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ИССЛЕДОВАНИЕ НОВОГО МНОГОФУНКЦИОНАЛЬНОГО ИНГИБИТОРА В ЛАБОРАТОРНЫХ УСЛОВИЯХ

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Впервые в лабораторных условиях изучены бактерицидно-ингибиторные свойства реагента С-1 в пластовых водах, содержащих сероводород и одновременно сероводород и углекислый газ, и на питательной среде «Постгейт-Б», содержащей сульфатредуцирующие бактерии «Desulfomicrobium» и «Desulfovibriodesulfurican». В эксперименте использовали концентрации 10, 15, 20, 25 и 30 мг/л реагента. Скорость коррозии в сероводородной среде при концентрации исследуемого реагента 30 мг/л составляет 0,0864 г/м^2·ч, а защитный эффект (Z) – 98,2%, в то время как в пластовой воде, где присутствуют оба газа, скорость коррозии составляет 0,252 г/м, а защитный эффект составляет 96,5%. При исследовании бактерицидного свойства реагента установлено, что он в большей степени снижает численность бактериальных клеток Desulfovibriodesulfurican по сравнению с бактериями Desulfomicrobium. По концентрации сероводорода, продуцируемого бактериями, рассчитывали бактерицидный эффект (S, %) реагента С-1 в среде, содержащей обе бактерии. На среде, содержащей Desulfovibriodesulfurican SRB, при концентрации ингибитора 30 мг/л величина бактерицидного эффекта составила 68% в первые сут., 80% - на вторые сут., 85% - на третьи сут., 90% - на четвертый день, 91% - на пятый день, 92% - на шестой день и 93% - на седьмой день. У бактерий Desulfomicrobium величина S составляет 55, 70, 80, 83, 84, 85, 85% соответственно. Таким образом, установлено, что реагент С-1 резко снижает количество биогенного сероводорода в питательной среде «Постгейт Б» с участием сульфатредуцирующих бактерий, но не останавливает полностью процесс сульфатредукции.

Ключевые слова: ингибитор, состав, реагент, коррозия, пластовая вода, сероводород, углекислый газ, сульфатредуцирующие бактерии, бактерицидное действие

STUDY OF A NEW MULTIFUNCTIONAL INHIBITOR UNDER LABORATORY CONDITION

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For the first time, the bactericidal-inhibitory properties of the S-1 reagent were studied in laboratory conditions in reservoir waters containing hydrogen sulfide and at the same time hydrogen sulfide and carbon dioxide, and in "Postgate-B" nutrient medium sulfate-reducing bacteria "Desulfomicrobium" and "Desulfovibriodesulfuricans" containing. Concentrations of 10, 15, 20, 25 and 30 mg/l of the reagent were used during the experiment. The corrosion rate in the hydrogen sulphide environment at a concentration of 30 mg/l of the studied reagent is 0.0864 g/m^2·hour, and the protection effect (Z) is 98.2%, while in the reservoir water where both gases are present, the corrosion rate is 0.252 g/m, and the protection effect is 96.5%. During the investigation of the bac-

tericidal property of the reagent, it was determined that it reduces the number of Desulfovibriodesulfuricans bacterial cells more in comparison with Desulfomicrobium bacteria. Based on the concentration of hydrogen sulfide produced by bacteria, the bactericidal effect (S,%) of reagent S-1 was calculated in the both bacteria containing medium. In the medium containing Desulfovibriodesulfuricans SRB, at a concentration of 30 mg/l of the inhibitor, the value of the bactericidal effect was 68% on the first day, 80% - on the second day, 85% - on the third day, 90% - on the fourth day, 91% - on the fifth day, 92% - on the sixth day, and 93% - on the seventh day. In Desulfomicrobium bacteria, the value of S is 55, 70, 80, 83, 84, 85, 85%, respectively. Thus, it was found that reagent C-1 sharply reduces the amount of biogenic hydrogen sulfide in the nutrient medium "Postgate B" with the participation of sulfate-reducing bacteria, but does not completely stop the process of sulfate reduction.

Key words: inhibitor, composition, reagent, corrosion, reservoir water, hydrogen sulfide, carbon dioxide, sulfate-reducing bacteria, bactericidal effect

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INTRODUCTION

Ensuring the reliability and continuity of the operation of oil industry equipment, including pipeline systems, is one of the most important tasks in the development of oil and gas fields and the subsequent transportation of hydrocarbons. However, the corrosion aggressiveness of the operating environment in this industry is quite high, and in its turn, this is the transformation of the operating environment into an aggressive corrosion environment due to mineralized formation water, hydrogen sulfide, carbon dioxide and, most importantly, sulfate-reducing bacteria. [1-5]. The most dangerous for gas and oil pipelines is condensate formed when the temperature of oil and gas drops. Condensate combines a two-phase corrosion system and corrosion processes occur in its water part [6-10]. Corrosion of well steel equipment, as well as highway and technological pipelines shortens their service life and increases repair costs, and can also cause serious damage to the environment. Thus, damage to equipment can lead to salinization of the soil with aggressive groundwater, contamination of the soil and natural water pools with oil and its products [11-13].

One of the effective methods of corrosion protection of field equipment and pipelines in the oil and gas industry is the use of corrosion inhibitors. For this, individual combinations or compositions of a number of substances are used, which contribute to the sharp reduction of corrosion losses of metals in harsh conditions with mineral salts and aggressive gases (H_2S, O_2, CO_2) and sulfate-reducing bacteria in technological environments. Inhibitor protection of field equipment and pipelines from corrosion. is the most widespread and economically justified method [14]. By changing the concentration of the inhibitor or using an inhibitor with different anti-corrosion properties, it is possible to reduce the corrosion rate to a minimum level without making fundamental changes in the existing technological schemes [15]. At present, inhibiting the internal surface of underground oil pipelines is the main method of internal corrosion protection. Protection of installations and equipment from corrosion caused by aggressive environments is one of the important tasks facing petroleum engineers. The use of inhibitors is one of the most effective methods of combating corrosion of metals in various aggressive environments. [16-20].

The purpose of the work is to study the protective effectiveness of the new multifunctional corrosion inhibitor under laboratory conditions.

RESEARCH METHODOLOGY

The process of internal corrosion is caused by the contact of the internal surface of the pipeline with liquid. It is known that formation waters contain sodium, magnesium and calcium chlorides, and in some cases sulfides, bromides, iodides and borates. Besides CO_2 and O_2, elemental sulfur, H_2S, mercaptans and other organic sulfur compounds can also be dissolved in formation water. From the sulfur compounds, hydrogen sulfide is the most aggressive corrosion agent against steel equipment. The aggressiveness of other sulfur compounds is mainly due to their ability to produce H_2 as a result of their decomposition. Carbon dioxide unambiguously increases the corrosion aggressiveness of formation water. The reason for this is the

decrease in the pH of the environment during the dissolution of carbon dioxide in the formation water. The presence of oxygen in the formation water (solubility decreases with the increase in salinity of the water) facilitates the depolarization of the corrosion process.

In the presented article, Ct3 steel samples with 30×20×1 mm dimensions were used during laboratory tests. The area of the samples taken for testing is calculated according to the following formula:

$$S_N = 2ah + 2ab + 2hb$$

where, S_N – area of the steel sample, m^2; a – sample length, mm; b – sample width, mm; h – height of the sample, mm.

Since a = 30mm, b = 20mm, h = 1mm, the area of the steel sample taken for testing was $S_N = 2\cdot30\cdot1 + 2\cdot30\cdot20 + 2\cdot20\cdot1 = 1300$ mm^2 = 0.0013 m^2.

The surface of the steel plates was polished on a grinding machine in accordance with the requirements of the GOST 2789-73 standard, and after cleaning the surfaces with acetone and alcohol, they were washed with distilled water. In order to activate the surface, the steel plates were kept in a 15% HCl solution for 60 s, after it was washed first in running water then – in distilled water, dried with filter paper, kept in a desiccator for sixty minutes, and weighed on an analytical balance with an accuracy of $5\cdot10^{-5}$ g. The experiments were carried out in parallel in the same medium without and with the inhibitor for comparison [21]. To conduct the laboratory test, steel samples prepared according to the regulations were placed in the corrosion environment without inhibitor and inhibitor added and the time was recorded.

After the laboratory test was completed, the steel plates were removed from the environment and cleaned of corrosion products on the surface. For this, the plates were cleaned with cotton in a solution made of 10% hydrochloric acid and 40% formalin, washed in running water and dried with acetone. The plates were kept in a desiccator for 10-12 h to bring them to a constant weight both before and after the experiment. The plates were then weighed again [22].

The gravimetric method is used to test corrosion inhibitors. The method consists of determining the mass loss of metal samples in test environments without and with inhibitors, and then evaluating the protective effect of the inhibitor based on the change in corrosion rate. The water part of the water-oil environment was chosen as the test medium, because the well oil samples presented contain suspended formation water [28-29].

Metal loss (Δm) is calculated by the following equation.

$$\Delta m = m_0 - m_1$$

During the experiment, metal loss is calculated for three steel plates and the average mass is found. During gravimetric tests, the mass indicator of the corrosion rate in both reagentless and reagent media is characterized by K_m and is calculated by the following mathematical equation [23].

$$K_m = \frac{m_0 - m_1}{S\cdot\tau},\ q/(m^2\cdot h)$$

where, m_0 is the mass of the sample before the tests, g; m_1 – mass of the sample after the tests, g; S – average surface area calculated for three samples, m_2; τ – duration of the test, hours.

Based on the corrosion rate, the penetration coefficient is determined as follows.

$$K_p = \frac{8760K_m}{\rho}\cdot10^{-3}\ \text{mm/year}$$

where, K_p – penetration coefficient, mm/year, K_m – corrosion rate, g/m^2·h, ρ – density of the investigated metal, g/cm^2, 8760 – the number of h in a year as a constant quantity.

The equation used to calculate the retardation factor is as following.

$$\gamma = \frac{K_0}{K_{inh}}$$

where, K_0 – without inhibitors, $K_{inh.}$ – corrosion rate in the presence of an inhibitor, (g/m^2·h).

The protective effect of the inhibitor is calculated by the following equation [23, 24].

$$Z = \frac{K_0 - K_{inh}}{K_0}100\%$$

where, K_0 – Corrosion rate of sample in medium without inhibitor (g/m^2·h), $K_{inh.}$ – corrosion rate of the sample in inhibitory medium (g/m^2·h).

"Postgate-B" nutrient medium was used for germination and development of sulfate-reducing bacteria, and the experiments were conducted according to NASE standard methodology [25]. Microscopic appearance of bacteria was studied in MBI-6 microscope. A limited dilution method was used to quantify viable tissues.

The number of sulfate-reducing bacteria cells in 1 ml of initial suspension is calculated by the following formula [25]:

$$M = \frac{1000an}{hs}$$

where M is the number of cells in 1 ml suspension, a – the average number of cells per square cell, h – chamber depth (in mm) n_0, S – area of the square mesh (in mm^2), n – the degree of dilution of the suspension.

$$1000\ \text{mm}^3 = 1\ \text{ml}$$

The growth factor (N, %) of sulfate-reducing bacterial cells in the presence of bactericide is calculated by the following equation [26]:

$$N, \% = \frac{100(n_0 - n_{inh})}{n_0}$$

where, n_0 – the number of microorganisms in reagent-free medium, n_{inh} – the number of microorganisms in the reagent medium.

According to the amount of hydrogen sulfide, the bactericidal effect of the reagent (S, %) is calculated by the following formula [26]:

$$S, \% = \frac{C_0 - C_{inh}}{C_{inh.}} 100$$

where, C_0 is concentration of biogenic hydrogen sulfide in reagent-free medium, C_{inh} – concentration of biogenic hydrogen sulfide in the reagent medium.

The coefficient of variation of the concentration of hydrogen sulfide (γ_C) is calculated by the following mathematical equation [26]:

$$\gamma_C = \frac{C(H_2S)_0}{C(H_2S)_{inh.}}$$

where, $C(H_2S)_0$ is concentration of hydrogen sulfide in reagent-free medium, $C(H_2S)_{inh}$ – concentration of hydrogen sulfide in the reagent medium.

The mixture of "Desulfomicrobium" and "Desulfovibriodesulfuricans" species of sulfate-reducing bacteria was used for the study.

RESULTS AND THEIR DISCUSSION

A significant influence on the corrosion of equipment during field development, operation and oil transportation is related to parameters such as the pH of formation water and the presence of dissolved oxygen in water. Corrosion of metals is accelerated by salts dissolved in mineralized water and high temperature of formation water [9]. The presence of hydrogen sulfide (H_2S), carbon dioxide (CO_2) and SRB in formation waters is considered the most important threat in the corrosion of metal equipment.

1.3-Dichloro-2 (2-iodo-1)(propanin-2-1yl)oxy)-ethyl) benzene $C_{11}H_9Cl_{2l}O$. (provisional name-S-1) was used as an inhibitor to conduct laboratory tests for determining the effectiveness of inhibitors. [27].

The efficiency of S-1 inhibitor was studied under laboratory conditions in formation waters containing hydrogen sulfide (H_2S) and carbon dioxide (CO_2) and in "Postgate-B" nutrient medium containing sulfate-reducing bacteria "Desulfomicrobium" and "Desulfovibriodesulfuricans".

Table 1 presents the results of laboratory tests to determine the effectiveness of inhibitor S-1 in oil and reservoir waters containing hydrogen sulfide (H_2S).

As it can be seen from Table 1, the corrosion protection efficiency increases wit the inhibitor concentration increase in oil and hydrogen sulfide containing formation water.

The highest effect is observed at a reagent concentration of 30 mg/l. At this time, the corrosion rate is 0.0864 g/m^2·h, and the protective effect of the inhibitor (Z) is 98.2% (Table 1).

Table 2 shows the results of laboratory tests conducted on the effectiveness of S-1-inhibitor in formation water containing both hydrogen sulfide (H_2S) and carbon dioxide (CO_2).

Table 2 shows that the corrosion rate is higher in the environment where hydrogen sulfide and carbon dioxide coexist, and this can be explained by increasing the acidity of the carbon dioxide environment. The overall protection effect of S-1-inhibitor is relatively weak and it is 96.5% at a concentration of 30 mg/l. At this time, the corrosion rate decreases from 7.2 g/m^2·h to 0.252 g/m^2·h.

Table 1

Protection efficiency of S-1 inhibitor in formation water containing hydrogen sulfide

Таблица 1. **Эффективность защиты ингибитора С-1 в пластовых водах, содержащих сероводород**

İnhibitor	Inhibitor concentration, mg/l		Corrosion rate, g/m^2·h		Mass loss, g	Retardation factor, γ	Protection effect, Z, %
			Without inhibitor	With an inhibitor			
S-1	C_1	10	4.8	0.9696	0.007	4.95	79.8
S-1	C_2	15	4.8	0.8448	0.006	5.68	82.4
S-1	C_3	20	4.8	0.5952	0.004	8.06	87.6
S-1	C_4	25	4.8	0.2736	0.002	17.54	94.3
S-1	C_5	30	4.8	0.0864	0.0006	55.55	98.2

Table 2

Protection efficiency of S-1 inhibitor in hydrogen sulfide and carbon dioxide environment

Таблица 2. **Эффективность защиты ингибитора С-1 в среде сероводорода и углекислого газа**

İnhibitor	Inhibitor concentration, mg/l		Corrosion rate, g/m^2·h		Mass loss, g	Retardation factor, γ	Protection effect, Z, %
			Without inhibitor	With an inhibitor			
S-1	C_1	10	7.2	1.71	0.013	4.21	76.2
S-1	C_2	15	7.2	1.33	0.010	5.41	81.4
S-1	C_3	20	7.2	0.820	0.006	8.78	88.6
S-1	C_4	25	7.2	0.453	0.003	15.89	93.7
S-1	C_5	30	7.2	0.252	0.001	28.57	96.5

During the investigation of the bactericidal property of the S-1 reagent, it was found that the life activity of sulfate-reducing bacteria effectively decreases in the "Postgate-B" nutrient medium. Thus, the

number of both types of sulfate-reducing bacteria decreases sharply with the increase in the concentration of the S-1 reagent in the "Postgate-B" medium (Fig. 1, 2). As it can be seen from Figure 1, with the increase of the concentration of S-1 inhibitor from 10 mg/l to 30 mg/l, the effect on the growth factor of Desulfovibriodesulfuricans bacterial cells increases from 20% to 64% on the first day of the test. However, in Desulfomicrobium bacteria, this coefficient increases from 10% to 64% and rises to 53% (Fig. 2).

In the test carried out in "Postgate-B" environment, when the concentration of the reagent was increased to 30 mg/l, its effect on the growth rate of sulfate-reducing bacteria cells is 75% on the second day, 80% – on the third day, 86% – on the fourth day, 88% – on the fifth day, and 89% – on the sixth day and finally on the seventh day it is 90% (Fig. 1). In the second case (Fig. 2) the effect is 63, 68, 75, 79, 80, 80%, respectively. From the comparison of the results of the experiments with the participation of both bacteria it is known that the effect of the reagent in the environment containing Desulfovibriodesulfuricans bacteria is higher than in the Desulfomicrobium bacteria containing environment.

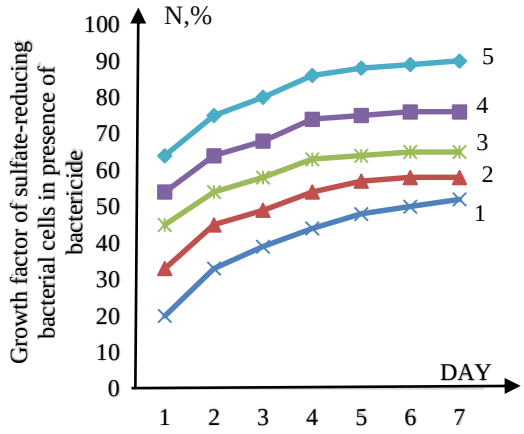

Fig. 1. Effect of S-1 on the growth rate of Desulfovibriodesulfuricans bacterial cells N (%): 1-10; 2-15; 3-20; 4-25, 5-30 mg/l
Рис. 1. Влияние С-1 на скорость роста бактериальных клеток Desulfovibriodesulfuricans N (%): 1–10; 2-15; 3-20; 4-25, 5-30 мг/л

Thus, the studied reagent S-1 reduced the number of Desulfovibriodesulfuricans bacterial cells more than that of Desulfomicrobium bacteria.

Based on the concentration of hydrogen sulfide produced by bacteria, the bactericidal effect (S, %) of reagent S-1 was calculated in the medium containing both bacteria (Fig. 3, 4).

Fig. 3 and 4 show that, with the increase in the concentration of S-1 reagent, its bactericidal effect also increases, and the highest value is observed at a concentration of 30 mg/l %, the second day it is 80%, the third day – 85%, the fourth day – 90%, the fifth day – 91%, the sixth day – 92% and the seventh day – 93%. In Desulfomicrobium bacteria, the value of S is 55, 70, 80, 83, 84, 85, 85%, respectively.

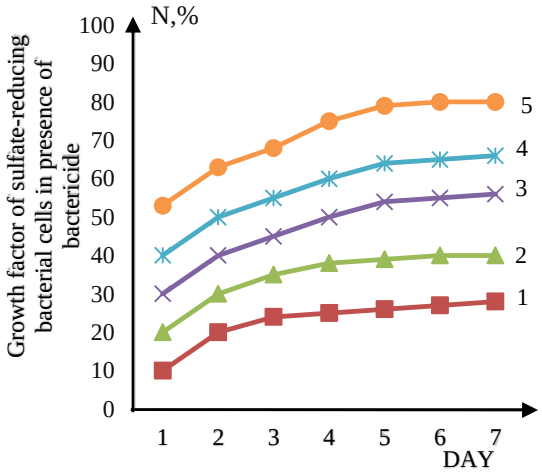

Fig. 2. Effect of S-1 on the growth rate of Desulfomicrobium bacterial cells N (%): 1-10; 2-15; 3-20; 4-25, 5-30 mg/l
Рис. 2. Влияние С-1 на скорость роста бактериальных клеток Desulfomicrobium N (%): 1–10; 2-15; 3-20; 4-25, 5-30 мг/л

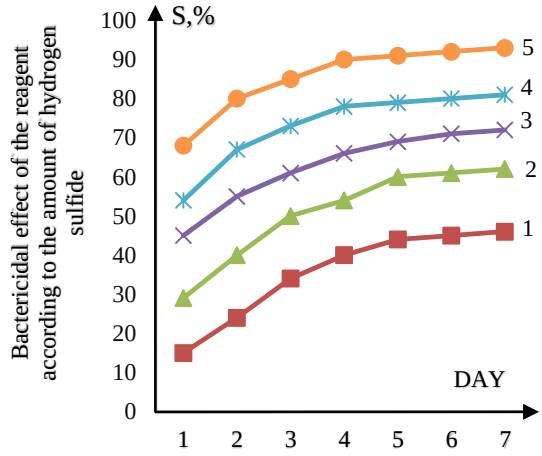

Fig. 3. Bactericidal effect of S-1 (Desulfovibriodesulfuricans): 1-10; 2-15; 3-20; 4-25, 5-30 mg/l
Рис.3.Бактерицидное действие С-1 (Desulfovibriodesulfuricans): 1-10; 2-15; 3-20; 4-25, 5-30 мг/л

Thus, reagent S-1 sharply reduces the amount of biogenic hydrogen sulfide in the "Postgate B" nutrient medium with sulfate-reducing bacteria, but does not completely stop the sulfate reduction process. It is clear that the S-1 reagent prevents the growth of sulfate-reducing bacteria in the nutrient medium, but it cannot completely stop the metabolic process in them.

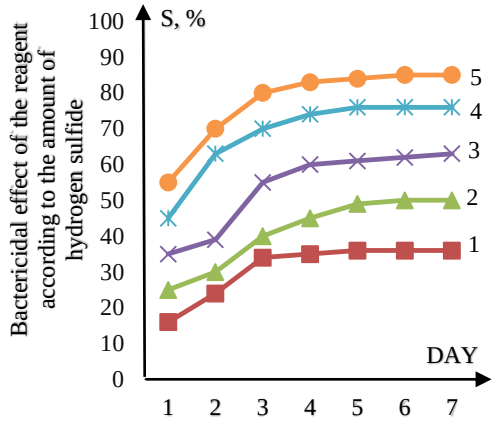

Fig. 4. Bactericidal effect of S-1 (Desulfomicrobium): 1-10; 2-15; 3-20; 4-25, 5-30 mg/l

Рис. 4. Бактерицидное действие С-1 (Desulfomicrobium): 1-10; 2-15; 3-20; 4-25, 5-30 мг/л

CONCLUSION

In order to study the bactericidal-inhibitory properties of the S-1 reagent, for the first time, laboratory tests were carried out in hydrogen sulfide and carbon dioxide containing formation waters and in sulfate-reducing bacteria "Desulfomicrobium" and "Desulfovibriodesulfuricans" containing "Postgate-B" nutrient medium. During the experiment 10, 15, 20, 25 and 30 mg/l concentrations of the reagent were used.

It has been shown that the corrosion protection effect increases as the concentration of S-1 inhibitor increases in formation waters containing hydrogen sulfide and $H_2S + CO_2$, and the highest effect occurs at a concentration of 30 mg/l in both environments. In hydrogen sulphide medium, the corrosion rate is 0.0864 g/m^2·h, and the protective effect of the inhibitor (Z) is 98.2%.

The bactericidal property of the S-1 reagent was studied in the "Postgate-B" nutrient medium which contains sulfate-reducing bacteria "Desulfomicrobium" and "Desulfovibriodesulfurican". The experiments were carried out for seven days, and the effect of the reagent on the growth rate of bacterial cells and the amount of biogenic hydrogen sulfide in the medium were studied.

It was determined that with an increase in the concentration of the inhibitor from 10 mg/l to 30 mg/l, the effect on the growth rate of Desulfovibriodesulfuricans bacteria cells increases from 20% to 64% on the first day of the test. But in Desulfo microbium bacteria this coefficient increases from 64% to 53%. The effect on the growth rate of Desulfovibriodesulfuricans bacteria cells was 75% on the second day, 80% – on the third day, 86% – on the fourth day, 88% – on the fifth day, 89% – on the sixth day, and 90% – on the seventh day. It was 79, 80, 80%.

The result of the calculation showed that as the concentration of reagent S-1 increases, its bactericidal effect also increases and the highest value is observed, at a concentration of 30 mg/l. In an environment with Desulfovibriodesulfuricans the value of the bactericidal effect at 30mg/l concentration inhibitor the first day was 68%, second day – 80%, third day – 85%, fourth day – 90%, fifth day – 91%, sixth day – 92% and seventh day – 93%. In Desulfomicrobium bacteria, the value of S is 55, 70, 80, 83, 84, 85, 85%, respectively.

Авторы заявляют об отсутствии конфликта интересов, требующего раскрытия в данной статье.

The authors declare the absence a conflict of interest warranting disclosure in this article.

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