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Properties of Salicylidene-Aniline as a Corrosion Inhibitor in Oil and Petroleum Products Transportation Systems

Sergey G. Kuren ✉[1], **Svetlana A. Volyanik**[2], **Mariya A. Savenkova**[2], **Elvira A. Zaitseva**
[1] Don State Technical University, Rostov-on-Don, Russian Federation
[2] Rostov State Transport University, Rostov-on-Don, Russian Federation
[3] Southern Federal University, Rostov-on-Don, Russian Federation
✉ sergejgrigorevich@yandex.ru

Abstract
Introduction. Risks of accidents and fires during transportation and pumping of oil and petroleum products is a significant problem of technosphere safety. The reasons may be leaks due to corrosion damage to pipelines, tanks and oil storage tanks. In view of the possible serious financial, environmental and social consequences of such incidents, it is important to constantly seek new, more effective approaches to preventing corrosion processes. It is obvious, for example, that at present not all chemical compounds capable of suppressing or inhibiting the destruction of metals and alloys have been thoroughly studied. The presented scientific work is partly aimed at overcoming the lack of data in this area. The objective is to investigate an insufficiently studied compound salicylidene-aniline, which can be a corrosion inhibitor similar to other nitrogen-containing compounds similar in structure and composition.
Materials and Methods. The gravimetric method was used to study the inhibitory, adsorption and complexing properties of salicylidene-aniline. The experiments were carried out with St3 steel plates. The mass of metal samples without an inhibitor and with an inhibitor was recorded before and after exposure in the test media. The effect was determined by the change in the corrosion rate. When setting up experiments, the authors were guided by GOST 9.905–82 "Unified system of corrosion and ageing protection. Corrosion test methods. General requirements". The volume of the corrosive medium was determined according to GOST 9.506–87 "Unified system of corrosion and ageing protection. Corrosion inhibitors of metals in water-petroleum media. Methods of protective ability evaluation". The corrosion criteria were taken from GOST 9.908–85 "Unified system of corrosion and ageing protection. Metals and alloys. Methods for determination of corrosion and corrosion resistance indices". The calculations were based on the valuation principle, which sets the ratio of a multiplicative metrized linear order on a set of particular criteria. An integral indicator based on mathematical and methodological approaches was used for ranking.
Results. The effect of hydrochloric acid medium on steel samples St3 was studied. Four concentrations of the inhibitor were taken: 0 %, 0.01 %, 0.1 %, and 0.2 %. The mass of metal samples without an inhibitor and with an inhibitor was recorded before and after exposure in the test media. The effect was determined by the change in the corrosion rate. The mass index of corrosion was calculated. The surface quality during destruction and corrosion inhibition was determined in five stages: the elements to be evaluated were selected, the purpose of the evaluation was formulated, the elements of the technical condition of the object under study were found, the essence of determining the usefulness or value of the criterion was described, and the essence of optimization was explained. The studied properties were ranked with respect to the multiplicative metrized linear order on a set of particular criteria. For the calculations, the task was set — to determine the components of vector B in accordance with one of the evaluation stages. We are talking about the stage when the usefulness or value of the criterion is analyzed by points on the numerical axis indicating the state of the object "better — worse". We constructed an indicator z approximating a known or specified (learning) matrix of

paired relationships between objects. The resulting indicator allowed us to assess the technical condition of the surface during the course of corrosion and when it slowed down due to the addition of salicylidene-aniline. The result was obtained for samples with a surface area from $10.1\cdot10^{-4}$ m^2 to $11.9\cdot10^{-4}$ m^2. During the experiment, the mass of the metal decreased by 0.2–0.8 times with the inhibitor and almost by 3.5 times without it. The mass index of corrosion was recorded from 0.15 g/m$^2\cdot$h to 0.48 g/m$^2\cdot$h. At the same time, the protective ability of the inhibitor was quite high: the minimum was 77.4 %, the maximum was 94.8 %. This is a convincing indicator. It is also worth mentioning such an advantage of salicylidene-aniline as a low danger. Its hazard class is III (for comparison: the hazard class of aniline is a level higher — II).

Discussion and Conclusion. The authors propose to use salicylidene-aniline as a means to increase the safe service life of oil pipelines and tanks. The potential of this composition as an effective corrosion inhibitor, highly soluble in oil and petroleum products, has been proven. Such properties of salicylidene-aniline as slow oxidation and moderate toxicity are noted

Keywords: inhibitory properties of salicylidene-aniline, protective ability of the inhibitor, inhibitory additives, ability to complex formation, inhibitor concentration

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Научная статья

Свойства салицилиден-анилина как ингибитора коррозии в системах транспортировки нефти и нефтепродуктов

С.Г. Курень ✉[1], **С.А. Воляник**[2], **М.А. Савенкова**[2], **Э.А. Зайцева**[3]

[1] Донской государственный технический университет, г. Ростов-на-Дону, Российская Федерация

[2] Ростовский государственный университет путей сообщения, г. Ростов-на-Дону, Российская Федерация

[3] Южный федеральный университет, г. Ростов-на-Дону, Российская Федерация

✉sergejgrigorevich@yandex.ru

Аннотация

Введение. Значимая проблема техносферной безопасности — риски аварий и пожаров при транспортировке и перекачке нефти и нефтепродуктов. Причинами могут быть утечки из-за коррозионных повреждений трубопроводов, цистерн, резервуаров нефтехранилищ. Ввиду возможных серьезных финансовых, экологических и социальных последствий таких происшествий важно постоянно изыскивать новые, более эффективные, подходы к предотвращению коррозионных процессов. Очевидно, например, что в настоящее время досконально изучены далеко не все химические соединения, способные подавлять или тормозить разрушение металлов и сплавов. Представленная научная работа призвана отчасти преодолеть недостаток данных в этой области. Цель — исследовать малоизученное соединение салицилиден-анилин, которое может быть ингибитором коррозии аналогично другим, сходным по строению и составу азотсодержащим составам.

Материалы и методы. Для исследования ингибиторных, адсорбционных и комплексообразующих свойств салицилиден-анилина использовали гравиметрический метод. Проводили опыты с пластинами из стали Ст3. Фиксировалась масса металлических образцов без ингибитора и с ингибитором до и после выдержки в испытуемых средах. Эффект определяли по изменению скорости коррозии. При постановке экспериментов ориентировались на ГОСТ 9.905–82 «Единая система защиты от коррозии и старения. Методы коррозионных испытаний». Объем коррозионной среды определили по ГОСТу 9.506–87 «Единая система защиты от коррозии и старения. Ингибиторы коррозии металлов в водно-нефтяных средах». Критерии коррозии взяли из ГОСТа 9.908–85 «Металлы и сплавы. Методы определения показателей коррозии и коррозионной стойкости». Расчеты основывались на принципе оценивания, который задает отношение мультипликативного метризованного линейного порядка на множестве частных критериев. Для ранжирования использовали интегральный показатель, основанный на математическом и методическом подходах.

Результаты исследования. Изучено воздействие солянокислой среды на образцы стали Ст3. Брали четыре концентрации ингибитора: 0 %, 0,01 %, 0,1 %, 0,2 %. Зафиксировали массу металлических образцов без ингибитора и с ингибитором до и после выдержки в испытуемых средах. Эффект определили по изменению скорости коррозии. Рассчитали массовый показатель коррозии. Качество поверхности при разрушении и ингибировании коррозии определяли в пять этапов: выбрали оцениваемые элементы, сформулировали цель оценивания, нашли элементы технического состояния исследуемого объекта, описали суть определения полезности или ценности критерия, пояснили суть оптимизации. Исследуемые свойства ранжировали по отношению мультипликативного метризованного линейного порядка на множестве частных критериев. Для расчетов поставили задачу — определить компоненты вектора B в соответствии с одним из этапов оценки. Речь идет о стадии, когда полезность или ценность критерия анализируется по точкам на числовой оси, указывающим на состояние объекта «лучше — хуже». Построили показатель z, аппроксимирующий известную или задаваемую (обучающую) матрицу парных взаимосвязей между объектами.

Полученный в итоге показатель позволяет оценивать техническое состояние поверхности в процессе протекания коррозии и при ее замедлении за счет добавления салицилиден-анилина. Получен результат для образцов с площадью поверхности от $10{,}1\cdot10^{-4}$ м^2 до $11{,}9\cdot10^{-4}$ м^2. За время эксперимента масса металла уменьшалась в 0,2–0,8 раза с ингибитором и почти в 3,5 раза без него. Фиксировался массовый показатель коррозии от 0,15 г/м^2·ч до 0,48 г/м^2·ч. При этом защитная способность ингибитора оказалась достаточно высокой: минимальная — 77,4 %, максимальная — 94,8 %. Это убедительный показатель. Стоит также упомянуть такое преимущество салицилиден-анилина, как невысокая опасность. Класс его опасности — III (для сравнения: класс опасности анилина на уровень выше — II).

Обсуждение и заключение. Предлагается использовать салицилиден-анилин как средство, повышающее срок безопасной службы нефтепроводов и цистерн. Доказан потенциал этого состава как эффективного ингибитора коррозии, хорошо растворимого в нефти и нефтепродуктах. Отмечены такие свойства салицилиден-анилина, как медленное окисление и умеренная токсичность.

Ключевые слова: игибиторные свойства салицилиден-анилина, защитная способность ингибитора, ингибирующие добавки, способность к комплексообразованию, концентрация ингибитора

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Introduction. Transportation and storage of aggressive liquids, including oil and petroleum products, generate risks of accidents with severe consequences. Special attention needs to be paid to the condition of pipes and tanks and the possibility of their damage. Oil pipelines, railway tanks and tank trucks are constantly in contact with an aggressive environment. They are made of steel, which raises the question of preventing or slowing down corrosion processes. The consequences of such destruction can be accidents and catastrophes accompanied by fires and explosions.

Corrosion inhibitors can be nitrogen-containing organic bases, primarily aliphatic and aromatic amines and their derivatives [1–3]. Azomethines are derivatives of amines. They are promising as weakly basic inhibitors (pKa bases are 1–2 orders of magnitude smaller than those of the initial amines).

Aniline (phenylamine) $C_5H_5NH_2$ is used, in particular, to increase the anti-detonation of fuel. With 1 % of aniline, the octane number of gasoline increases by 3–4 points. However, it is better not to use aniline in its pure form. During long-term storage, such an additive is oxidized, which reduces the quality of gasoline. Aniline derivatives, such as phosphates, are added to solutions of strong electrolytes to inhibit corrosion of carbon steel.

The hazard class of aniline is II (highly hazardous substance), and the hazard class of salicylidene-aniline is III (moderately hazardous). The lesser danger of salicylidene-aniline is a convincing reason for a close study of its anticorrosive properties. In addition, the known protective capabilities of heteroaromatic compounds on the example of piperidines have unequivocally confirmed their qualities as inhibitors of acid corrosion of non-alloy steel St3 [3]. At the same time, poorly soluble substances are formed in interaction with positively charged ions of the corroding metal. They are deposited in the form of ultrathin films (no more than 10 nm) and protect against further corrosion damage.

Anticorrosive protective chemicals are used as inhibitory additives — additives to aggressive media and in combination with protective coatings [2–3]. Complexing agents with anticorrosive properties: complexones (for example, sodium ethylenediaminetetraacetate (trilon B) $Na,H(OOCCH_2)_2N — (CH_2)_2 — N(CH_2COO)_2Na,H$), various complex compounds of d-elements and mixtures based on them. The leading place in this group is occupied by phosphonates of alkaline and alkaline-earth metals [1–5].

The other group includes nitrogen- and sulfur-containing heterocycles. Nitrogen-containing compounds form less toxic products of interaction with a corrosive environment, therefore they are preferable for the development and creation of effective anticorrosive additives [3].

Quaternary ammonium salts of aliphatic and heteroaromatic nitrogenous bases, including derivatives of alcohols, aldehydes, and carboxylic acids, have proven themselves well as protective anticorrosive agents for St3 steel. They dissolve well and are stable in working environments, act in a large pH range [6].

At the same time, in view of the potential serious financial, environmental and social threats associated with corrosion of oil and petroleum products transportation systems, more effective approaches to preventing destructive processes should be continuously sought. It is obvious, for example, that not all chemical compounds capable of suppressing or inhibiting the destruction of metals and alloys have been thoroughly studied. The presented scientific work is to supplement the data in this area.

The study objective is to create compositions with high anticorrosive properties based on aniline derivatives and compounds of the azomethine class. An aniline derivative is a representative of the class of azomethines-salicylidene-aniline. We believe that this composition or compositions based on it should be used as a corrosion inhibitor of steel, which will eventually increase the safety of transportation of oil and petroleum products.

Materials and Methods. The following features of salicylidene-aniline were studied:

– inhibitory properties;

– anticorrosive activity;

– ability to complex formation;

– adsorption capabilities.

Inhibitor molecules form a contact layer on the metal surface due to the donor-acceptor bond between the electron pairs of nitrogen atoms and the free d-orbitals of the complexing agent atom (in this case, iron). Such compounds form a stronger film than many other inhibitors. As a result, micro-galvanic vapors are formed on the surface of the steel and anodic and cathodic depolarization occurs, due to which the protective properties of the inhibitor are manifested.

The gravimetric research method was used in work [1]. It is based on fixing the mass of metal samples without an inhibitor and with an inhibitor before and after the exposure in test media. The anticorrosive activity of the tested compound is estimated by the change in the corrosion rate. For each sample, the holding time in an inhibited medium is equal to the time in an environment without an anticorrosive agent [1–3].

The authors of the research used plates made of steel grade St3 according to GOST 9.905–82 "Unified system of corrosion and ageing protection. Corrosion test methods. General requirements". The volume of the corrosive medium (0.25n HCl) is 30 cm^3 in accordance with GOST 9.506–87 "Unified system of corrosion and ageing protection. Corrosion inhibitors of metals in water-petroleum media. Methods of protective ability evaluation". 0 %, 0.01 %, 0.1 % and 0.2 % salicylidene-aniline were added in the medium. Corrosion criteria was according to GOST 9.908–85 "Unified system of corrosion and ageing protection. Metals and alloys. Methods for determination of corrosion and corrosion resistance indices".

Results. The effect of hydrochloric acid medium on steel samples was considered. The results were recorded taking into account different concentrations of salicylidene-aniline and thus the anticorrosive effect was determined (Fig. 1).

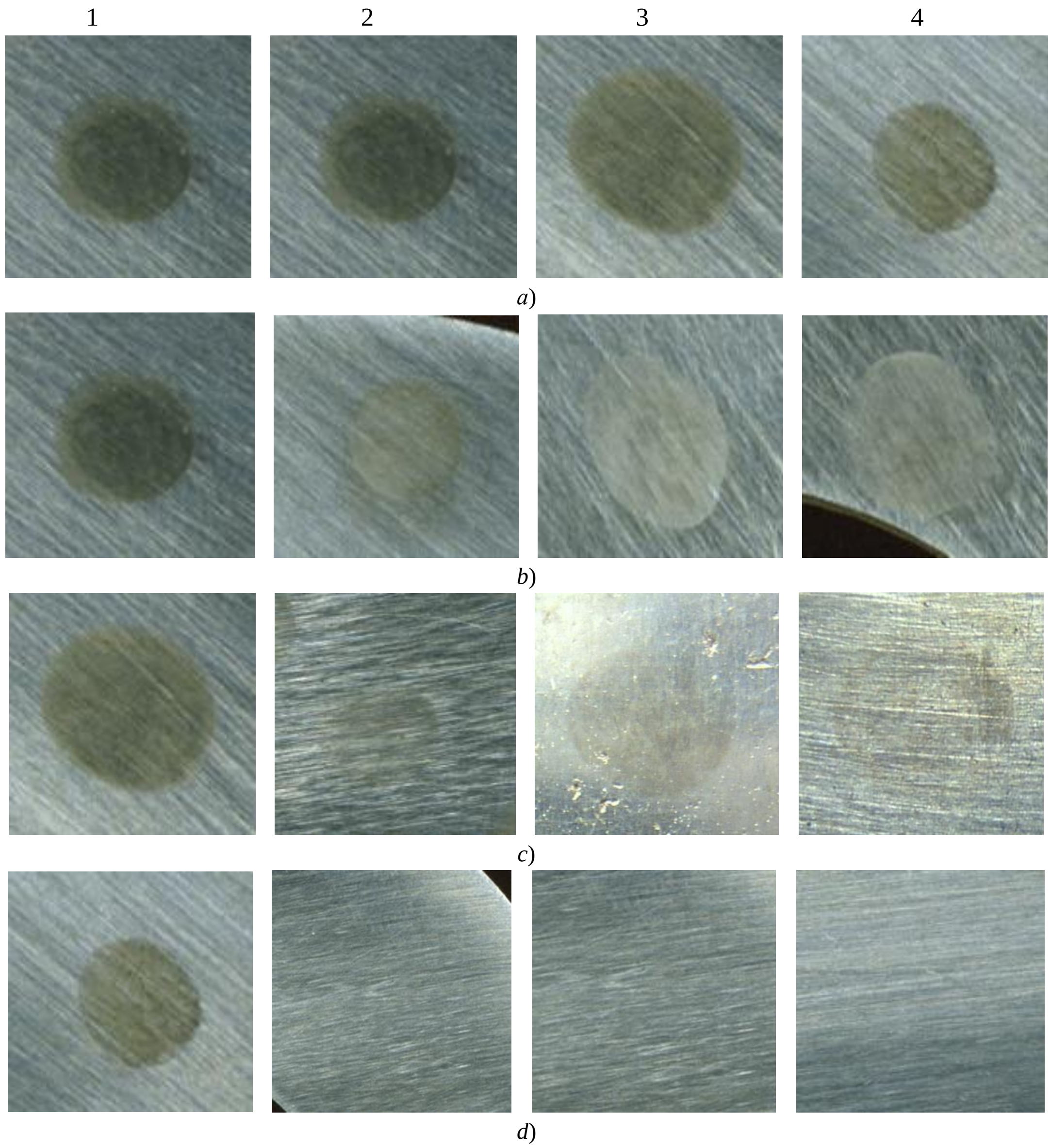


Fig. 1. Photo of corrosion damage — a type of rust stain on the surface of St3 steel samples in hydrochloric acid medium at inhibitor concentrations, %: *a* — 0; *b* — 0.01; *c* — 0.1; *d* — 0.2. Samples are numbered from top to bottom, their images after exposure are arranged vertically

Mass index of corrosion *j* was calculated by formula:

$$j = \Delta m / (S \cdot \tau), \text{г} / \text{м}^2 \cdot \text{ч или г} / \text{м}^2 \cdot \text{год},$$

where Δm — decrease in the mass of metal (g) during metal corrosion during time τ (hour or year) on the surface *S* (m^2).

The surface quality during corrosion and its inhibition (Fig. 1) was evaluated in five stages [7].

1. Based on the preliminary analysis, the problem was defined, the evaluated elements were indicated.

2. Structural analysis allowed us to formulate the main objective of the assessment and the possibilities of achieving it.

3. For the analysis of uncertainty, the search for unifying elements of the actual technical condition of the object was conducted.

4. The usefulness or value of the criterion was analyzed by the points of the numerical axis indicating the state of the object "better — worse".

5. Optimization was the search for a way to achieve the necessary technical condition.

Methods of constructing the structure of goals and the formation of a list of criteria were discussed in [8]. It was assumed that the initial array of objects was set and the study identified the best of them in terms of technical condition. Ranking was carried out according to a special integral indicator based on mathematical and methodological approaches. The main principles of selection were described in [9].

The main element of the approach under consideration was the rule (or principle) of estimating π, which set the ratio of a multiplicative metrized linear order on a set of particular criteria.

For a pair of objects a_ν and a_μ the choice was based on the following principles:

– $a_\nu > a_\mu$ — the first object was "better" than the second by the factors considered;

– $a_\nu \sim a_\mu$ — the objects were evaluated equally;

– $a_\nu < a_\mu$ — the first object was "worse" than the second by the factors considered.

Pairs of objects were characterized by comparison vectors $S_{\nu\mu}$. The principle of generalized criteria and so-called lexicographic approaches were useful for research — with a strong preference on a set of particular criteria. In this variant, the objects were equivalent ($a_\nu \sim a_\mu$), and their estimates corresponded to the established minimum values [10].

Let us consider the condition:

$$a_\nu \sim a_\mu \Leftrightarrow x_i^\nu \geq d_i,\ \ x_i^\mu \geq d_i\ , i = 1, \dots, m,$$

where d_i — the given threshold value (sufficiency level); m — the number of indicators under consideration; $x_{i\nu}$ and $x_{i\mu}$ — the estimates of the objects being compared (ν-th and μ-th).

The direct use of this principle was limited by the possibility of its application in practice, since it implied a strong preference on a set of criteria $K = \{K_1, K_2, \dots, K_m\}$ in the form:

$$K_1 \geq K_2 \geq K_3 \geq \cdots \geq K_m.$$

It was more expedient to set a metrized multiplicative relation of linear order, that is, to apply a generalized criterion.

In this case,

$$a_\nu \succ a_\mu \Leftrightarrow \sum_{i=1}^{m} a_i K_i(a_\mu);\ \ a_\nu \sim a_\mu \Leftrightarrow \sum_{i=1}^{m} a_i\, K_i(a_\nu) = \sum_{i=1}^{m} a_i K_i\,(a_\mu).$$

Here a_i — the coefficients satisfying, for example, the condition

$$\sum_{i=1}^{m} a_i = 1.$$

The task was to determine the components of vector B in accordance with the 4-th stage of evaluation and to construct an indicator z, approximating an objectively known or specially specified (training) matrix of paired relationships between artificial objects (in [11] these were vehicles):

$$Q = \| q_{rk} \|_{p,p}\ .$$

Here p — the number of artificial objects under consideration, which determined the size of the matrix Q; q_{rk} — the elements of the matrix; r, k — the artificial objects on the numerical axis "better — worse".

On the *z* axis, the square of the distance between the *r-th* and *k-th* artificial objects (protection options) had the form:

$$d_{rk}(B) = (z_r - z_k)^2 = \left[\sum_{j=1}^{m} b_j(x_{rj} - x_{kj})\right]^2,$$

$$D(B) = \| d_{rk} \|_{p,p}.$$

The matrix *D* (*B*) was evaluated using the functional

$$J(B) = \sum_{r=1}^{p-1} \sum_{k=r+1}^{p} [d_{rk}(B) - q_{rk}]^2.$$

The desired integral criterion was the function *Z*, if *J*(*B*) was minimal and the vector *B* corresponded to the set conditions. The obtained indicator was used to assess the technical condition of the surface quality as a result of corrosion and its deceleration due to the addition of salicylidene-aniline (Table 1, Fig. 1).

Table 1

Anticorrosive properties of Salicylidene-aniline for St3 steel

Sample number	S, 10^{-4} m^2	τ, h	$\Delta m = m_0 - m$, г	Inhibitor concentration, %	Mass corrosion index *j*, g/m^2·h ($j = \Delta m / S \cdot \tau$)	Protective ability of inhibitor Z, %
1	10.22	120	3.4590	0	2.8205	–
2	10.11	120	0.7815	0.01	0.4793	77.4
3	11.9	120	0.3363	0.1	0.2542	90.2
4	11.13	120	0.1805	0.2	0.1482	94.8

As it can be seen from the table and Fig. 1, the corrosion damage of the steel surface decreased with an increase in the concentration of salicylidene-aniline.

To explain the anticorrosive activity and good adsorption ability of N-salicylidene-aniline on a steel surface, its photoinitiated structural rigidity was theoretically studied. This made it possible to find out in which tautomeric form this structure had a high energy of complexation of adsorption complexes on the surface of the protected metal [12-16].

Photochromic transition of the ketone form to the enol form was provided by proton transfer between the isomeric forms of the N-salicylidene-aniline molecule.

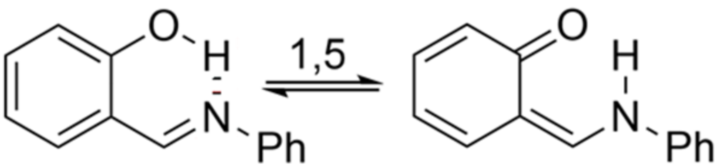
The semi-empirical method of quantum chemistry PM3 (parametric method 3) was used for a detailed determination:

– sequences of elementary stages of the main photochromic process;

– geometric characteristics of all intermediate and final photo-colored systems;

– charge distributions on atoms [17].

Salicylidene-aniline, like other anilines, is of interest because of the intramolecular hydrogen bond formed between oxygen and nitrogen atoms in a more stable cis-enol form. In fact, the forms of OH and NH are in equilibrium. Depending on the position of the hydrogen atom in this connection, O—H—N anils exhibit two tautomeric forms: enol-imine and keto-enamine (Fig. 2). The cis form is more stable. But the trans form has a better adsorption capacity. Tautomerism occurs under the influence of external factors such as radiation, temperature and pressure.



a) *b*)

Fig. 2. Keto-enol tautomerism: *a* — CA (enol); *b* — keto form

When the temperature decreases, the enol form turns into a ketone form. We proved that it was better adsorbed on the steel surface. Cis-enol is colorless. When irradiated with ultraviolet light, it turns into a red trans-ketoform. The keto isomer can be reversibly discolored thermally or photochemically under the action of visible light [18].

Discussion and Conclusion. The results of the study suggest that salicylidene-aniline actively inhibits corrosion of St3 steel. The degree of protection reaches 77.4–94.8 %.

Quantum chemical calculations using the semi-empirical PM3 method allowed us to explain the photochromism of salicylidene-aniline due to keto-enol tautomerism. Due to the monomolecular prototropic acid-base dichotomy, salicylidene-aniline has photochromism and luminescence. This distinguishes it from other azomethines.

The structure of the adsorption complex with higher stabilization energy was revealed, which explained the better adsorption capacity of the keto form of the inhibitor.

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About the Authors:

Sergey G. Kuren, Cand. Sci. (Eng), Associate Professor of the Transport Systems and Logistics Operation Department, Don State Technical University (1, Gagarin Sq., Rostov-on-Don, 344003, RF), AuthorID, ORCID, sergejgrigorevich@yandex.ru

Svetlana A. Volyanik, Cand. Sci. (Eng), Associate Professor, head of the Chemistry Department, Rostov State Transport University (2, Rostovskogo Strelkovogo Polka Narodnogo Opolchenya Sq., Rostov-on-Don, 344038, RF), AuthorID, ORCID, him@rgupa.ru

Mariya A. Savenkova, Cand. Sci. (Eng), Associate Professor of the Chemistry Department, Rostov State Transport University (2, Rostovskogo Strelkovogo Polka Narodnogo Opolchenya Sq., Rostov-on-Don, 344038, RF), AuthorID, ORCID, him@rgupa.ru

Elvira A. Zaitseva, Student of the Chemical Faculty, Southern Federal University (7, Zorge St., Rostov-on-Don, 344090, RF), ORCID, gems10@rambler.ru

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SG Kuren: academic advising; formulation of the basic concept, goals and objectives of the study; conducting quantum chemical calculations and experiment; conclusions correction.

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Об авторах:

Сергей Григорьевич Курень, кандидат технических наук, доцент кафедры эксплуатации транспортных систем и логистики Донского государственного технического университета (344003, РФ, г. Ростов-на-Дону, пл. Гагарина, 1), AuthorID, ORCID, sergejgrigorevich@yandex.ru

Светлана Алексеевна Волянник, кандидат технических наук, доцент, заведующая кафедрой химии Ростовского государственного университета путей сообщения (344038, РФ, г. Ростов-на-Дону, пл. Ростовского Стрелкового Полка Народного Ополчения, 2), AuthorID, ORCID, him@rgupa.ru

Мария Андреевна Савенкова, кандидат химических наук, доцент кафедры химии Ростовского государственного университета путей сообщения (344038, РФ, г. Ростов-на-Дону, пл. Ростовского Стрелкового Полка Народного Ополчения, 2), AuthorID, ORCID, him@rgupa.ru

Эльвира Александровна Зайцева, студентка химического факультета Южного федерального университета (344090, РФ, г. Ростов-на-Дону, ул. Зорге, 7), ORCID, gems10@rambler.ru

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С.А. Воляник — проведение эксперимента, подготовка текста, формулирование выводов.

М.А. Савенкова —проведение эксперимента, анализ результатов исследований, доработка текста.

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