

IMPROVING THE METHOD OF DETERMINING THE MASS FRACTION OF MAGNESIUM CARBONATE AND THE STUDY OF THE CHEMICAL COMPOSITION OF CARBONATE ROCKS FOR THE EFFECTIVE CONDUCT OF THE TECHNOLOGICAL PROCESS OF SUGAR PRODUCTION

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ABSTRACT

The article considers an improved method for determining the content of magnesium carbonate in the carbonate rock. An adjusted method for determining the content of magnesium carbonate was included in the complete establishment of chemical analysis of limestone, which includes the determination of moisture, impurities insoluble in hydrochloric acid, the amount of one and a half oxides of aluminum and iron, calcium carbonate and magnesium carbonate (advanced method), calcium sulfate, alkali metal oxides of potassium and sodium. The obtained experimental data were entered into a single table and summed up the material balance of all components of the carbonate rock. As a result, it was found that this technique includes the following criteria: it is the most accurate, most accessible, and cheap. The use of adjusted methods for determining the content of calcium and magnesium carbonate in limestone will make it possible to establish the objective chemical composition of the carbonate rock and avoid several technological problems. Namely, the excess of uncontrolled magnesium carbonate contributes to the formation of the liquid phase, which in turn reduces the concentration of chemically active lime and promotes the formation of melts in the lime kiln, deteriorating filtration rates, clogging the evaporating station, and so on. Therefore, having information about the real component composition of limestone, the technologist will be able to adjust the technological process in advance, which will lead to the preservation of natural resources while the quality of finished products will not decrease.

Keywords: carbonate rock; material balance; sugar industry; dolomite; lime department

INTRODUCTION

In Ukraine, limestone is used in the technological process of sugar production to purify diffusion juice from non-sugars. Initially, limestone is fired at high temperatures to obtain lime and carbon dioxide. Then the lime is quenched with water to obtain lime milk. Lime milk ($\text{Ca}(\text{OH})_2$) and carbon dioxide (CO_2) are used to effectively remove non-sugars from diffusion juice.

The quality of these components ($\text{Ca}(\text{OH})_2$ and CO_2) depends on the degree of the condition of the limestone. According to Ukrainian requirements, the content of calcium carbonate (CaCO_3) in the carbonate rock must be at least 93.00%. The mass fraction of impurities in limestone should not exceed:

- substances that are insoluble in hydrochloric acid – not more than 3.00%;
- oxides of aluminum and iron – in the amount not exceeding 1.50%;
- magnesium carbonate – not more than 2.50%;
- calcium sulfate – not more than 0.40%;

- alkali metal oxides of potassium and sodium – in the amount not exceeding 0.25%;

- impurities (clay, etc.) – not more than 3.00%.

Only by using conditioned components for the purification of diffusion juice, it is possible to receive carbonized juices of high purity. After the non-sugars are removed from the diffusion juice, the sucrose content accumulates in the system.

The yield and quality of the final product – sugar – will depend on the concentration of sucrose and the purity of the juice. Sugar is used as a finished product for human consumption and as an intermediate in the production of other foods (Figure 1).

For the needs of the sugar factory, only calcium carbonate is considered a useful component of the carbonate rock, all other components are harmful impurities (Zheplinska et al., 2020). In nature, very rare limestones whose chemical composition would be close to calcite. Magnesium is often a component of limestone, if the content of magnesium oxide (MgO) in it is 4 – 17%, then such limestone is called dolomitized (Zheplinska et al., 2019).

Magnesium carbonate ($MgCO_3$) contained in the carbonate rock can lead to several subsequent adverse events during its (carbonate rock) firing. Magnesium carbonate, which is part of dolomite ($CaMg(CO_3)_2$) promotes the formation of a liquid phase during its thermal dissociation. The liquid phase, in turn, envelops the grains of active calcium oxide with a non-reactive film, which leads to the fact that part of the potentially chemically active lime becomes inactive. The formed melt enters the pores of active lime, as a result of which the dissociation of a significant part of the carbonate rock will not be able to pass in full. In addition, the liquid phase in significant quantities can enter the lower zone of the furnace where it meets the colder gas solidifies with the formation of sintered depths. This phenomenon can lead to a "hang" of the furnace, and in less optimistic forecasts, and to the shutdown of the plant.

During the firing of limestone, the proportion of $MgCO_3$ contained in it decomposes into MgO and carbon dioxide (CO_2). Once on the plant machine during the quenching of magnesium oxide, we obtain magnesium hydroxide ($Mg(OH)_2$), and later during the saturation of magnesium carbonate ($MgCO_3$). The latter ($MgCO_3$) significantly impairs the filtration rates of saturated juice precipitates. In the face of such a situation, when there is a significant content of magnesium in lime milk, the technologist tries to maintain a high pH value (not less than 11) on the first saturation, and before the second saturation not to give lime at all (Sukhenko et al., 2019). Such measures are carried out because $Mg(OH)_2$ begins to precipitate at pH 11 and above. It is in this state that this component ($Mg(OH)_2$) has a crystalline structure and is most easily removed from the system in contrast to magnesium carbonate, which has an amorphous structure.

Also, in the case of saturation of juices to a slightly alkaline or neutral reaction, Ca and Mg go into solution in the form of acidic carbon salts and at vigorous boiling of the juice precipitates at the evaporator station.

On the other hand, scientists (Mushtruk et al., 2020a) have studied the use of magnesium oxide for the purification of diffusion juice in the laboratory. As a result of research comparing the classical method of purification and the one proposed by the authors, it was found that the quality of purified juices and syrup was much better, namely the content of colloidal substances and color decreased, but the filtration performance deteriorated.

To date, Ukrainian scientists (Sheiko et al., 2019) have studied the effect of the use of dolomitized limestone on the process of purification of diffusion juice. As a result, recommendations for the use of dolomitized limestone in a sugar, factory have been developed.

Therefore, despite both the positive and negative effects of magnesium on the technological process of the sugar plant, it is very important to monitor the concentration of this component.

The current method of determining the mass fraction of magnesium carbonate in carbonate rocks DSTU 1451-96 (1996), is based on the acidimetric determination of the mass fraction of magnesium carbonate in limestone by calculating the percentage of trilon B solution, which went to titrate the magnesium content to the mass of limestone. The disadvantage of this method is the high

error in determining the mass fraction of magnesium carbonate in limestone.

Other authors presented a method (Palamarchuk et al., 2019), which involves analysis by the complexometric method. The disadvantage of this method is its high cost because a large amount of sample is taken for titration, as well as significantly inflated test results.

It is impossible to give a complete objective chemical analysis of limestone without the use of correct methods of analysis of all components of the carbonate rock. Therefore, the inclusion of the proposed method for the determination of magnesium carbonate in the carbonate rock (Mushtruk et al., 2020b) and the method for the determination of calcium carbonate in the carbonate rock to (Somaratne et al., 2019) comprehensive determination of the chemical composition of limestone will provide objective indicators of $CaCO_3$ and $MgCO_3$. In turn, this will allow the technologist to take the necessary measures in advance to conduct the technological process, which will give the maximum economic effect and save natural resources (limestone, fuel).

Our research aimed to improve the method of determining the mass fraction of magnesium carbonate in the carbonate rock to increase the objectivity of the results and the introduction of an adjusted method to establish the full chemical composition of limestone.

Scientific hypothesis

To increase the accuracy of experimental data, the improved method should be based on the material balance of all components of the carbonate rock. This will make it possible to self-check the accuracy of the results obtained, as having summed up the balance of all components of limestone, approximately we should get a result of 100%. Given this fact and improving the method of determining the content of calcium carbonate and magnesium carbonate in the carbonate rock and entering the results into the material balance, we hope to get the sum of all components approximately 100%, which will indicate the accuracy of the data.

MATERIAL AND METHODOLOGY

Samples

Carbonate rocks of 10 different deposits were used for experiments, namely with (Bogdanivsky, Hlynsko-Rozbyshivsky, Malodivyttsky, Milkivsky, Kybyntsiivsky, Pryluky, Rybalsky, Pivdenno-Panasivsky, Velykobubnivsky, Sukhodolivsky). To determine the concentration of magnesium carbonate, the following utensils and devices were used.

Chemicals

Distilled water (producer «Inter-Synthesis» Limited Liability Company, Ukraine).

Hydrochloric acid (HCl, producer «Inter-Synthesis» Limited Liability Company, Ukraine, chemically pure for analysis).

Sulfuric acid (H_2SO_4 , producer «Inter-Synthesis» Limited Liability Company, Ukraine, chemically pure for analysis).

Hydrofluoric acid (HF, producer «Inter-Synthesis» Limited Liability Company, Ukraine, chemically pure for analysis).

Ammonia (NH₃, producer «Inter-Synthesis» Limited Liability Company, Ukraine, chemically pure for analysis).

Sodium hydroxide (NaOH, producer «Inter-Synthesis» Limited Liability Company, Ukraine, chemically pure for analysis).

Ethyl alcohol (C₂H₅OH, producer «Inter-Synthesis» Limited Liability Company, Ukraine, chemically pure for analysis).

Indicator acid chromium dark blue (C₁₆H₉ClN₂Na₂O₉S₂, producer «Inter-Synthesis» Limited Liability Company, Ukraine, chemically pure for analysis).

Dry murexide indicator (C₈H₈N₆O₆, producer «Inter-Synthesis» Limited Liability Company, Ukraine, chemically pure for analysis).

Trilon B (C₁₀H₁₄N₂Na₂O₈, producer «Inter-Synthesis» Limited Liability Company, Ukraine, chemically pure for analysis).

Ammonium carbonate ((NH₄)₂CO₃, producer «Inter-Synthesis» Limited Liability Company, Ukraine, chemically pure for analysis).

Instruments

Analytical scales (BTHE-6-H1K-1, producer "Inter-Synthesis" Limited Liability Company, Ukraine).

Drying cabinet (DC-300, producer "Laboratory equipment" Limited Liability Company, Ukraine).

Desiccator (EximLab 150, producer "Laboratory equipment" Limited Liability Company, Ukraine).

Chemical cups (CC-100, CC-150, CC-200, CC-250, CC-500, producer "Laboratory equipment" Limited Liability Company, Ukraine).

Petri dish (producer "Inter-Synthesis" Limited Liability Company, Ukraine).

Measuring flasks (MF-100, MF-150, MF-200, MF -250, MF-500, producer "Laboratory equipment" Limited Liability Company, Ukraine).

Muffle furnace (SNOL 8.2/1100, producer "Laboratory equipment" Limited Liability Company, Ukraine).

Measuring pipettes (MP-0.001, MP-0.002, MP-0.005, MP-0.01, MP-0.015, producer "Inter-Synthesis" Limited Liability Company, Ukraine).

Conical flask (CF-100, CF-150, CF-200, CF-250, CF -500, producer "Laboratory equipment" Limited Liability Company, Ukraine).

Burette for titration (producer "Laboratory equipment" Limited Liability Company, Ukraine).

Filters (producer "Laboratory equipment" Limited Liability Company, Ukraine).

Photometer (eXact® Micro 20, producer "Inter-Synthesis" Limited Liability Company, Ukraine).

Laboratory Methods

For technological evaluation of carbonate rocks, their complete chemical analysis was performed, which includes the determination of mass fractions of calcium carbonate (CaCO₃) (according to an improved method); impurities insoluble in hydrochloric acid (SiO₂); the sum of one and a half oxides of aluminum and iron (Fe₂O₃ + Al₂O₃); magnesium carbonate (MgCO₃) (according to an improved method); calcium sulfate (CaSO₄); alkali metal oxides of potassium and sodium (K₂O + Na₂O); moisture content.

Determination of moisture content was carried out by the method of which the essence is that the weight loss of a sample of limestone after drying at a temperature of 105 – 110 °C.

The method of determining the content of the mass fraction of impurities insoluble in hydrochloric acid, which was used was that a portion of the carbonate rock was treated with hydrochloric acid. Silicates that are part of limestone decompose to form metal chlorides and silicic acids. The resulting solution was evaporated and dried to convert the silicic acid into an insoluble form. The precipitate was then washed with hydrochloric acid to convert to a solution of salts of iron, aluminum, and magnesium, which are poorly soluble in water. The washed silicic acids were filtered, calcined, and weighed.

The method of determining the mass fraction of iron and aluminum was based on the treatment of the filtrate obtained after precipitation of impurities insoluble in hydrochloric acid, aqueous ammonia solution. As a result of such actions, iron, and aluminum ions in a slightly alkaline medium precipitate in the form of iron and aluminum hydroxides. The precipitate obtained was filtered off and calcined, after which iron and aluminum oxides were obtained and weighed.

The mass fraction of calcium sulfate was determined by a weight method based on precipitated anion SO₄²⁻-barium chloride, which forms insoluble barium sulfate in an acidic environment. The resulting solution was then filtered, calcined, and weighed.

The content of calcium carbonate and magnesium carbonate was determined by a complexometric method, which is based on the fact that calcium and magnesium with indicators (murexide and acid chromium dark blue) in an alkaline environment form complexes with a certain color. When titrated with Trilon B, these complexes form colorless compounds. The titration is considered complete when the first stable color change of the solution takes place.

To determine the mass fraction of alkali metal oxides of potassium and sodium in the carbonate rock, 1 g of crushed carbonate rock was taken from the prepared sample and placed in a crucible, the carbonate rock was moistened with distilled water; 8 – 10 cm³ of the hydrofluoric acid solution was added. in a ratio of 1:2, then evaporated on an electric stove, without bringing to a boil, until almost complete cessation of sulfuric anhydride vapor. A dry cooled precipitate was obtained in which 8 – 10 cm³ of hydrochloric acid diluted in a ratio of 1:3 was added and heated to dissolve the salts. The container was transferred to a 100 cm³ volumetric flask, cooled, neutralized with ammonia solution until the complete separation of alkali metal oxides of potassium and sodium, added 10 cm³ of saturated ammonium carbonate solution, and adjusted the contents of the flask with distilled water to the mark, mixed and filtered. alkali metals of potassium and sodium in the carbonate rock.

Description of the Experiment

All measurements of instrument readings were performed 5 times. The number of repetitions of each experiment to determine one value was also 5 times.

Statistical Analysis

Mathematical and statistical processing of experimental data was carried out in determining the criteria of Cochran's C test, Fisher, and Student's *t*-test. The accuracy of the data was determined using the Cochran criterion, and the adequacy of the mathematical model was checked using the Fisher and Student criteria.

Statistical processing was performed in Microsoft Excel 2019 values were estimated using mean and standard deviations.

RESULTS AND DISCUSSION

To increase the accuracy of experimental data, the improved method should be based on the material balance of all components of the carbonate rock. This will make it possible to self-check the accuracy of the results obtained, as having summed up the balance of all components of limestone, approximately we should get a result of 100%. The authors of scientific works (Buniowska et al., 2017; Verma et al., 2018; Shmyrin, Kanyugina and Kuznetsov, 2017) engaged in similar research but obtained other scientific results. Given this fact and improving the method of determining the content of calcium carbonate and magnesium carbonate in the carbonate rock and entering the results into the material balance, we hope to get the sum of all components approximately 100%, which will indicate the accuracy of the data.

The establishment of the component composition of limestone began with the determination of its moisture content. According to the results of the research (Figure 2), it is seen that all samples contained a low moisture content. The authors of scientific works (Nadeem et al., 2018; Avalos-Llano, Molina and Sgroppo, 2020; Rahimi et al., 2020) do not believe that the concentration (CO₂) is not an important factor in the technological process of sugar production.

Therefore, the concentration of moisture contained in the experimental samples of limestone will not affect the dissociation process, and consequently the technological process as a whole.

According to the recommendations DSTU 1451-96 (1996), which are put forward to carbonate rocks in Ukraine, to further use them in the sugar industry, the mass fraction of impurities insoluble in hydrochloric acid should not exceed 3%. The obtained data (Figure 3) indicate that samples No. 3, 6, 9, 10 contain an increased concentration of silicon dioxide. The authors of the following scientific papers (Bober et al., 2020; Luo et al., 2019; Dhar et al., 2015) other methods for determining impurities were used for similar scientific research.

During the firing of such limestones, refractory compounds such as calcium and magnesium silicates can be formed. It is these compounds (calcium silicates) that cause irreversible loss of lime. Once on the plant machine, silicates can settle on the heating surface of the evaporator. Given all the above negative consequences, the use of carbonate rocks under No. 3, 6, 9, and 10 in the sugar industry is not possible.

Limestone's were further analyzed for the content of one and a half oxides of aluminum and iron (Al₂O₃ + Fe₂O₃). The authors of scientific works (Guo et al., 2018; Zheplinska, Mushtruk and Salavor, 2020; Jiang, 2015; Almohammed et al., 2015) recommends not to use such samples in the production process.

Super-important components, as they form low-melting aluminates and ferrites from CaO during firing, which are the main component of the liquid phase in the furnace. It (liquid phase) reduces the mass fraction of free lime in quicklime because a significant part of bound lime is formed (in the form of aluminates and calcium ferrites),

and also envelops chemically active lime with non-reactive film, thereby increasing the concentration of inactive lime. Melting leads to soldering of pieces of limestone between themselves and the lining, which in turn increases the amount of overheating and the stability of the lining. The authors of the following scientific works (Ivanova et al., 2021; Lee et al., 2012; Lebovka et al., 2007) recommend not to use such samples in the production process. The approximate composition of quicklime is given in (Noguiera Felix et al., 2019).

Impurities Al₂O₃ + Fe₂O₃ passing into lime milk and further on the machine of the plant get into juice promoting formation in the saturated juices of a deposit which is badly filtered.

According to the recommendations of DSTU 1451-96 (1996) Al₂O₃ + Fe₂O₃, the content of one and a half oxides in limestones, which are suitable for firing in lime-fired kilns at the sugar factory, should be in the amount of not more than 1.5% by weight of dry matter. In subsequent scientific works (Katariya, Arya and Pandit, 2020; Kim et al., 2016; Kozelová et al., 2011; Krasulya et al., 2016) such studies were not conducted.

Observing Figure 3, we see that samples No. 1, 3, 5, 6, 7, 9, 10 contain an increased concentration of iron and aluminum oxides.

Therefore, given the above dangers, the use of limestone with a high content of Al₂O₃ + Fe₂O₃ can not be recommended for the needs of the sugar factory.

The next test component was calcium sulfate (CaSO₄). The recommended normative value DSTU 1451-96 (1996) of CaSO₄ content in carbonate rock is not more than 0.4% by weight of dry matter. The greatest danger that this impurity can cause is that it easily precipitates on the surface of the evaporator. As well as the process of hydration of lime in the presence of CaCO₄ and CaCO₃ is greatly slowed down.

As the results of research (Figure 4) show, samples No. 1, 3, 6, 10 exceed the recommended norms (calcium sulfate content), so their use will lead to the above negative consequences.

Determination of calcium and magnesium carbonate content was carried out as follows:

Pipette 15 cm³ of solution into a 250 cm³ conical flask, add 100 cm³ of distilled water and 25 cm³ of 20% sodium hydroxide solution from a volumetric flask containing the filtrate after precipitation of iron and aluminum oxides. After 1 – 2 min on the tip of a spatula made 0.1 – 0.2 g of murexide in a mixture with potassium chloride. The solution was stirred and titrated with 0.1 M solution of Trilon B until the transition from red to purple, which was observed on a black background. The authors of scientific works (Dehghannya et al., 2018; Zheplinska et al., 2021; Zhu et al., 2016; Zdziennicka et al., 2017) used other standardized methods to conduct similar studies.

In parallel with the breakdown, distilled water was monitored for calcium carbonate content. To control the distilled water for the content of calcium carbonate, 25 cm³ of 20% sodium hydroxide solution was added to the distilled water. After 1 – 2 min on the tip of a spatula made 0.1 – 0.2 g of murexide in a mixture with potassium chloride. The solution was stirred and titrated with 0.1 M solution of Trilon B until a transition from red to purple, which was observed on a black background.



Figure 1 Photo of the final product (beet sugar).

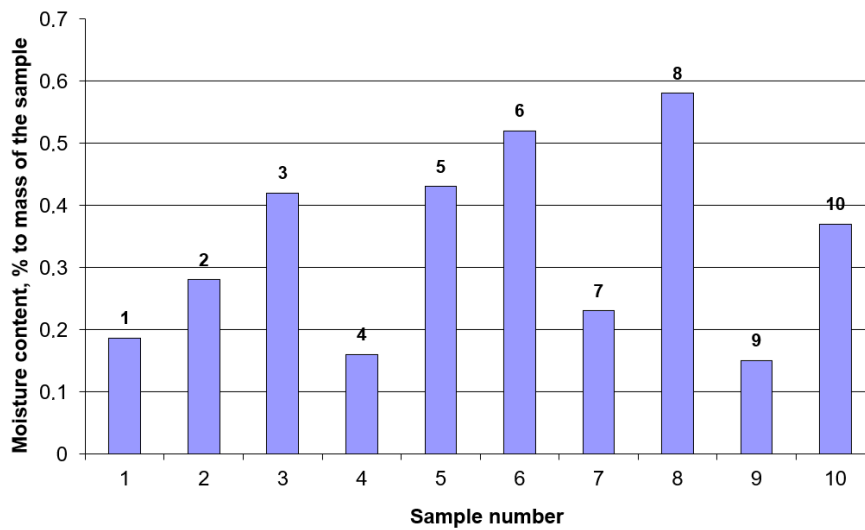


Figure 2 Moisture content in the studied samples of carbonate rocks.

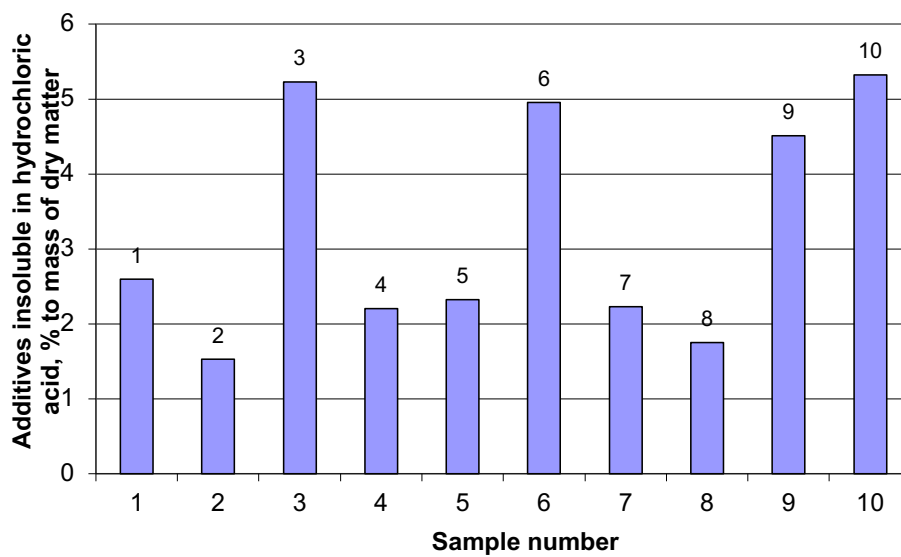


Figure 3 The content of additives insoluble in hydrochloric acid in the studied samples of carbonate rocks.

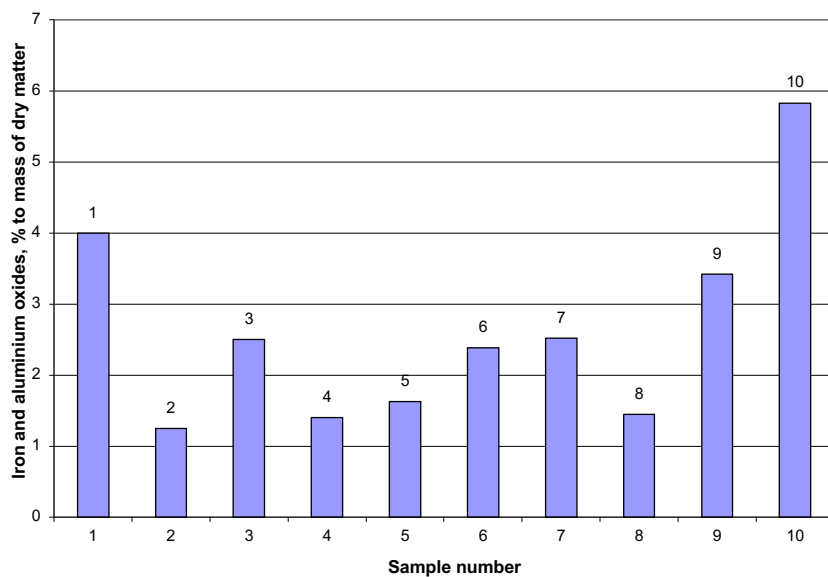


Figure 4 The content of iron and aluminum oxides in the studied samples of carbonate rocks.

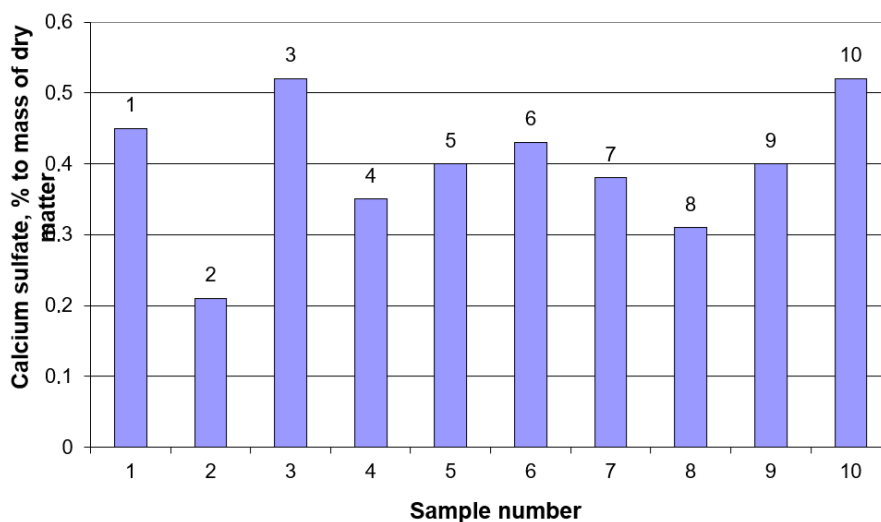


Figure 5 The content of calcium sulfate in the studied samples of carbonate rocks.

Table 1 The content of calcium carbonate in the studied samples of carbonate rocks.

Sample	According to DSTU 1451-96 (1997)		According to the newly introduced method	
	% to probe mass	% to dry matter mass	% to probe mass	% to dry matter mass
No. 1	180.68	181.02	90.23	90.4
No. 2	190.33	190.86	95.03	95.3
No. 3	176.09	176.83	87.82	88.20
No. 4	186.89	187.19	93.35	93.5
No. 5	185.19	185.98	92.4	92.8
No. 6	177.83	178.75	88.86	89.32
No. 7	182.53	182.99	91.12	91.35
No. 8	187.25	188.34	93.66	94.2
No. 9	174.85	175.11	87.37	87.5
No. 10	167.25	167.87	83.49	83.8

The difference between the volume of 0.1 M solution of Trilon B, which went to the titration of the sample, which used the filtrate after precipitation of iron and aluminum oxides, and the volume of 0.1 M solution of Trilon B, which went to the titration, which controlled distilled water, makes it possible to set the volume of 0.1 M solution of Trilon B, which went to the titration of calcium – b, cm³. After that, the mass fraction of calcium carbonate in the carbonate rock was calculated (Smetanska et al., 2021).

In Ukraine, only high-calcium carbonate rocks are used for the sugar industry, in which the CaCO₃ content is at least 93% by weight of dry matter. The results of determining the content of the mass fraction of calcium carbonate in the carbonate rock are given in Table 1 and Figure 5.

Observing Figure 5 we see that samples No. 1, 3, 5, 6, 7, 9, 10 are not recommended for use in the technological process of sugar production due to the low content of calcium carbonate.

Control over the content of magnesium carbonate in the carbonate rock was carried out as follows:

The volume of Trilon B spent on titration of magnesium in the carbonate rock was calculated by the difference between the volumes of Trilon B spent on titration of calcium and magnesium – a, and separately calcium – b.

To determine the volume of Trilon B, which went to the titration of calcium and magnesium, from the filtrate after precipitation of iron and aluminum oxides, took a sample of 15 cm³, which corresponds to 0.15 g of carbonate rock and is a sample of carbonate rock – m, in grams, which was taken into account during the calculation of the mass fraction of magnesium carbonate in the carbonate rock according to formula 1. To the sample was added 100 cm³ of distilled water, 10 cm³ of ammonia buffer solution and on the tip of a spatula made 0.1 – 0.2 g of dark chromium acid indicator mixture, stirred vigorously with a glass rod, and titrated with 0.1 M solution of Trilon B until the transition of red-purple to blue. The author of (Sasikumar, Chutia and Deka, 2019) used other standardized methods to conduct similar studies.

In parallel with the breakdown, distilled water was monitored for magnesium and calcium carbonate content. Control of distilled water for the content of magnesium and calcium carbonate involves the introduction into the distilled water of 10 cm³ of ammonia-buffer solution and dark blue acid chromium indicator, followed by titration of water with 0.1 M solution of Trilon B. The difference between the volume of 0.1 M solution of Trilon B, spent on the titration of the sample, which uses the filtrate after precipitation of iron and aluminum oxides, and a volume of 0.1 M solution of Trilon B, spent on titration, where the control of distilled water, made it possible to establish the volume of 0.1 M solution of Trilon B spent on titration of calcium and magnesium – a, cm³.

The mass fraction of magnesium carbonate in the carbonate rock was calculated by the formula:

$$X = \left[\frac{(a-b) \cdot K \cdot 0.004498 \cdot 100}{m} \right] \cdot \frac{100}{100-W}, \quad (1)$$

Where:

X – mass fraction of magnesium carbonate in the carbonate rock, %;

a is the volume of 0.1 M solution of Trilon B, which went to the titration of calcium and magnesium, cm³;

b is the volume of 0.1 M solution of Trilon B, which went to the titration of calcium, cm³;

0.004498 – coefficient indicating the mass of magnesium carbonate, which titrates 1 cm³ of Trilon B solution with a concentration of 0.1 mol.dm⁻³, g;

K is the correction factor for the solution of Trilon B;

m – portion of carbonate rock, corresponding to 0.15 g;

W – a mass fraction of moisture, %.

The results of determining the mass fraction of magnesium carbonate in the carbonate rock are shown in Table 2 and Figure 7.

From Table 2 and Table 3, and Figure 6 we see that the results of research obtained by the method DSTU 1451-96 (1996) have slightly inflated indicators. Therefore, the above-mentioned improved method for determining MgCO₃ was proposed, which gives more accurate results (Table 3).

Given that the share of magnesium carbonate in carbonate rocks should not exceed 2.5% DSTU 1451-96 (1996), we see that samples No. 3, 5, 6, 9, 10 do not meet the stated standards. Therefore, in those samples where there is an inflated content of MgCO₃ technologist, it is necessary to correct the process following the recommendations given at the beginning of the article.

Control of alkali metal oxides of potassium and sodium (K₂O + Na₂O) is carried out to reduce the concentration of molasses in the system, which they are. K₂O and Na₂O also form fusible compounds during firing. The mass fraction of potassium and sodium oxides in the carbonate rock according to the recommendations DSTU 1451-96 (1996) should not exceed the amount of not more than 0.25% by weight of dry matter. Figure 8 shows that samples No. 1 and 3 cannot be recommended for use in the sugar industry.

To objectively conduct a technological audit of the lime department of the sugar factory, it is necessary to use the correct methods of control of the parameters of technological indicators. One component of the technological audit of the lime department is the chemical composition of the carbonate rock, which is fed for firing. The quality of finished products and their cost will mainly depend on the condition of raw materials. Therefore, to objectively assess the quality of the studied limestones, the concentration of all components included in them was summarized in one table (Table 3) and the balance of all impurities and calcium carbonate was adjusted for each sample. When determining the chemical composition of the carbonate rock, the sum of the mass fractions of CaCO₃ and impurities should be approximately 100%. From the Table 3 we see that all analyzed samples according to the method DSTU 1451-96 (1996) have inflated results. In turn, the lined material balance of all components of the carbonate rock and in all samples according to the proposed method (Cervantes-Elizarrarás et al., 2017) approached 100%. This indicates the high accuracy of the method and the possibility of its use in the production process.

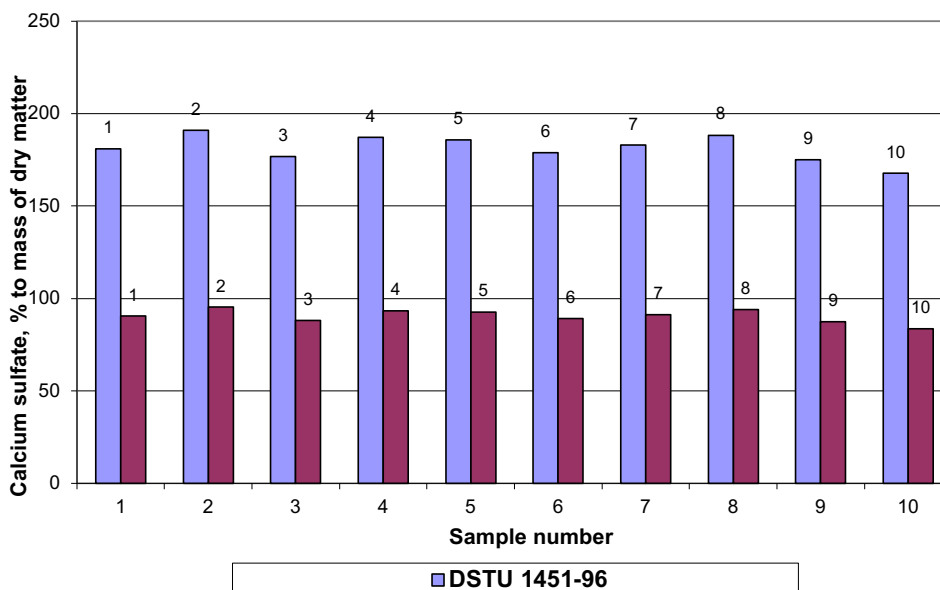


Figure 6 The content of calcium carbonate in the studied samples of carbonate.

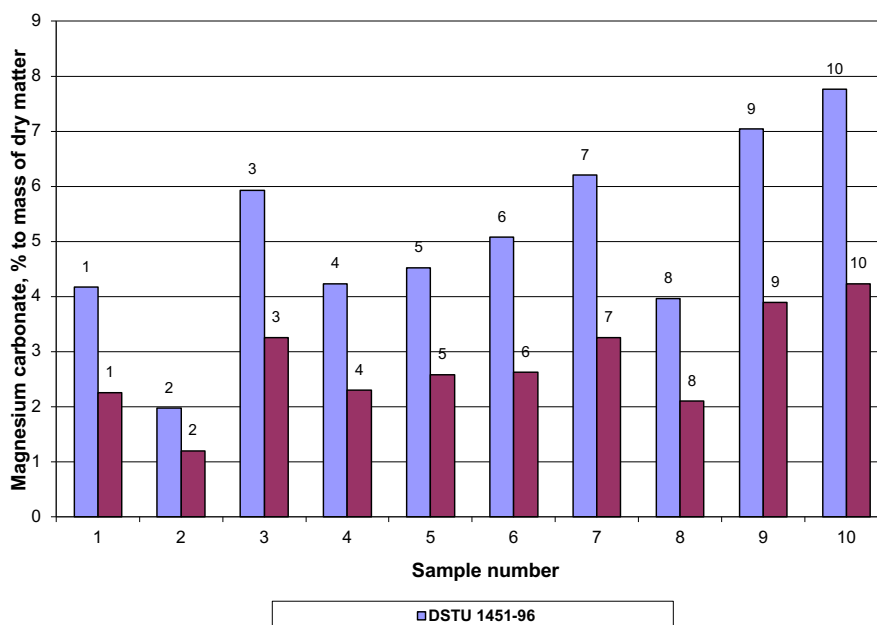


Figure 7 The content of magnesium carbonate in the studied samples of carbonate rocks.

Table 2 The content of magnesium carbonate in the studied samples of carbonate rocks.

Sample	According to DSTU 1451-96 (1997)		According to the newly introduced method	
	% to probe mass	% to dry matter mass	% to probe mass	% to probe mass
No. 1	4.16	4.17	2.25	2.25
No. 2	1.97	1.97	1.2	1.2
No. 3	5.90	5.93	3.24	3.25
No. 4	4.22	4.23	2.15	2.3
No. 5	4.50	4.52	2.57	2.58
No. 6	5.06	5.08	2.61	2.62
No. 7	6.18	6.2	3.24	3.25
No. 8	3.94	3.96	2.08	2.10
No. 9	7.03	7.04	3.88	3.89
No. 10	7.73	7.76	4.21	4.23

Table 3 Chemical composition of the studied samples of carbonate rocks.

Carbon bedrock component		Sample 1				Sample 2			
		Following method DSTU 1451-96 (1997)		According to the newly introduced method		Following method DSTU 1451-96 (1997)		According to the newly introduced method	
		% to probe mass	% to dry matter mass	% to probe mass	% to dry matter mass	% to probe mass	% to dry matter mass	% to probe mass	% to dry matter mass
1	2	3	4	5	6	7	8	9	10
1.	Moisture	0.186		0.186		0.28		0.28	
	Moderate	0.186	-	0.186	-	0.28	-	0.28	-
	Impurities insoluble in hydrochloric acid, (SiO ₂)	2.6	2.6	2.6	2.6	1.52	1.53	1.52	1.53
2.	Moderate	2.6	2.6	2.6	2.6	1.52	1.53	1.52	1.53
	Oxides (Al ₂ O ₃ +Fe ₂ O ₃)	3.96	3.99	3.96	3.99	1.25	1.25	1.25	1.25
3.	Moderate	3.98	4.10	3.98	4.10	1.25	1.25	1.25	1.25
	Calcium carbonate (CaCO ₃)	3.97	4.0	3.97	4.0	1.25	1.25	1.25	1.25
4.	Moderate	180.67	181.01	90.24	90.5	190.32	190.85	95.03	95.3
	Magnesium carbonate (MgCO ₃)	180.69	181.03	90.22	90.3	190.34	190.87	95.03	95.3
5.	Moderate	180.68	181.02	90.23	90.4	190.33	190.86	95.03	95.3
	Calcium sulfate (CaSO ₄)	4.16	4.16	2.24	2.24	1.97	1.97	1.2	1.2
6.	Moderate	4.16	4.18	2.26	2.26	1.97	1.97	1.2	1.2
	Alkali metal oxides (K ₂ O+Na ₂ O)	4.16	4.17	2.25	2.25	1.97	1.97	1.2	1.2
7.	Moderate	0.45	0.45	0.45	0.45	0.21	0.21	0.21	0.21
	Summary of the carbon bedrock component	0.45	0.45	0.45	0.45	0.21	0.21	0.21	0.21
		0.30	0.30	0.30	0.30	0.15	0.15	0.15	0.15
		0.30	0.30	0.30	0.30	0.15	0.15	0.15	0.15
			192.54		100		195.97		99.64

Continuation of Table 3.

Carbon bedrock component		Sample 3				Sample 4			
		Following method DSTU 1451-96 (1997)		According to the newly introduced method		Following method DSTU 1451-96 (1997)		According to the newly introduced method	
		% to probe mass	% to dry matter mass	% to probe mass	% to dry matter mass	% to probe mass	% to dry matter mass	% to probe mass	% to dry matter mass
1	2	3	4	5	6	7	8	9	10
1.	Moisture	0.42		0.42		0.16		0.16	
	Moderate	0.42	-	0.42	-	0.16	-	0.16	-
2.	Impurities insoluble in hydrochloric acid, (SiO ₂)	5.21	5.23	5.21	5.23	2.20	2.20	2.20	2.20
	Moderate	5.21	5.23	5.21	5.23	2.20	2.20	2.20	2.20
3.	Oxides (Al ₂ O ₃ +Fe ₂ O ₃)	2.49	2.50	2.49	2.50	1.4	1.4	1.4	1.4
	Moderate	2.49	2.50	2.49	2.50	1.4	1.4	1.4	1.4
4.	Calcium carbonate (CaCO ₃)	176.08	176.82	87.82	88.20	186.90	187.20	93.35	93.5
	Moderate	176.10	176.84	87.82	88.20	186.88	187.18	93.35	93.5
5.	Magnesium carbonate (MgCO ₃)	5.90	5.93	3.24	3.25	4.22	4.23	2.15	2.3
	Moderate	5.90	5.93	3.24	3.25	4.22	4.23	2.15	2.3
6.	Calcium sulfate (CaSO ₄)	0.52	0.52	0.52	0.52	0.35	0.35	0.35	0.35
	Moderate	0.52	0.52	0.52	0.52	0.35	0.35	0.35	0.35
7.	Alkali metal oxides (K ₂ O+Na ₂ O)	0.28	0.28	0.28	0.28	0.20	0.20	0.20	0.20
	Moderate	0.28	0.28	0.28	0.28	0.20	0.20	0.20	0.20
	Summary of the carbon bedrock component		191.29		99.98		195.57		99.95

Continuation of Table 3.

Carbon bedrock component		Sample 5				Sample 6			
		Following method DSTU 1451-96 (1997)		According to the newly introduced method		Following method DSTU 1451-96 (1997)		According to the newly introduced method	
		% to probe mass	% to dry matter mass	% to probe mass	% to dry matter mass	% to probe mass	% to dry matter mass	% to probe mass	% to dry matter mass
1	2	3	4	5	6	7	8	9	10
1.	Moisture	0.43		0.43		0.52		0.52	
	Moderate	0.43	-	0.43	-	0.52	-	0.52	-
2.	Impurities insoluble in hydrochloric acid, (SiO ₂)	2.31	2.32	2.31	2.32	4.92	4.95	4.92	4.95
	Moderate	2.31	2.32	2.31	2.32	4.92	4.95	4.92	4.95
3.	Oxides (Al ₂ O ₃ +Fe ₂ O ₃)	1.62	1.63	1.62	1.63	2.37	2.38	2.37	2.38
	Moderate	1.62	1.63	1.62	1.63	2.37	2.38	2.37	2.38
4.	Calcium carbonate (CaCO ₃)	185.18	185.97	92.4	92.8	177.83	178.75	88.86	89.32
	Moderate	185.20	185.99	92.4	92.8	177.83	178.75	88.86	89.32
5.	Magnesium carbonate (MgCO ₃)	185.19	185.98	92.4	92.8	177.83	178.75	88.86	89.32
	Moderate	4.50	4.52	2.57	2.58	5.06	5.08	2.61	2.62
6.	Calcium sulfate (CaSO ₄)	4.50	4.52	2.57	2.58	5.06	5.08	2.61	2.62
	Moderate	0.4	0.4	0.4	0.4	0.43	0.43	0.43	0.43
7.	Alkali metal oxides (K ₂ O+Na ₂ O)	0.4	0.4	0.4	0.4	0.43	0.43	0.43	0.43
	Moderate	0.23	0.23	0.23	0.23	0.21	0.21	0.21	0.21
	Moderate	0.23	0.23	0.23	0.23	0.21	0.21	0.21	0.21
	Summary of the carbon bedrock component		195.08		99.96		191.8		99.91

Continuation of Table 3.

Carbon bedrock component		Sample 7				Sample 8			
		Following method DSTU 1451-96 (1997)		According to the newly introduced method		Following method DSTU 1451-96 (1997)		According to the newly introduced method	
		% to probe mass	% to dry matter mass	% to probe mass	% to dry matter mass	% to probe mass	% to dry matter mass	% to probe mass	% to dry matter mass
1	2	3	4	5	6	7	8	9	10
1.	Moisture	0.23		0.23		0.58		0.58	
	Moderate	0.23	-	0.23	-	0.58	-	0.58	-
2.	Impurities insoluble in hydrochloric acid, (SiO ₂)	2.22	2.23	2.22	2.23	1.74	1.75	1.74	1.75
	Moderate	2.22	2.23	2.22	2.23	1.74	1.75	1.74	1.75
3.	Oxides (Al ₂ O ₃ +Fe ₂ O ₃)	2.51	2.52	2.51	2.52	1.44	1.45	1.44	1.45
	Moderate	2.51	2.52	2.51	2.52	1.44	1.45	1.44	1.45
4.	Calcium carbonate (CaCO ₃)	182.53	182.99	91.12	91.35	187.26	188.35	93.66	94.2
	Moderate	182.53	182.99	91.12	91.35	187.24	188.33	93.66	94.2
5.	Magnesium carbonate (MgCO ₃)	6.18	6.2	3.24	3.25	3.94	3.96	2.08	2.10
	Moderate	6.18	6.2	3.24	3.25	3.94	3.96	2.08	2.10
6.	Calcium sulfate (CaSO ₄)	0.38	0.38	0.38	0.38	0.31	0.31	0.31	0.31
	Moderate	0.38	0.38	0.38	0.38	0.31	0.31	0.31	0.31
7.	Alkali metal oxides (K ₂ O+Na ₂ O)	0.20	0.20	0.20	0.20	0.18	0.18	0.18	0.18
	Moderate	0.20	0.20	0.20	0.20	0.18	0.18	0.18	0.18
Summary of the carbon bedrock component			194.52		99.93		195.99		99.99

Continuation of Table 3.

Carbon bedrock component		Sample 9				Sample 10			
		Following method DSTU 1451-96 (1997)		According to the newly introduced method		Following method DSTU 1451-96 (1997)		According to the newly introduced method	
		% to probe mass	% to dry matter mass	% to probe mass	% to dry matter mass	% to probe mass	% to dry matter mass	% to probe mass	% to dry matter mass
1	2	3	4	5	6	7	8	9	10
1.	Moisture	0.15		0.15		0.37		0.37	
	Moderate	0.15	-	0.15	-	0.37	-	0.37	-
2.	Impurities insoluble in hydrochloric acid, (SiO ₂)	4.50	4.51	4.50	4.51	5.30	5.32	5.30	5.32
	Moderate	4.50	4.51	4.50	4.51	5.30	5.32	5.30	5.32
3.	Oxides (Al ₂ O ₃ +Fe ₂ O ₃)	3.41	3.42	3.41	3.42	5.81	5.83	5.81	5.83
	Moderate	3.41	3.42	3.41	3.42	5.81	5.83	5.81	5.83
4.	Calcium carbonate (CaCO ₃)	174.85	175.11	87.37	87.5	167.26	167.88	83.49	83.8
	Moderate	174.85	175.11	87.37	87.5	167.24	167.86	83.49	83.8
5.	Magnesium carbonate (MgCO ₃)	7.03	7.04	3.88	3.89	7.73	7.76	4.21	4.23
	Moderate	7.03	7.04	3.88	3.89	7.73	7.76	4.21	4.23
6.	Calcium sulfate (CaSO ₄)	0.4	0.4	0.4	0.4	0.52	0.52	0.52	0.52
	Moderate	0.4	0.4	0.4	0.4	0.52	0.52	0.52	0.52
7.	Alkali metal oxides (K ₂ O+Na ₂ O)	0.23	0.23	0.23	0.23	0.20	0.20	0.20	0.20
	Moderate	0.23	0.23	0.23	0.23	0.20	0.20	0.20	0.20
Summary of the carbon bedrock component		190.71		99.95		187.5		99.9	

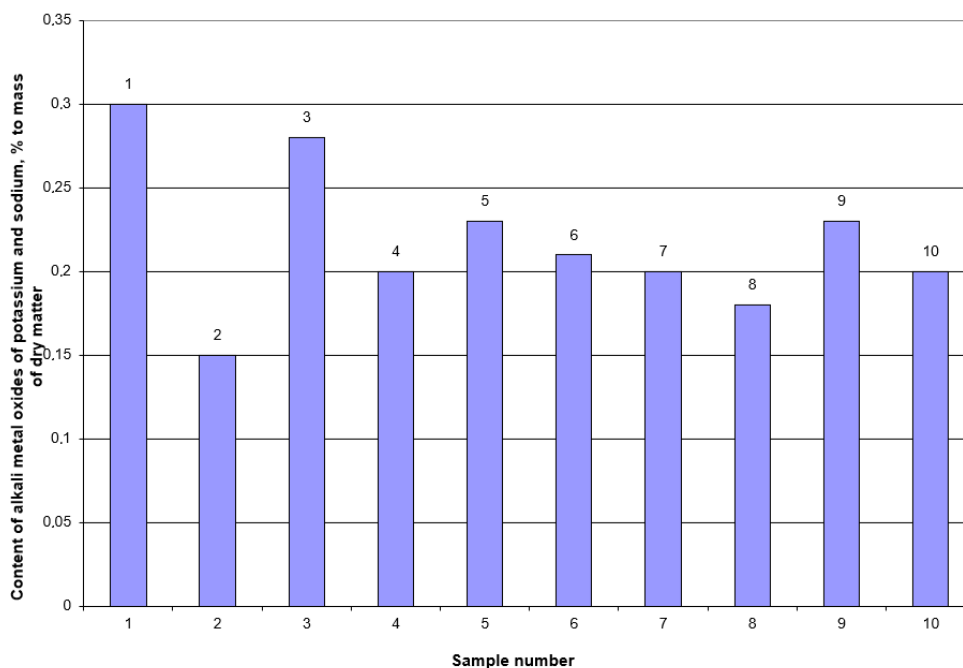


Figure 8 The content of alkali metal oxides of potassium and sodium in the studied samples of carbonate rocks.

CONCLUSION

Therefore, the use of the proposed method for determining the content of magnesium and calcium carbonate in limestone will allow the technologist to obtain timely objective information on the component composition of the carbonate rock. Knowing the exact concentration of $MgCO_3$, you can use its positive effect on the technological process, eliminating the negative factors of technological methods. Thus, as a result, we get finished products of good quality, save natural resources (limestone, fuel) and improve the ecological condition of the planet (by reducing harmful emissions into the atmosphere).

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