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SUBSTANTIATION OF THE OPTIMAL METHOD FOR DETERMINING THE REACTIVITY OF LIME MILK IN MINE WATER TREATMENT

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This article examines the accuracy of determining the reactivity of lime milk, which is used as the primary reagent for the removal of heavy metal ions from mine water. A comparison of four titrimetric methods was conducted, differing in sample preparation conditions (grinding, boiling), sample mass, and the use of correction coefficients. It was found that the average reactivity of lime milk ranged from 38.2% to 45.7%, with an overall mean value of 42.9% across all experiments. These results are significantly below the regulatory range of 52-70% established for this reagent. The most reproducible results were obtained using the method involving two sample masses and preliminary grinding of the sample. The low reactivity values are attributed to the presence of impurities (Mg, Si, Al, Fe, gypsum, sand), which reduce the effective $Ca(OH)_2$ content. The study concludes that stricter quality control of lime used for lime milk preparation is necessary, as well as an adjustment of the technological regulations for reagent dosing at treatment facilities.

Keywords: lime milk, reactivity, titrimetric analysis, quality control, impurities, calcium hydroxide, mine water treatment, heavy metals, reagent efficiency.

ОБОСНОВАНИЕ ОПТИМАЛЬНОГО МЕТОДА ОПРЕДЕЛЕНИЯ РЕАКЦИОННОЙ СПОСОБНОСТИ ИЗВЕСТКОВОГО МОЛОКА ПРИ ОЧИСТКЕ ШАХТНЫХ ВОД

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В статье рассмотрены вопросы точности определения реакционной способности известкового молока, используемого в качестве основного реагента при очистке шахтных вод от ионов тяжёлых металлов. Проведено сравнение четырёх титриметрических методик, различающихся условиями пробоподготовки (растирание, кипячение), массой навески и применением корректирующих коэффициентов. Показано, что средние значения реакционной способности известкового молока составляют 38,2-45,7 %, а среднее значение по совокупности экспериментов - 42,9 %. Установлено, что полученные результаты существенно ниже нормативного диапазона (52–70 %), регламентированного для данного реагента. Выявлено, что наиболее воспроизводимые результаты даёт методика с использованием двух навесок и предварительным растиранием пробы. Низкие значения активности объясняются наличием примесей (Mg, Si, Al, Fe, гипс, песок), снижающих содержание эффективной $Ca(OH)_2$. Сделан вывод о необходимости ужесточения контроля качества извести, используемой для приготовления известкового молока, а также корректировки технологического регламента дозирования реагента на очистных сооружениях.

Ключевые слова: известковое молоко, реакционная способность, титриметрический анализ, контроль качества, примеси, кальций гидроксид, очистка шахтных вод, тяжелые металлы, эффективность реагентов.

ШАХТА СУЛАРЫН ТАЗАРТУ КЕЗІНДЕ ӘК СҮТІНІҢ РЕАКЦИЯЛЫҚ ҚАБІЛЕТІН АНЫҚТАУДЫҢ ОҢТАЙЛЫ ӘДІСІН НЕГІЗДЕУ

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Бұл мақалада шахталық суларды ауыр металл иондарынан тазарту үшін негізгі реагент ретінде қолданылатын әк сүтінің реакциялық қабілетін анықтау дәлдігі мәселелері қарастырылады. Үлгіні алдын ала өңдеу (ұнтақтау, қайнату), салмақ массасы және түзету коэффициенттерін қолдану шарттары бойынша ерекшеленетін төрт титриметриялық әдіс салыстырылды. Зерттеу нәтижелері бойынша әк сүтінің орташа реакциялық қабілетінің 38,2-45,7 % аралығында, ал барлық тәжірибелер бойынша орташа мәні - 42,9 % екені анықталды. Бұл көрсеткіштер осы реагентке арналған нормативтік диапазоннан (52-70 %) едәуір төмен. Ең қайталанатын және сенімді нәтижелер екі түрлі салмақ және үлгіні алдын ала ұнтақтау арқылы алынатын әдіс бойынша алынды. Белсенділіктің төмен мәндері қоспалардың (Mg, Si, Al, Fe, гипс, құм) болуымен, яғни тиімді $Ca(OH)_2$ мөлшерінің төмендеуімен түсіндіріледі. Әк сүтін дайындауға пайдаланылатын әктің сапасын бақылауды күшейту, сондай-ақ тазарту құрылыстарындағы реагент мөлшерлеудің технологиялық регламентін түзету қажеттігі туралы қорытынды жасалды.

Түйін сөздер: әк сүті, реакциялық қабілеті, титриметриялық талдау, сапа бақылауы, қоспалар, кальций гидроксиді, шахталық суларды тазарту, ауыр металдар, реагент тиімділігі.

Introduction. The contamination of mine water with heavy metals and acidic compounds is one of the most serious environmental challenges faced by mining regions. According to [1, 2], the discharge of insufficiently treated mine water into surface water bodies leads to elevated concentrations of copper, zinc, manganese, and iron ions, as well as an increase in sulfate load. This contributes to the disruption of ecosystem processes, the accumulation of toxic compounds in bottom sediments, and the degradation of drinking water quality in nearby areas [3].

Estimates from the International Energy Agency indicate that the global annual volume of mine water requiring treatment exceeds 10 billion cubic meters, with a significant portion attributed to coal and non-ferrous metal industries [4]. In countries with a highly developed mining sector - such as China, Australia, the United States, and South Africa- mine water treatment is a mandatory requirement for industrial mine operation [5].

In the literature, various alternative reagents for mine water treatment are widely discussed, including strong oxidizing agents and polymer-based flocculants. For example, the oxidizing reagent "Fernel," based on potassium ferrate (K_2FeO_4, 25.2-40.3 wt.%) and alkali (KOH, 47.2-68.1 wt.%), demonstrates high oxidative capacity and is used for the removal of heavy metals from industrial wastewater [6]. Studies also indicate that ferrate(VI) (FeO_4^{2-}) can effectively oxidize contaminants, resulting in the formation of stable Fe(III) precipitates [7, 8]. However, despite its strong oxidizing reactivity, the use of ferrate is often limited by high costs and challenges associated with large-scale industrial application.

At the same time, the use of alkaline neutralizing reagents remains a fundamental method in

industrial practice. For instance, several authors [9] have demonstrated the efficiency of alkaline neutralization using calcium hydroxide under high concentrations of Fe and Mn in mine waters.

In addition, industrial practice frequently involves the combined use of reagents. In a pilot-scale mine water treatment facility at a non-ferrous metal mining site, lime, a coagulant, and a polymer flocculant were used simultaneously, which enabled the optimized precipitation of heavy metals into the sludge phase [10].

Thus, although reagents such as ferrate or polymeric flocculants demonstrate good treatment performance, among the available purification technologies, alkaline-based neutralization processes remain the most effective and economically feasible [11, 12], which is particularly important for the long-term operation of treatment facilities [13].

Lime milk - a water-based suspension of calcium hydroxide $Ca(OH)_2$ - is commonly used to adjust pH and precipitate heavy metal hydroxides [14,15]. Global practice shows that lime milk continues to be a cost-effective reagent for both large-scale treatment plants and localized systems at industrial facilities [16-18].

The effectiveness of lime milk depends primarily on its reactivity, i.e., the content of chemically available calcium hydroxide [19, 20]. Technological regulations typically specify that lime milk reactivity should fall within the range of 52-70%, although actual values are often lower [21]. This discrepancy is generally attributed to the quality of the initial lime, the presence of impurities (Mg, Si, Al, Fe, gypsum, sand), and the conditions under which the suspension is prepared.

In industrial settings, quality control of lime milk at treatment facilities and production sites is typically performed using standard titrimetric methods [22]. These methods are relatively simple, cost-effective, and suitable for routine use in on-site laboratories. However, the reproducibility of results and the selection of titration parameters-such as sample mass, sample preparation, and temperature - are often critical factors that influence the accuracy of reactivity assessment. In many cases, deviations from the normative reactivity range remain undetected, leading to reagent dosing errors and a subsequent reduction in mine water treatment efficiency.

Inaccurate assessment of lime milk reactivity can cause two types of problems. In the case of under-dosing, water remains contaminated with heavy metals and acids. On the other hand, excessive dosing results in so-called secondary pollution: elevated calcium ion concentrations, increased pH levels, and excessive formation of precipitates (calcium carbonates and sulfates) in the treated water. This complicates the operation of treatment facilities and may have negative impacts on receiving water bodies [23].

Therefore, the accurate determination of lime milk reactivity and the selection of an appropriate analytical method are essential for environmentally safe and economically efficient mine water treatment.

The objective of this study is to compare various titrimetric methods for determining lime milk reactivity, to identify factors influencing the accuracy and reproducibility of results, and to assess the practical implications of reagent reactivity variations for mine water treatment processes.

Materials and methods. The object of this study was actual samples of lime milk used at the mine water treatment facilities of the Belousovsky mine for the precipitation of heavy non-ferrous metal salts. Lime milk is prepared in a mechanical classifier by slaking quicklime with process water to obtain a 5% $Ca(OH)_2$ suspension. Depending on the quality of the initial lime -characterized by the content of active component (52-70%) the daily reagent consumption ranges from 0.34 to 0.46 tons, with a suspension density of approximately 1.03 g/cm^3.

Since the lime milk suspension is in a constant state of solid phase sedimentation, the samples were thoroughly mixed prior to sampling. Analyses were performed using various methods, with the sample mass being no less than 2 g. This is because smaller

sample masses tend to overestimate the amount of free lime and, consequently, the calculated reactivity of the lime milk. The determination of free lime content was carried out by titration. Titration was performed slowly, as rapid addition of the titrant causes the rate of $Ca(OH)_2$ neutralization to exceed its dissolution rate. This leads to partial reaction of the acid with unreacted or overburnt lime particles. As a result, the measured free lime content may be overestimated, and the calculated reactivity of the lime milk may increase by 6-8%.

In this study, 1 N solutions of hydrochloric acid and sodium hydroxide were used, prepared from fixanal standards immediately before analysis. To ensure the reliability of the results and to eliminate random and gross errors, each sample of lime milk of the same quality used in the treatment of mine water at the Belousovsky mine was analyzed using several basic methods [24,25]. This approach is justified by the fact that existing methods for determining free and total lime content are largely subjective, and even when analyzing the same sample, discrepancies in calculated reactivity may reach 3-5% or more.

Method Using Two Sample Masses (with Grinding and Boiling)

To determine the free lime content (A_free), approximately 5 g of well-mixed lime milk was weighed on an analytical balance into a porcelain mortar. A few drops of phenolphthalein were added, and the sample was titrated with a 1 N solution of hydrochloric acid while thoroughly grinding the lime particles with a pestle until complete and stable decolorization was achieved. The titration was carried out slowly. The free lime content (%) as *CaO* relative to the mass of the lime milk was calculated using the following formula:

$$A_{free} = \frac{V_{HCl} \cdot 0,028 \cdot 100}{a} \qquad (1)$$

where: V_{HCl} - volume of 1N hydrochloric acid solution used for titration, in cm³; 0.028 - the titer of 1 N *HCl* solution with respect to *CaO* (g/cm³); a - sample mass of lime milk used for the determination of free lime, in grams.

To determine the total lime content (A_{total}), approximately 5 g of lime milk was placed into a 150 cm³ conical flask. Then, 50 cm³ of 1 N hydrochloric acid solution was added, the flask was covered with a watch glass, and the mixture was boiled for 1-2 minutes. After cooling, the solution was titrated with 1 N sodium hydroxide solution using methyl orange as an indicator, until the color changed from red to yellow. The total lime content (%) as *CaO* relative to the mass of the lime milk was calculated using the following formula:

$$A_{total} = \frac{(50 - V_{NaOH}) \cdot 0,028 \cdot 100}{b} \qquad (2)$$

where: V_{NaOH} - volume of 1 N sodium hydroxide solution used for titration, in cm³; b - sample mass of lime milk used for the determination of total lime, in grams.

The reactivity ($A_{reactivity}$) of the lime milk, expressed as a percentage, was calculated using the formula:

$$A_{reactivity} = \frac{A_{free}}{A_{total}} \cdot 100 \qquad (3)$$

Method Using a Single Sample Mass (with Grinding and Boiling)

To determine the free lime content, approximately 5 g of well-mixed lime milk was weighed on an analytical balance into a porcelain mortar. A few drops of phenolphthalein were added, and the sample was titrated with a 1 N hydrochloric acid solution while thoroughly grinding the lime particles with a pestle until complete and stable decolorization was achieved. The titration was performed slowly. The free lime content (%) as *CaO* relative to the mass of the lime milk was calculated using formula (1). To determine the total lime content, the contents of the mortar were transferred to a 250 cm³ flask. The pestle and mortar were rinsed with distilled water, which was also poured into the flask. Then, 50 cm³ of 1 N hydrochloric acid solution was added, the flask was covered with a watch glass, and the mixture was boiled for 1-2 minutes. After cooling, titration was

carried out with a 1 N sodium hydroxide solution using methyl orange as an indicator until the color changed from red to yellow. The total lime content (%) as *CaO* relative to the mass of the lime milk was calculated by the formula:

$$A_{total} = \frac{[(V_{HCl} + 50) - V_{NaOH}] \cdot 0,028 \cdot 100}{a} \quad (4)$$

where: V_{HCl} - volume of 1 N hydrochloric acid solution used for titration during the free lime determination, in cm³; 50 -volume of 1 N hydrochloric acid solution added for the total lime determination, in cm³; V_{NaOH} - volume of 1 N sodium hydroxide solution used for titration during the total lime determination, in cm³; a - sample mass of lime milk, in grams.

The reactivity ($A_{reactivity}$) of the lime milk, expressed as a percentage, was calculated using the formula:

$$A_{reactivity} = \frac{V_{HCl}}{((V_{HCl} + 50) - V_{NaOH})} \cdot 100 \quad (5)$$

Method Using a Single Sample Mass with Correction Coefficient Determination (with Grinding, without Boiling)

Table 1 - Experimental Results for Determining the Correction Coefficient of 1 N Hydrochloric Acid Solution

Experiment No.	Mass of Anhydrous Sodium Carbonate, g	$V(HCl), cm^3$	K_{HCl}	$K_{cp}(HCl)$
1	1,6000	30,0	1,0063	1,0174
2	1,6500	30,5	1,0207	
3	1,7100	31,5	1,0243	
4	1,6200	30,0	1,0189	
5	1,6324	30,1	1,0233	
6	1,6168	30,4	1,0035	
7	1,7650	32,6	1,0215	
8	1,9529	36,1	1,0207	

Before starting the analysis, correction coefficients for the hydrochloric acid solution (K_{HCl}) and sodium hydroxide solution (K_{NaOH}). were determined. To determine the correction coefficient for the 1 N HCl solution based on anhydrous sodium carbonate, anhydrous sodium carbonate was dried in a drying oven for 1 hour at 250-270°C using a reagent-grade sample. The 1N hydrochloric acid solution was prepared by diluting the corresponding concentrated acid. To prepare 1 dm³ of 1 N HCl solution, 90 cm³ of acid with a density of ρ = 1.174 g/cm³ was carefully poured into water with stirring, and the volume was then brought up to 1 dm³ with water. A weighed portion of anhydrous sodium carbonate was placed into a conical flask, 40 cm³ of water was added, followed by 3-4 drops of methyl orange indicator. The solution was titrated from a burette with 1 N hydrochloric acid solution. Then, the solution was heated and boiled for 2-3 minutes to remove carbon dioxide, and after cooling, the titration was continued until the reappearance of the color change. Three parallel experiments were conducted, and the average volume of hydrochloric acid consumed during titration was determined (Table 1). The correction coefficient (*K*) was calculated using the formula:

$$K_{HCl} = \frac{m_1 \cdot 1000}{M_1 \cdot C_1 \cdot V_{cp}(HCl)} \quad (6)$$

where: m_1 - mass of the sodium carbonate sample; in grams; M_1 - molar mass of the equivalent of the standard substance, 53.00 g/mol; C_1 - nominal molar concentration of the substance in the solution, mol/dm³; $V_{cp}(HCl)$ - average volume of the analyzed hydrochloric acid solution consumed for titration, in cm³.

To determine the correction coefficient of the 1 N sodium hydroxide solution using oxalic acid, 2 g of oxalic acid was weighed and placed into a conical flask, dissolved in 50.0 cm³ of carbon dioxide-free water, and heated to 60-70 °C. The contents of the flask were titrated while hot with sodium hydroxide solution in the presence of 2 drops of phenolphthalein until a persistent pink color appeared that did not fade for 50–60 seconds. The correction coefficient (K) of the sodium hydroxide solution was calculated using the formula (Table 2):

$$K = \frac{m}{V \cdot a} \quad (7)$$

where: V - volume of sodium hydroxide solution consumed in titration, cm³; m - mass of the standard substance, g; a - mass of the standard substance equivalent to 1 ml of sodium hydroxide solution with concentration C(NaOH) = 1 mol/dm³, a = 0.06304 g for oxalic acid.

Table 2 - Experimental Results for Determining the Correction Coefficient of 1 N Sodium Hydroxide Solution

Experiment No.	Mass of Oxalic Acid, g	$V(NaOH), cm^3$	K_{NaOH}	$K_{cp}(NaOH)$
1	2,0005	31,8	0,9979	1,0011
2	2,0003	31,7	1,0010	
3	2,0004	31,7	1,0010	
4	2,0004	31,7	1,0010	
5	2,0005	31,7	1,0011	
6	2,0008	31,7	1,0012	
7	2,0004	31,6	1,0042	
8	2,0009	31,7	1,0013	

For the determination of free lime, approximately 5 g of well-mixed lime milk was weighed on technical scales in a porcelain mortar. A few drops of phenolphthalein were added, and the sample was titrated with 1 N hydrochloric acid solution while carefully grinding the lime particles with a pestle until complete and stable decolorization was achieved. Titration was carried out slowly. The content of free lime (%) *CaO* relative to the mass of lime milk was calculated using the formula:

$$A_{free} = \frac{V_{HCl} \cdot K_{HCl} \cdot 0,028 \cdot 100}{a} \quad (8)$$

where: V_{HCl} is the volume of 1 N hydrochloric acid solution used for titration, cm³; K_{HCl} is the correction coefficient for the 1 N hydrochloric acid solution; 0.028 is the titer of 1 N hydrochloric acid solution in terms of *CaO*; and *a* is the lime milk sample mass taken for free lime determination, g.

For the determination of total lime, the contents of the mortar were transferred to a 250 cm³ flask. The pestle and mortar were rinsed with distilled water, which was also poured into the flask. Then 50 cm³ of 1 N hydrochloric acid solution was added, and the mixture was titrated with 1 N sodium hydroxide solution using methyl orange as an indicator until the red color changed to yellow. The total lime content (%) *CaO* relative to the mass of lime milk was calculated by the formula:

$$A_{total} = \frac{\left[(V_{HCl} + 50) \cdot K_{HCl} - V_{NaOH} \cdot K_{NaOH}\right] \cdot 0,028 \cdot 100}{a} \quad (9)$$

where: V_{HCl}- is the volume of 1 N hydrochloric acid solution used in the free lime titration, cm³; K_{HCl} - is the correction coefficient for hydrochloric acid; 50 cm³ is the volume of 1 N hydrochloric acid added for total lime determination; V_{NaOH} - is the volume of 1 N sodium hydroxide solution used in the total lime titration, cm³; K_{NaOH} - is the correction coefficient for sodium hydroxide; a – and a is the lime milk sample mass, g.

The reactivity ($A_{reactivity}$) of the lime milk (%) was calculated using the formula:

$$Aactivity = \frac{V_{HCl} \cdot K_{HCl}}{\left[(V_{HCl} + 50) \cdot K_{HCl} - V_{NaOH} \cdot K_{NaOH}\right]} \cdot 100 \quad (10)$$

Method with a single sample and determination of correction coefficients (without boiling and without grinding)

Before starting the analysis, correction coefficients for hydrochloric acid and sodium hydroxide solutions were determined. For free lime determination, approximately 5 g of well-mixed lime milk was weighed on technical scales in a porcelain mortar. A few drops of phenolphthalein were added, and the sample was titrated slowly with 1 N hydrochloric acid until complete and stable decolorization was achieved. Free lime content (%) *CaO* relative to the lime milk mass was calculated using formula (8).

For total lime determination, the mortar contents were transferred to a 250 cm³ flask. The pestle and mortar were rinsed with distilled water, which was poured into the flask. Then 50 ml of 1 N hydrochloric acid solution was added, and titration was performed with 1 N sodium hydroxide solution using methyl orange indicator until the color changed from red to yellow. Total lime content (%) *CaO* relative to the lime milk mass was calculated using formula (9). Lime milk reactivity (%) was calculated using formula (10).

Results and discussion. During the study, several methods for determining the reactivity of lime milk were tested. These methods differed in terms of accuracy, labor intensity, and suitability for use under industrial conditions. In real-world practice at treatment facilities, the choice of analytical method is guided by the need to balance data reliability with operational efficiency.

Table 3 - Results of Lime Milk Reactivity Determination

Experiment No.	Indicator – Phenolphthalein			Indicator – Methyl Orange			Lime milk reactivity Act, %
	Lime Milk Mass a, g	V_{HCl}, used for titration, cm³	Free Lime Content A_{free}, %	Lime Milk Mass b, g	V_{NaOH}, used for titration,	Total lime content A_{total}, %	
1	4,98	1,2	0,6747	5,11	47,0	1,6438	41,04
2	4,96	1,3	0,7339	5,08	47,2	1,5433	47,55
3	5,05	1,3	0,7208	5,10	47,3	1,4824	48,62
AVERAGE							45,74± 4,10%

To assess the reactivity of lime milk, a series of experiments was conducted using the double-sample method and various indicators (phenolphthalein and methyl orange). This approach allowed for simultaneous determination of both free and total lime content, thus making the results more reliable. In each experiment, the amounts of free and total lime were determined, on the basis of which the active portion of the reagent was calculated. The experimental results are presented in Table 3.

As shown in Table 1, the reactivity of lime milk in the analyzed samples ranged from 41.04% to 48.62%, with an average value of 45.74%. Statistical analysis revealed that the range of values was 7.58%, the standard deviation was 4.10%, and the coefficient of variation amounted to 8.97%. This variability indicates the influence of suspension heterogeneity and the specifics of the titration process.

Overall, the method demonstrated sufficiently high accuracy and reproducibility. However, the analysis requires more time and reagents, making it less convenient for repeated determinations under real-world conditions at operating treatment facilities.

To optimize the quality control procedure, a single-sample method was tested, which allows the analysis time to be reduced by nearly half. The experimental results obtained using this method are presented in Table 4.

Table 4 - Experimental Results of Lime Milk Reactivity Determination

Experiment No.	Indicator – Phenolphthalein			Indicator – Methyl Orange		Lime milk reactivity Act, %
	Lime Milk Mass a, g	V_{HCl}, used for titration, cm³	Free Lime Content A_{free}, %	V_{NaOH}, used for titration, cm³	Total lime content, A_{total} %	
1	5,10	1,2	0,6588	49,0	1,2078	54,55
2	5,08	1,2	0,6614	48,8	1,3228	50,00
3	5,14	1,3	0,7082	47,2	2,2335	31,71
AVERAGE						45,42±11,85%

Table 5 - Experimental Results for Determining the Reactivity of Lime Milk

Experiment No.	K_{cp} (HCl)	K_{cp} $(NaOH)$	Indicator – Phenolphthalein			Indicator – Methyl Orange		Lime milk reactivity Act, %
			Lime Milk Mass a, g	V_{HCl}, used for titration, cm³	Free Lime Content A_{free}, %	V_{NaOH}, used for titration, cm³	Total lime content, A_{total} %	
1	1,0174	1,0011	5,140	1,2	0,6651	49,2	1,5722	42,31
2			5,026	2,1	1,1898	48,6	2,4064	49,44
3			5,072	1,1	0,6177	48,8	1,7122	36,08
4			5,100	1,1	0,6143	49,4	1,3728	44,75
5			5,042	1,1	0,6214	49,0	1,6111	38,57
AVERAGE								42,23±5,02%

The reactivity of the lime milk in the analyzed samples varied from 31.71% to 54.55%. The average reactivity value was 45.42%. Statistical analysis revealed a range of values of 22.84%, a standard deviation (σ) of 11.85%, and a coefficient of variation (V) of 26.1%. Thus, the single-sample method demonstrated a significant scatter of results, exceeding acceptable limits for analytical measurements.

To improve the accuracy of lime milk reactivity determination, a single-sample method with the application of correction coefficients was used, with samples analyzed without boiling. The use of correction coefficients helps to account for possible deviations in titrant volumes and thereby increases the reliability of the obtained results. The experimental data are presented in Table 5.

The reactivity of lime milk determined by this method ranged from 36.08% to 49.44%. The average value was 42.23%, which is significantly lower than the expected regulatory standards. Statistical analysis revealed considerable data variability: the standard deviation was 5.02%, and the coefficient of variation reached 11.9%, indicating low reproducibility of the method.

For further analysis, a method with a single sample weight was applied, using correction coefficients, but the samples were analyzed without boiling and without grinding the lime milk. The absence of grinding may additionally affect the completeness of calcium hydroxide dissolution, which impacts the accuracy of the results (Table 6).

Table 6 - Experimental Results for Determining the Reactivity of Lime Milk

Experiment No.	K_{cp} (HCl)	K_{cp} $(NaOH)$	Indicator – Phenolphthalein			Indicator – Methyl Orange		Lime milk reactivity Act, %
			Lime Milk Mass a, g	V_{HCl}, used for titration, cm³	Free Lime Content A_{free}, %	V_{NaOH}, used for titration, cm³	Total lime content, A_{total} %	
1	1,0174	1,0011	5,120	1,3	0,7234	49,5	1,4699	49,21
2			5,030	2,2	1,2457	46,7	3,5207	35,38
3			5,032	2,0	1,1320	47,9	2,7371	41,36
4			5,068	2,0	1,1240	47,1	3,1605	35,56
5			5,052	0,9	0,5074	49,2	1,3841	36,66
6			5,028	1,0	0,5665	49,0	1,5589	36,34
7			4,97	1,1	0,6304	48,5	1,9167	32,89
AVERAGE								38,20±5,02%

The reactivity of lime milk by this method ranged from 32.89% to 49.21%. The average value was 38.20%, which is significantly below the expected regulatory standards. The range of values was 16.32%, the standard deviation was 5.62%, and the coefficient of variation was 14.7%.

To assess the accuracy and reproducibility, the results obtained by all four titration methods were compared. Table 7 presents the average values of lime milk reactivity, as well as the main statistical indicators.

The two-sample method allows for the separate determination of free and total lime content, providing a more comprehensive understanding of the composition of lime milk. In the conducted experiments (Table 7), the reagent reactivity ranged from 41.04% to 48.62%, with an average value of 45.74 ± 4.10%. Using two samples yields more reliable results compared to methods utilizing a single sample, as it accounts for both the dissolved

and the precipitated parts of calcium hydroxide. However, the method remains quite sensitive to the conditions of the analysis: the results are influenced by the sample mass and the titration rate. Thus, the two-sample method provides the highest accuracy and is used as the primary control technique for adjusting reagent consumption in accordance with the technological regulations.

Table 7 - Comparative Results of Different Titration Methods

Method	Range of A_act, %	Average value, %	σ (Standard deviation), %	V (Coefficient of variation), %
Two samples	41,04 – 48,62	45,74	4,10	8,97
One sample	31,71 – 54,55	45,42	11,85	26,1
One sample with correction factor (without boiling)	36,08 – 49,44	42,23	5,02	11,9
One sample with correction factor (without boiling and grinding)	32,89 – 49,21	38,20	5,62	14,7

The single-sample method is a simplified version of the analysis used for operational production control. It allows for halving the analysis time, which is especially important when multiple determinations are required during a shift. However, the obtained results showed extremely high variability - the coefficient of variation was 26.1%, indicating low reproducibility of the method. The large scatter is explained by several factors: heterogeneity of the lime milk suspension, errors in weighing the sample, as well as differences in the dissolution and neutralization rates of calcium hydroxide during titration. Thus, the single-sample method is convenient and sufficiently reliable for rapid assessment of lime milk reactivity, but it does not provide the necessary accuracy and reliability for regular analytical control.

The application of the single-sample method with the use of a correction factor represents a modification of the accelerated method, which takes into account adjustments for systematic titration errors. This simplifies the analysis procedure while simultaneously improving the accuracy of titrant volume measurement. However, the obtained results showed a significant variability in reactivity values (ranging from 36.08% to 49.44%). The average value was 42.23%, which is noticeably below the standard level. A coefficient of variation of 11.9% indicates relatively low reproducibility of the method. Despite the use of correction factors, the influence of factors such as suspension heterogeneity, titration speed, and differences in lime component solubility remains. Thus, the single-sample method without boiling, even with the use of a correction factor, provides only an approximate estimation of lime milk reactivity and cannot be considered a sufficiently reliable control method for the operation of treatment facilities.

The application of the single-sample method without boiling and without grinding revealed an even wider range of lime milk reactivity values compared to previous approaches. The average value was low (38.20%), and the variability between individual measurements was significant. Statistical indicators confirm the low reproducibility of the method: the coefficient of variation was 14.7%, which substantially exceeds the acceptable limits for analytical determinations. A key factor affecting the results is the absence of grinding the sample, which limits the completeness of calcium hydroxide dissolution and leads to an underestimation of reactivity. Although some high values were recorded (up to 49.21%), they did not compensate for the overall downward trend in

average values. Thus, despite its simplicity, this method proved to be the least reliable among the investigated techniques and can only be used as a rough express analysis rather than for regular production control.

A comparative analysis of the obtained data showed that the highest reproducibility was achieved by the two-sample method: the coefficient of variation was less than 10%, which falls within acceptable limits for analytical measurements. This method also demonstrated the smallest range of values (41.04-48.62%). The single-sample method proved to be the least reliable, with a coefficient of variation reaching 26.1% and a value range exceeding 22%. This indicates a high sensitivity of the method to the analysis conditions, making it unsuitable for precise control. The use of correction factors partially improved reproducibility (standard deviation decreased to 5.02–5.62%), but the omission of boiling and grinding the samples negatively affected the completeness of active component dissolution, leading to an underestimation of average reactivity values (38.20-42.23%).

Conclusions. The analysis of the obtained data (Table 7) showed that the reactivity of 5% lime milk used at the mine water treatment facilities of the Belousovsky mine for the precipitation of heavy metal salts averages 42.9%, which is significantly lower than the values specified in the technological regulations (52-70%). This discrepancy requires continuous monitoring of the actual lime consumption and adjustment of the daily reagent dosage.

The reasons for the reduced reactivity are related both to the composition of the raw material and to the technological conditions of its processing. Unsaturated lime produced by the calcination of calcium-magnesium rocks is used to prepare the lime milk. Besides pure calcium and magnesium oxides, the calcined product contains compounds with silicon, aluminum, and iron, as well as underburnt residues. These impurities reduce the rereactivity of the lime, and its instability in air additionally leads to partial carbonation.

The effectiveness of lime milk depends on the solubility and dissolution rate of calcium hydroxide, which are influenced by temperature, crystal structure, impurity content, and slaking conditions. The actual reactivity was insufficient to ensure the normative pH level of 10.5 at the standard dosage, which may reduce the efficiency of the treatment process.

The conducted research demonstrated that the titration conditions significantly affect the reactivity analysis results. Key factors include the titration rate, the necessity of grinding the sample, the use of boiling for determining total lime content, the quality of prepared titrant solutions, and the presence of foreign components (gypsum, sand, aluminum and iron oxides, magnesium hydroxide).

Comparison of titration methods showed that simplified approaches (single sample, with correction factors) produce substantially higher variability and lower reproducibility. The two-sample method provides the highest accuracy and stability of results, ensuring reliable quality control of lime milk under production conditions.

Thus, the following conclusions can be drawn from the results:

1. The actual reactivity of lime milk is below normative values and requires adjustment of reagent consumption.

2. The decrease in reactivity is related to the quality of the initial lime, its structure, and impurity content.

3. Analysis results depend on the titration methodology.

4. For production control, the two-sample method is recommended as the most accurate and reproducible.

At the same time, in the practice of the Belousovsky mine’s treatment facilities, it is advisable to establish a lime milk reactivity control system based on the following principle: regular laboratory control using the two-sample method; operational control during shifts using the single-sample method or with correction factors; express control at the preparation site using the single-sample method without boiling and grinding. This approach provides a balance between analytical

accuracy and ease of application in production conditions.

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