in metallurgy (including manganese production), 25% in glass manufacturing, 10% in agriculture, 10% in the production of chemicals and pigments, 10% in the electronics industry, and 5% in other sectors. The main sources of selenium production include the roasting of waste from sulfuric acid plants, pulp-and-paper mills, and the processing of anode slimes from copper electrorefining plants (Kudryavtsev, 1961). The selenium dioxide (SeO_2) generated in these processes is reduced with sulfur dioxide (SO_2) to elemental selenium, which is then upgraded to commercial-grade material by recrystallization, vacuum distillation, hydride-based purification, and other methods (Volodin & Trebukhov, 2017).

The high reactivity of selenium complicates its purification from most impurities. To address this issue, various technological schemes and industrial equipment have been developed to improve both the quality and the quantity of produced technical selenium (Kenzhaliev et al., 2018).

The Republic of Kazakhstan is one of the world's major regions possessing significant reserves of rare and rare-earth metals (Rakishev, 2016). With the growing production of solar cells, laser systems, LED lamps, and photodetectors, selenium has become a strategically important raw material. The primary producers of technical selenium in Kazakhstan are Kazakhmys Corporation LLP and Kazzinc LLP. However, these major metallurgical enterprises obtain selenium in non-commercial form as a by-product of their primary operations; its value is nearly an order of magnitude lower than that of commercial-grade selenium, which plays a critical role in the semiconductor and high-tech industries (Kenzhaliev et al., 2018; Dodonov, 2011).

Current global economic conditions demand the production of high-value products with significant added value. The collapse in global copper prices (Naumov & Naumova, 2010) contributed to the establishment in 2022 of a selenium refining unit at the production site of Kazakhmys Progress LLP (Balkhash, Republic of Kazakhstan), processing crude selenium produced at the Balkhash Smelting Plant of Kazakhmys Smelting LLP.

The main part of the research

The Laboratory of Vacuum Processes of the "Institute of Metallurgy and Ore Beneficiation" JSC at Satbayev University (Almaty, Republic of Kazakhstan) has developed a vacuum-distillation technology for crude selenium refining using vapor filtration (Innovation Patent No. 28695, 2013; Innovative patent No. 27273, 2012; Patent No. 37275, 2023).

The Republic of Kazakhstan is one of the world's largest producers of technical-grade selenium, a material exhibiting semiconductor and optical properties. These unique characteristics have enabled selenium to be widely used in the production of semiconductors and solar cells, driving a growing global demand for high-purity selenium. At the facilities of Kazakhmys Smelting LLP, using a technology developed by the JSC "Institute of Metallurgy and Ore Beneficiation" of Satbayev University, the first industrial-scale production of refined selenium from crude selenium feedstock has been established in Kazakhstan (Kazakhmys, 2025).

The production capacity is 70 t/year, with the potential to increase throughput to 140 t/year. The process is environmentally clean (carried out in sealed equipment that prevents toxic emissions), reagent-free (based on the physical properties of selenium), and allows the recovery of up to 2 kg of gold and up to 60 kg of silver annually that were previously lost during the sale of crude selenium (Table 1).

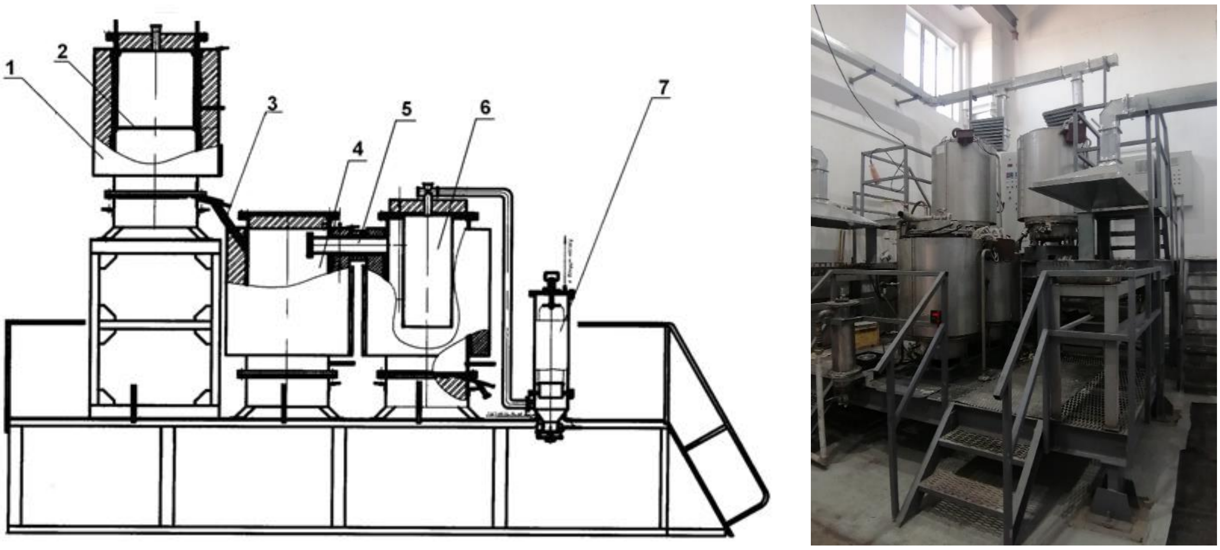
Table 1. The dynamics of global selenium prices over the years:

Year	2004	2006	2010	2014	2017	2024
Price in USD per kg	4.0	42.0	165.0	85.0	60.0	38.0

The present work provides the results of technological trials of this process and equipment.

The bulk density of dried crude selenium is 1.7 g/cm³ without tapping and 2.12 g/cm³ with tapping; the angle of repose is 47°. X-ray fluorescence analysis using an Axios PANalytical wavelength-dispersive spectrometer determined the following elemental composition, wt. %: 89.511 Se; 2.032 O; 1.594 S; 0.766 Mg; 0.759 Na; 1.684 Pb; 0.311 Al; 1.011 Te; 1.595 Cl; 0.101 Ag; 0.109 Si; 0.199 Sb; 0.131 Cu; 0.112 Fe; 0.003 P; 0.082 As. X-ray diffraction analysis performed on a D8 Advance (BRUKER) diffractometer with Co radiation identified the following major phases in the crude selenium: Se (Selenium); $AsSe_9Cu_{0,05}$ (Copper Arsenic Selenide); CSe_2 (Carbon Selenide).

The technological scheme for producing commercial-grade selenium at Kazakhmys Progress LLP includes preliminary washing of crude selenium to neutral pH to remove residual acidity, followed by drying in a vacuum oven at 95-105 ℃ and a residual pressure of 40.0 kPa until the moisture content falls below 3%. The material is then fed into a melting furnace, where residual moisture is removed, and the material undergoes zone refining (seigeration) with melt filtration through a metal mesh at 460-480 ℃ under atmospheric pressure. The remelted crude selenium is delivered via heated pipelines to the evaporator of the vacuum distillation unit. Distillation is conducted at 440-460 ℃ in the evaporator and 160-180 ℃ in the condenser under a residual pressure of 0.13-0.4 kPa with continuous vacuum pumping. The refined selenium is cast into molds at atmospheric pressure, producing ingots weighing 4.8-5.5 kg. The dry distillation residue, enriched in precious metals, is returned to the head of the process for treatment in the Kaldo furnace. The figure shows the apparatus chain of the vacuum distillation unit and a photograph of the industrial selenium-refining area.



a) b)

a) Circuit diagram of the apparatus: 1 - intake melting furnace; 2 - "false bottom" in the furnace; 3 - heated pipeline; 4 - evaporator; 5 - steam line; 6 - condenser; 7 - bag filter; b) - photo of the production site.

Figure 1. Circuit diagram of a vacuum distillation unit for refining rough selenium and a photograph of the production site of Kazakhmys Progress LLP for refining rough selenium in Balkhash

To determine the content of the main and impurity elements, a sample of remelted selenium taken after the melting furnace was analyzed by X-ray fluorescence. The composition, wt. %, was: 95.611 Se; 1.639 O; 0.371 S; 0.238 Mg; 0.296 Na; 0.465 Pb; 0.077 Al; 0.621 Te; 0.516 Cl; 0.011 Ag; 0.071 Si; 0.032 Sb; 0.021 Cu; 0.031 Fe. The yield of remelted, filtered selenium was ~74%, while slag constituted ~24%. The material balance discrepancy of - 0.35% is attributed to the relatively constant quantity of selenium retained as skull (garnisage) on the bottom of the melting furnace. Selenium distribution across the process products was as follows: 79.9% of selenium transferred into the melt (and subsequently into the vacuum distillation unit), and 16.4% remained in the slag.

The silver content in products after seigeration was determined by XRF analysis and amounted to: 0.393 wt. % in slag and 0.011 wt. % in remelted selenium. Thus, 91.76% of the silver contained in crude selenium was transferred into the slag.

As a result of vacuum distillation of the remelted crude selenium, a metal containing 99.54% of the main component was obtained, corresponding to grade ST1 according to GOST 10298–2018 [3], suitable for export. XRF analysis of the refined selenium ingots showed the following composition, wt. %: 99.543 Se; 0.217 O; 0.003 S; 0.003 Pb; 0.021 Mg; 0.007 Ni; 0.005 Al; 0.122 Te; 0.005 Cl; 0.003 Si; 0.031 Sb; 0.033 Cr; 0.002 Cu; 0.005 Fe. The yield of refined selenium was 96.07%. Selenium recovery to the raffinate was 98.96%. No selenium was detected in the particulate residue after distillation, whereas the silver content in this residue was 2.05 wt. %.

Additional recovery of selenium from melting-furnace slags through vacuum distillation increases its overall extraction to 94.1%. However, this significantly increases energy consumption and, consequently, processing costs. Therefore, melting-furnace slags, which constitute ~20% of the output and contain considerable amounts of precious metals, are fed to the head of the process - the Kaldo furnace - for further selenium extraction. This technological approach ensures more stable operation of vacuum equipment and reduces losses of selenium and noble metals across the range of intermediate products formed during the processing of commercial-grade crude selenium (secondary sublimates and residues).

Conclusions

The industrial-scale implementation of this work has enabled the Republic of Kazakhstan to produce refined selenium with a purity exceeding 99.9%, which is in high demand across global metal markets. In early 2025, more than 100 tonnes of refined selenium were exported. At present, efforts are underway to produce ultra-high-purity selenium with a purity level above 99.99%.

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