DEVELOPMENT OF A LABORATORY SYSTEM FOR GOLD AND SILVER PHASE ANALYSIS AND DISTRIBUTION ASSESSMENT

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Abstract

This paper presents the design, manufacturing, and commissioning of a specialized laboratory installation for gold and silver phase analysis. The developed system provides increased processing capacity, improved homogenization geometry of chemical solutions, and enhanced sample representativeness. The installation reduces electricity consumption, improves productivity through the application of high-pressure filtration and closed washing systems, and automates key laboratory procedures, thereby minimizing operator-related subjectivity. The proposed laboratory workflow enables continuous monitoring of precious metal phase distribution in ores and processing products, supporting data-driven technological decisions aimed at increasing recovery and concentrate quality.

Keywords: *gold, silver, phase analysis, laboratory equipment, automation, mineral processing*

1. INTRODUCTION

Phase analysis of gold and silver is a key laboratory method used to determine the forms of precious metal occurrence in ores and mineral processing products, including free particles, cyanidable forms, oxide-coated phases, sulfide-associated gold, and finely dispersed gold. This information is essential for selecting appropriate beneficiation routes, optimizing technological parameters, and minimizing metal losses during processing (Gül et al., 2012; Surimbayev et al., 2024).

Reliable phase analysis is part of an integrated approach to ore characterization that begins at the exploration stage. Geological and geophysical investigations provide essential information on ore body structure, lithological variability, and zones of mineralization, thereby reducing geological uncertainty and supporting representative sampling strategies (Grigorova, 2020; Tomova, 2024). Geophysical methods such as electrical resistivity tomography and other structural imaging techniques contribute to improved understanding of ore heterogeneity, which is a key factor influencing both laboratory test representativeness and subsequent processing performance.

Numerous studies have shown that gold recovery efficiency depends not only on total metal grade but also on the mineralogical mode of occurrence and degree of liberation of precious metals (Benzaazoua et al., 2007; Chen et al., 2018). As a result, phase analysis serves as a critical link between geological–geophysical characterization and technological decision-making. By identifying the dominant forms of gold and silver occurrence, phase analysis provides the experimental basis for selecting appropriate beneficiation routes and for predicting potential metal losses during processing.

Despite its importance, conventional laboratory phase analysis is associated with several practical limitations. Traditional workflows involve multiple manual operations, large volumes of aggressive chemical solutions, long processing times, and extensive washing steps between analytical stages. These factors increase labor intensity, energy consumption, and the risk of sample contamination or loss of solid material, while also limiting result reproducibility. Furthermore, the use of toxic reagents, particularly cyanide solutions, requires strict safety control and favors closed-system laboratory operation (Hilson and Monhemius, 2006; Han et al., 2024).

In recent years, phase analysis has increasingly been applied not only for primary ore characterization but also as a monitoring tool for tracking gold and silver distribution in flotation concentrates, middlings, and tailings from operating mineral processing plants. Such applications impose additional requirements on laboratory equipment, including the ability to process multiple samples simultaneously, ensure uniform agitation, perform efficient closed washing, and maintain accurate mass balance calculations

for both solid and liquid phases. However, standard laboratory installations are often not designed to meet these requirements, which limits the practical implementation of phase analysis in process optimization and control.

Several authors have emphasized the need for methodological and technical improvements in laboratory-scale mineral processing studies, particularly with respect to automation, reproducibility, and operator safety (Gökelma et al., 2016; Liu et al., 2022). In this context, the development of specialized laboratory equipment tailored to the specific requirements of gold and silver phase analysis represents a necessary step toward improving analytical efficiency and data reliability.

The aim of the present study is to describe the design, manufacturing, and commissioning of a specialized laboratory system for gold and silver phase distribution analysis. The proposed installation integrates improved workflow solutions, including high-pressure filtration, closed washing, automated agitation of up to 20 samples, and enhanced process control. The developed approach increases laboratory productivity, improves analytical accuracy and reproducibility, reduces reagent consumption and energy costs, and provides reliable data that link geological-geophysical characterization with technological decision-making in mineral processing operations.

2. MATERIALS AND METHODS

The methodology adopted in Bulgaria for determining the phase distribution of gold and silver in ores and flotation products consists of four sequential stages, in which the metals are classified according to their form of occurrence: free particles, cyanidable phases, oxide-coated forms, sulfide-associated gold and silver, and finely dispersed phases (Fig. 1). The reagents used for phase analysis include H_2O_2, Hg, NaCN or KCN, HCl, HNO_3, $SnCl_2$, and NaOH.

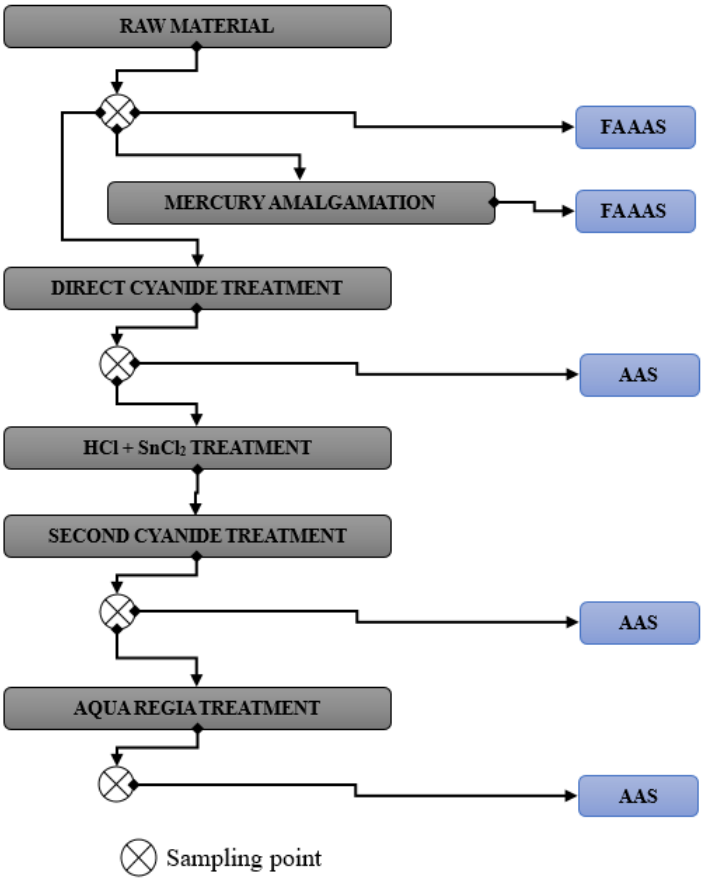

Fig. 1. Gold and silver phase analysis flowchart

The first stage of phase analysis involves mercury amalgamation to determine the content of free gold and silver particles. A dominant proportion of free precious metal particles indicates the suitability of gravity concentration as a primary beneficiation method.

Gravity concentration techniques exploit the significant density difference between gold (16-19.3 g/cm³) and the associated ore and gangue minerals (Surimbayev et al., 2024). These methods are most effective for relatively coarse particle sizes, with recovery efficiency increasing with particle size up to an optimum value, beyond which recovery may decline (Izerdem and Ergun, 2024). Gravity concentration is environmentally favorable and cost-effective, but it is generally applicable to particle sizes larger than 10 μm (Surimbayev et al., 2024). For free gold particles exceeding 125 μm, recovery efficiency may decrease due to process-related limitations (Surimbayev et al., 2024).

In the second stage, cyanidation is applied to determine the content of non-liberated but cyanidable gold and silver present as inclusions or intergrowths within ore and gangue minerals. Cyanidation is a well-established hydrometallurgical process, accounting for more than 75% of global gold production (Egan et al., 2016; Oraby et al., 2020; Shi, 2024). It was first applied on an industrial scale in New Zealand in 1889 and in South Africa in 1890 (Hilson and Monhemius, 2006). Gold dissolution during cyanidation proceeds according to the following reaction:

$$2Au + 4CN^- + O_2 + 2H_2O \rightarrow 2[Au(CN)_2]^- + 4OH^-$$

Cyanidation efficiency depends on mineralogical characteristics, particle size distribution, grinding fineness, reagent concentration, oxygen availability, pulp pH, agitation intensity, and the presence of impurities. These parameters are typically optimized through preliminary laboratory testing (Hilson and Monhemius, 2006; Egan et al., 2016; Han et al., 2024; Shi, 2024).

Gold recovery during cyanidation increases with higher oxygen and cyanide concentrations in the pulp and with finer ore grinding. Reduced particle size increases the contact surface between the cyanide solution and gold particles, accelerating dissolution. When both fine and coarse gold grains are present, gravity concentration is commonly applied prior to cyanidation. For safe cyanide use, the pulp pH must be maintained above 10, typically by adding calcium hydroxide [$Ca(OH)_2$] or sodium hydroxide (NaOH). At neutral or acidic pH values, cyanide decomposes with the release of highly toxic hydrogen cyanide gas (HCN) (Han et al., 2024), according to the reaction:

$$CN^- + H_2O \rightleftharpoons OH^- + HCN\uparrow$$

In the third stage of phase analysis, gold and silver associated with oxide coatings - primarily composed of iron oxides, hydroxides, and silicate phases are determined. These forms are poorly recovered by flotation and are not readily dissolved during cyanidation. The sample is treated with hydrochloric acid and tin dichloride to remove oxide coatings, followed by secondary cyanidation.

In the final stage, the sample is treated with aqua regia (HCl : HNO_3 = 3:1) to dissolve gold and silver associated with sulfide minerals or incorporated into their crystal lattice. The remaining undissolved portion represents finely dispersed precious metals that are generally considered unrecoverable under conventional processing conditions.

Two parallel analyses are performed for each sample, and the reported result represents the average value. In cases of significant discrepancy between parallel determinations, the analysis is repeated. Gold and silver contents in the initial samples and amalgamation residues are determined by fire assay, while samples with lower precious metal contents are analyzed by acid dissolution followed by atomic absorption spectroscopy.

Special attention is given to washing procedures between successive analytical stages, as insufficient washing may result in reagent carry-over, sample contamination, or the formation of hazardous gases. To address these challenges, improvements to the washing and filtration workflow were introduced, as described below.

When economically justified, finely dispersed gold occurring as very fine inclusions in pyrite and arsenopyrite or incorporated into the crystal lattice of these minerals (refractory or “invisible” gold) may

be recovered using additional processing techniques such as roasting or high-pressure oxidation in autoclaves, followed by flotation (Benzaazoua et al., 2007; Chen et al., 2018).

According to the adopted methodology, duplicate phase analyses are conducted for each sample to ensure reproducibility. After each analytical procedure, samples are prepared, duplicated, and analyzed. When determining cyanidable gold and silver content, a sample is taken from the liquid phase for chemical analysis in order to calculate metal extraction and to monitor free cyanide concentration and solution oxidation state. The residual solid phase is thoroughly washed and used as the starting material for the subsequent analytical stage.

Careless washing may result in the loss of solid material, leading to inaccuracies in phase analysis results. Conversely, insufficient washing may leave residual reagents that can interact with chemicals used in subsequent stages and cause the release of toxic hydrogen cyanide gas (HCN). In the traditional methodology, washing operations are carried out in large non-metallic vessels. For an initial sample mass of approximately 300 g, the minimum water volume required for washing is about 50 L. When processing ten samples, this results in approximately 500 L of washing solutions. The precipitation and neutralization of these solutions may take more than 14 days, significantly extending the overall duration of the phase analysis procedure.

In recent years, phase analysis has been applied not only to determine the forms of gold and silver occurrence in ores and to select beneficiation methods, but also as a key tool for monitoring precious metal distribution in flotation products from mineral processing plants.

3. RESULTS AND DISCUSSION

3.1. Phase Analysis Methodology Improvement and Laboratory Equipment Development

To improve productivity, analytical accuracy, and operational safety, a specialized laboratory installation for gold and silver phase analysis was designed and commissioned (Fig. 2). The methodology was enhanced by introducing a high-pressure filtration system for washing solid residues obtained at each analysis stage. This approach enables closed-system washing, minimizes operator exposure to hazardous solutions, and reduces the risk of sample loss or contamination. The system allows up to 20 technological products to be processed per day.

Fig. 2. Chemical phase analysis equipment

The methodological improvements began with the introduction of a high-pressure filtration system for washing products obtained at each stage of analysis. By selecting an appropriate filter surface, washing in a closed environment was achieved, eliminating prolonged operator contact with hazardous solutions. This system not only increases throughput up to 20 samples per day but also improves laboratory quality by minimizing the risk of sample contamination.

Subsequently, a new laboratory machine for gold and silver phase analysis was designed, capable of handling 20 positions simultaneously (Fig. 2). Special attention was given to agitation, as traditional methods are unsuitable for the delicate solutions used in phase analysis. The system employs a rotary disk (320 mm in diameter) with 4 rows of 5 slots, operating at a precisely controlled rotation speed of 28–30 rpm.

To accommodate the heterogeneous laboratory load - from a single sample weighing approximately 300-325 g to a full load of 20 samples totaling 6-6.5 kg, an inverter-controlled three-phase electric motor with a power rating of 0.75 kW was selected. The system ensures stable operation regardless of load variations and incorporates programmable timing control along with safety features for handling toxic reagents.

The improved methodology was applied to gold-bearing ores and flotation products, allowing accurate determination of phase distribution and metal balances. Results indicate that most gold and silver in the studied ore are present in cyanidable forms, followed by free and sulfide-associated phases. This advanced laboratory system provides reliable data for optimizing beneficiation strategies and minimizing precious metal losses.

The methodology was further adapted for flotation concentrates and middlings from polymetallic ores, which contain various metals in addition to precious metals. These adaptations required modifications to both the analytical procedures and the mathematical models used for calculations. For samples with oxide coatings, hydrochloric acid (HCl) and tin dichloride ($SnCl_2$) are applied to remove oxides, partially dissolving the solid phase and reducing its weight. Similarly, the determination of gold and silver associated with sulfides using aqua regia (HCl : HNO_3 = 3:1) also affects the sample weight. Consequently, additional measurements of each sample's weight before and after each analysis stage were introduced, together with a formula for calculating product balances.

Originally developed for gold-bearing ores, phase analysis was later adapted for flotation concentrates, tailings, and middling products (e.g., sands from hydrocyclones, classified products, and circulating load products). The improved methodology now enables determination of gold and silver forms throughout the production process, supporting process optimization and improved yield.

Phase analysis using the new laboratory machine was conducted on gold-bearing ore, with results presented in Tables 1 and 2, and Fig. 3.

Treatment	Au grade (g/t)	Ag grade (g/t)
Feed ore	0.180	0.320
Amalgamation	0.150	0.265
Cyanidation	0.057	0.120
Second cyanidation	0.048	0.107
Aqua regia	0.021	0.033

Table 1. Gold and silver phase analysis

Phase of presence	Au grade (g/t)	Au distribution (%)	Ag grade (g/t)	Ag distribution(%)
Free	0.030	16.18	0.055	17.19
Cyanidable	0.093	51.73	0.145	45.31
In oxide envelope	0.009	5.36	0.013	4.06
In sulfides	0.027	15.21	0.075	23.28
Finely dispersed	0.021	11.52	0.033	10.16

Table 2. Gold and silver phase distribution

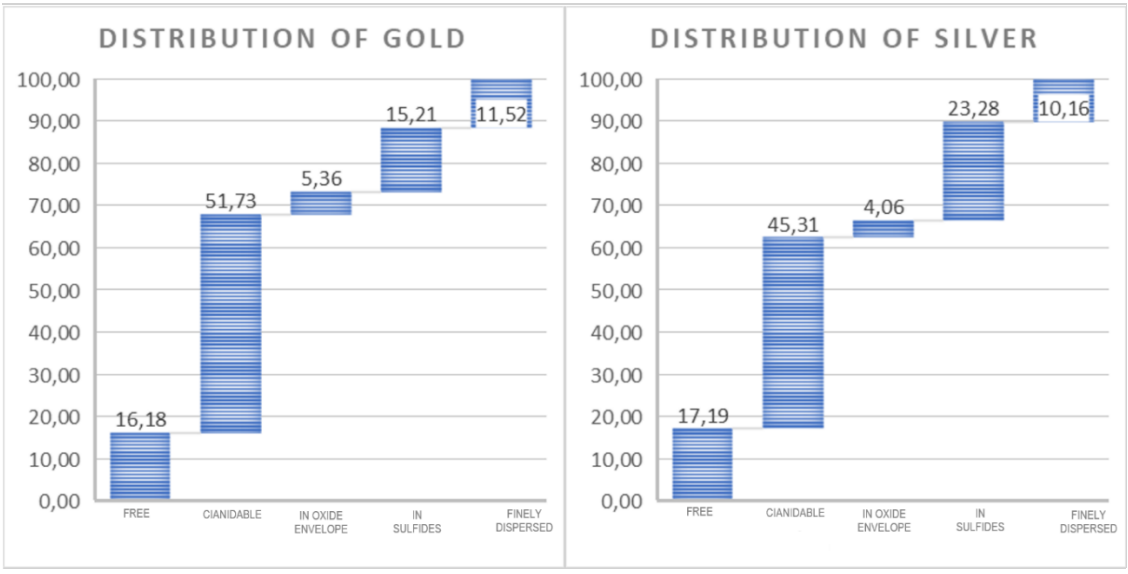

Fig. 3. Gold and silver phase distribution

Analysis shows that 51.73% of gold and 45.31% of silver are present in cyanidable forms, while free gold accounts for 16.18% and sulfide-bound gold for 15.21%. Oxide-coated gold represents 5.36%, and finely dispersed gold 11.52%. For silver, 23.28% is sulfide-bound, and 17.19% is free. These results demonstrate that hydrometallurgical methods, in combination with beneficiation, can effectively recover metals from the studied ore.

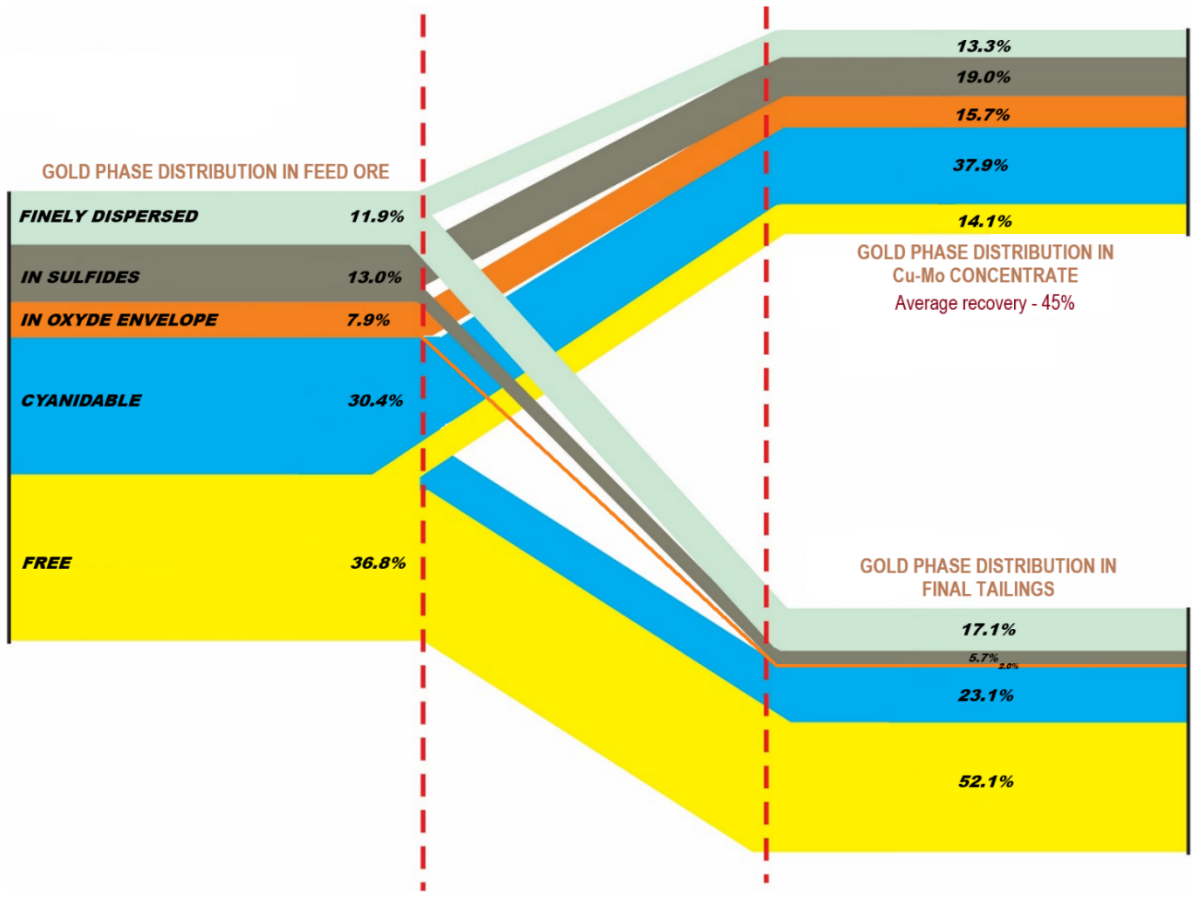

Fig. 4. Gold phase distribution in feed ore, concentrate and tailings

In a Cu-Mo ore beneficiation plant, gold recovery is relatively low (45-50%). Phase analysis using the new laboratory machine determined the forms and distribution of gold in the ore, concentrate, and final waste (Fig. 4). The study revealed that gold losses are primarily due to free gold particles in the waste. Enhancing gold recovery requires additional flotation process optimization or secondary gravity concentration.

Laboratory tests with a Knelson concentrator (model KC3MD) on 40 kg samples of final waste produced a concentrate with 16.6 g/t Au and 17.2% recovery. Twelve free gold particles larger than 0.125 mm were observed in the concentrate. These preliminary results justify further laboratory and pilot-scale studies to optimize gravity separation parameters.

The reported Knelson concentrator results represent preliminary laboratory-scale screening tests conducted on a composite sample of final flotation tailings. The purpose of these tests was to assess the presence, size characteristics, and recoverability of coarse free gold particles rather than to establish statistically optimized operating parameters. Gold recovery values are calculated relative to the gold grade of the flotation tailings feed used for the gravity separation tests. Due to the exploratory nature of the study, a limited number of tests were performed, and the results are reported as indicative values. A total of 12 free gold particles with sizes exceeding 0.125 mm were visually identified in the gravity concentrate, confirming the presence of recoverable free gold in the final tailings.

The positive outcomes of these preliminary tests provide technical justification for further laboratory and pilot-scale studies to evaluate process variability, repeatability, and optimize gravity separation parameters.

4. CONCLUSIONS

This study demonstrates that the development of specialized laboratory equipment can significantly improve the efficiency, reliability, and safety of gold and silver phase distribution analysis. The designed laboratory system integrates automated agitation, closed washing, and high-pressure filtration into a unified workflow, effectively overcoming key limitations of conventional phase analysis procedures related to labor intensity, sample losses, and operator-dependent variability.

The improved workflow enables the simultaneous processing of up to 20 samples under stable operating conditions, even in the presence of heterogeneous laboratory load regimes. The use of an inverter-controlled drive system ensures smooth operation under varying loads, contributes to reduced energy consumption, and enhances overall operational stability.

From an analytical perspective, the proposed system improves mass balance, accuracy, and reproducibility by minimizing reagent carry-over and solid material losses between successive analytical stages. In addition, closed-system washing and filtration significantly reduce operator exposure to hazardous reagents, particularly cyanide-containing solutions, thereby improving laboratory safety.

Applying the developed laboratory system to gold-bearing ores and flotation products confirms its suitability for reliable phase distribution studies. The obtained results provide essential information for selecting appropriate beneficiation strategies and identifying potential sources of precious metal losses. Overall, the proposed laboratory system represents an effective tool for supporting informed technological decision-making in mineral processing practice.

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